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Chem. & Phys
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JOURNAL
OF THE
SOCIETY OF CHEMICAL
INDUSTRY

VOL. XXXIX
1920

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138,657	255A	140,473	713A	142,206	440A	144,030	544A	147,270	724A	150,598	716A	152,401	783A
138,679	312A	140,478	426A	142,226	468A	144,051	741A	147,286	650A	150,638	697A	152,402	788A
138,745	255A	140,484	404A	142,237	450A	144,052	622A	147,311	593A	150,654	731A	152,403	788A
138,758	255A	140,505	405A	142,238	458A	144,079	555A	147,337	640A	150,741	714A	152,420	776A
138,762	256A	140,507	412A	142,293	469A	144,080	525A	147,338	725A	150,762	712A	152,427	788A
138,788	310A	140,508	412A	142,310	456A	144,083	542A	147,353	651A	150,765	726A	152,437	816A
138,796	308A	140,509	412A	142,315	456A	144,084	541A	147,360	593A	150,782	719A	152,447	788A
138,799	298A	140,513	417A	142,330	436A	144,119	520A	147,378	641A	150,785	711A	152,470	777A
138,802	256A	140,514	417A	142,334	488A	144,133	552A	147,391	590A	150,786	712A	152,477	809A
138,862	487A	140,527	426A	142,365	450A	144,142	548A	147,487	750A	150,787	715A	152,486	788A
138,905	714A	140,563	376A	142,376	440A	144,176	551A	147,956	597A	150,793	724A	152,495	833A
138,947	372A	140,568	415A	142,398	456A	144,185	548A	147,958	597A	150,802	727A	152,501	787A
138,948	372A	140,578	356A	142,416	460A	144,204	542A	147,964	623A	150,807	712A	152,505	788A
138,950	371A	140,635	392A	142,519	489A	144,326	549A	147,965	592A	150,810	726A	152,509	773A
138,954	371A	140,691	426A	142,522	487A	144,336	540A	147,967	594A	150,811	726A	152,513	779A
138,967	338A	140,709	419A	142,541	479A	144,342	547A	147,988	603A	150,819	731A	152,515	776A
138,981	303A	140,715	391A	142,576	495A	144,359	546A	147,999	601A	150,836	724A	152,526	783A
138,999	313A	140,740	781A	142,602	484A	144,374	537A	148,003	593A	150,917	722A	152,529	803A
138,999	313A	140,831	426A	142,615	482A	144,398	715A	148,027	622A	150,940	712A	152,548	775A
139,005	294A	140,893	365A	142,688	491A	144,478	551A	148,057	622A	151,024	759A	152,549	775A
139,018	287A	140,835	414A	142,710	475A	144,479	551A	148,074	640A	151,027	759A	152,550	833A
139,065	372A	140,844	365A	142,721	491A	144,520	741A	148,095	597A	151,029	719A	152,554	783A
139,091	289A	140,880	436A	142,729	478A	144,531	609A	148,587	640A	151,031	712A	152,607	823A
139,099	310A	140,898	392A	142,738	501A	144,563	541A	148,588	619A	151,038	712A	152,712	810A
139,100	310A	140,911	419A	142,763	502A	144,589	714A	148,615	626A	151,056	719A	152,724	810A
139,106	289A	140,915	420A	142,764	500A	144,629	820A	148,617	624A	151,072	739A	152,733	788A
139,115	290A	140,943	373A	142,771	514A	144,737	690A	148,630	619A	151,086	739A	152,740	825A
139,143	788A	140,948	413A	142,874	510A	144,744	573A	148,637	619A	151,087	715A	152,741	814A
139,153	527A	140,955	398A	142,875	527A	144,751	571A	148,641	659A	151,092	725A	152,752	821A
139,161	776A	140,967	469A	142,878	527A	144,802	575A	148,645	724A	151,098	722A	152,766	819A
139,191	788A	140,972	392A	142,879	517A	144,806	582A	148,659	711A	151,143	711A	152,780	820A
139,229	302A	141,001	391A	142,880	527A	144,819	545A	148,724	638A	151,184	723A	152,785	809A
139,232	483A	141,028	776A	142,882	518A	144,842	575A	148,734	638A	151,192	715A	152,791	813A
139,233	290A	141,089	413A	142,885	492A	144,868	565A	149,033	672A	151,229	761A	152,815	823A
139,234	291A	141,091	396A	142,898	529A	144,869	569A	149,038	682A	151,293	754A	152,818	824A
139,239	305A	141,093	405A	142,902	501A	144,895	582A	149,049	726A	151,314	739A	152,835	821A
139,247	302A	141,097	392A	142,934	491A	144,897	582A	149,055	682A	151,323	753A	152,841	825A
139,263	328A	141,113	409A	142,938	479A	144,961	563A	149,085	647A	151,328	748A	152,860	825A
139,267	297A	141,122	414A	142,941	475A	144,979	538A	149,086	652A	151,329	741A	152,879	821A
139,276	302A	141,124	408A	142,945	479A	144,989	565A	149,095	656A	151,339	748A	152,887	819A
139,278	482A	141,125	408A	142,946	495A	145,099	567A	149,120	659A	151,344	757A	152,898	809A
139,296	339A	141,132	413A	142,947	527A	145,101	566A	149,355	701A	151,366	739A	152,904	789A
139,315	297A	141,142	451A	142,955	491A	145,102	566A	149,365	664A	151,367	739A	152,914	819A
139,318	298A	141,172	440A	142,971	525A	145,113	569A	149,366	694A	151,374	753A	152,916	826A
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139,383	310A	141,216	416A	143,017	479A	145,124	577A	149,398	684A	151,420	754A	153,041	811A
139,390	288A												

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(Note.—The letter "R" indicates that a reissue of the patent is referred to.)

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1,186,522R	157A	1,320,376	7A	1,322,139	3A	1,323,834	118A	1,325,665	144A	1,327,001	182A	1,328,258	207A
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1,329,739	260A	1,331,732	287A	1,333,328	459A	1,331,745	327A	1,336,806	419A	1,338,357	450A	1,339,855	493A
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1,329,825	217A	1,331,802	288A	1,333,586	754A	1,335,009	412A	1,337,019	404A	1,338,440	460A	1,340,316	510A
1,329,852	221A	1,331,803	302A	1,333,619	338A	1,335,024	415A	1,337,027	463A	1,338,454	439A	1,340,353	491A
1,329,853	221A	1,331,804	302A	1,333,624	337A	1,335,079	376A	1,337,058	458A	1,338,473	450A	1,340,427	510A
1,329,859	257A	1,331,816	303A	1,333,631	326A	1,335,094	413A	1,337,062	416A	1,338,496	817A	1,340,437	493A
1,329,873	228A	1,331,877	310A	1,333,633	347A	1,335,097	401A	1,337,093	414A	1,338,566	778A	1,340,451	493A
1,329,898	235A	1,331,888	304A	1,333,654	371A	1,335,098	401A	1,337,094	393A	1,338,517	456A	1,340,480	493A
1,329,918	248A	1,331,903	305A	1,333,665	328A	1,335,157	361A	1,337,095	393A	1,338,518	456A	1,340,495	479A
1,330,003	217A	1,331,901	305A	1,333,666	338A	1,335,161	355A	1,337,106	404A	1,338,531	497A	1,340,532	510A
1,330,008	222A	1,331,905	305A	1,333,688	339A	1,335,162	380A	1,337,141	401A	1,338,566	451A	1,340,535	489A
1,330,014	222A	1,331,906	305A	1,333,694	261A	1,335,167	366A	1,337,144	396A	1,338,598	450A	1,340,518	492A
1,330,016	216A	1,331,909	290A	1,333,700	514A	1,335,171	414A	1,337,156	416A	1,338,613	489A	1,340,603	480A
1,330,025	263A	1,331,937	296A	1,333,701	529A	1,335,175	414A	1,337,170	391A	1,338,631	491A	1,340,633	475A
1,330,032	232A	1,331,964	288A	1,333,706	520A	1,335,176	411A	1,337,175	393A	1,338,651	419A	1,340,649	483A
1,330,058	751A	1,331,969	321A	1,333,716	327A	1,335,177	414A	1,337,191	488A	1,338,655	455A	1,340,653	479A
1,330,071	262A	1,331,989	301A	1,333,720	339A	1,335,199	416A	1,337,192	488A	1,338,661	445A	1,340,696	488A
1,330,127	202A	1,331,991	287A	1,333,721	339A	1,335,202	356A	1,337,209	457A	1,338,667	441A	1,340,708	498A
1,330,131	267A	1,332,018	290A	1,333,790	355A	1,335,203	356A	1,337,210	457A	1,338,678	831A	1,340,721	495A
1,330,136	235A	1,332,028	291A	1,333,796	338A	1,335,204	356A	1,337,239	488A	1,338,684	464A	1,340,776	495A
1,330,148	274A	1,332,058	297A	1,333,807	329A	1,335,209	415A	1,337,251	436A	1,338,691	766A	1,340,777	496A
1,330,164	256A	1,332,078	301A	1,333,819	334A	1,335,210	415A	1,337,253	401A	1,338,698	437A	1,340,793	510A
1,330,173	273A	1,332,092	288A	1,333,824	339A	1,335,257	403A	1,337,264	450A	1,338,709	437A	1,340,826	495A
1,330,174	273A	1,332,100	295A	1,333,848	336A	1,335,271	265A	1,337,276	456A	1,338,731	476A	1,340,838	495A
1,330,175	256A	1,332,113	295A	1,333,849	335A	1,335,279	366A	1,337,287	814A	1,338,734	498A	1,340,873	522A
1,330,195	256A	1,332,114	295A	1,333,850	335A	1,335,284	414A	1,337,298	437A	1,338,771	450A	1,340,888	657A
1,330,210	272A	1,332,115	295A	1,333,869	322A	1,335,338	376A	1,337,305	459A	1,338,803	463A	1,340,889	657A
1,330,219	256A	1,332,137	297A	1,333,871	322A	1,335,348	392A	1,337,306	776A	1,338,826	457A	1,340,905	517A
1,330,227	256A	1,332,138	294A	1,333,895	352A	1,335,370	413A	1,337,317	442A	1,338,831	449A	1,340,951	520A
1,330,231	270A	1,332,152	302A	1,333,914	375A	1,335,398	392A	1,337,339	459A	1,338,881	456A	1,341,039	525A
1,330,238	217A	1,332,164	287A	1,333,946	355A	1,335,438	392A	1,337,342	411A	1,338,909	459A	1,341,031	507A
1,330,246	484A	1,332,195	333A	1,333,950	375A	1,335,450	362A	1,337,380	445A	1,338,966	458A	1,341,040	525A
1,330,247	484A	1,332,203	328A	1,333,961	326A	1,335,474	414A	1,337,382	795A	1,338,979	467A	1,341,044	510A
1,330,263	268A	1,332,220	323A	1,333,965	339A	1,335,482	450A	1,337,402	460A	1,338,983	440A	1,341,100	520A
1,330,288	281A	1,332,284	323A	1,333,985	372A	1,335,490	373A	1,337,451	502A	1,338,984	440A	1,341,103	522A
1,330,331	255A	1,332,290	326A	1,334,004	371A	1,335,692	393A	1,337,488	476A	1,338,996	437A	1,341,119	517A
1,330,332	255A	1,332,307	339A	1,334,014	322A	1,335,695	391A	1,337,489	476A	1,338,999	437A	1,341,114	517A
1,330,369	275A	1,332,320	343A	1,334,023	322A	1,335,743	404A	1,337,496	477A	1,339,009	456A	1,341,115	517A
1,330,432	256A	1,332,348	326A	1,334,033	328A	1,335,758	393A	1,337,517	437A	1,339,022	441A	1,341,207	525A
1,330,433	256A	1,332,359	326A	1,334,049	342A	1,335,767	404A	1,337,523	410A	1,339,092	464A	1,341,250	522A
1,330,444	272A	1,332,369	326A	1,334,050	342A	1,335,768	404A	1,337,536	460A	1,339,112	440A	1,341,257	510A
1,330,463	257A	1,332,370	336A	1,334,060	343A	1,335,769	404A	1,337,538	460A	1,339,115	436A	1,341,258	510A
1,330,495	274A	1,332,380	322A	1,334,061	343A	1,335,770	404A	1,337,542	788A	1,339,134	460A	1,341,288	517A
1,330,543	744A	1,332,419	334A	1,334,089	372A	1,335,771	404A	1,337,543	457A	1,339,142	469A	1,341,299	507A
1,330,573	272A	1,332,422	337A	1,334,092	338A	1,335,772	404A	1,337,548	458A	1,339,143	469A	1,341,307	517A
1,330,581	267A	1,332,438	350A	1,334,150	359A	1,335,773	404A	1,337,561	488A	1,339,141	469A	1,341,361	513A
1,330,611	268A	1,332,439	334A	1,334,160	355A	1,335,774	404A	1,337,567	339A	1,339,145	469A	1,341,323	517A
1,330,624	260A	1,332,448	381A	1,334,170	325A	1,335,785	429A	1,337,665	488A	1,339,146	469A	1,341,337	517A
1,330,632	261A	1,332,460	335A	1,334,179	339A	1,335,788	429A	1,337,673	443A	1,339,147	469A	1,341,362	516A
1,330,738	267A	1,332,471	336A	1,334,180	325A	1,335,789	429A	1,337,703	457A	1,339,148	469A	1,341,508	520A
1,330,746	256A	1,332,488	331A	1,334,193	339A	1,335,790	429A	1,337,703R	603A	1,339,149	469A	1,341,510	519A
1,330,747	256A	1,332,489	331A	1,334,216	343A	1,335,829	356A	1,337,704	445A	1,339,150	469A	1,341,516	517A
1,330,772	267A	1,332,525	326A	1,334,231	355A	1,335,831	397A	1,337,724	458A	1,339,211	449A	1,341,517	511A
1,330,844	290A	1,332,529	327A	1,334,303	386A	1,335,846	414A	1,337,750	450A	1,339,219	469A	1,341,523	522A
1,330,846	303A	1,332,537	335A	1,334,309	321A	1,335,891	393A	1,337,775	468A	1,339,225	439A	1,341,550	519A
1,330,937	310A	1,332,541	361A	1,334,310	338A	1,335,925	362A	1,337,802	497A	1,339,242	448A	1,341,558	520A
1,330,992	293A	1,332,542	365A	1,334,331	754A	1,335,926	378A	1,337,811	451A	1,339,260	436A	1,341,561	523A
1,331,051	290A	1,332,562	382A	1,334,356	380A	1,335,949	405A	1,337,839	457A	1,339,266	450A	1,341,598	523A
1,331,085	291A	1,332,581	333A	1,334,357	380A	1,335,982	415A	1,337,877	445A	1,339,310	815A	1,341,601	518A
1,331,092	314A	1,332,585	339A	1,334,358	380A	1,335,983	415A	1,337,903	488A	1,339,344	450A	1,341,686	520A
1,331,104	294A	1,332,657	322A	1,334,360	386A	1,335,986	386A	1,337,984	439A	1,339,378	457A	1,341,705	523A
1,331,105	294A	1,332,674	339A	1,334,379	371A	1,335,991	415A	1,338,021	451A	1,339,393	494A	1,341,710	529A
1,331,125	310A	1,332,675	514A	1,334,384	363A	1,335,992	415A	1,338,033	451A	1,339,424	493A	1,341,734	517A
1,331,127	307A	1,332,680	528A	1,334,404	358A	1,336,017	378A	1,338,037	451A	1,339,428	494A	1,341,774	549A
1,331,175	302A	1,332,684	321A	1,334,405	358A	1,336,056	366A	1,338,040	783A	1,339,431	479A	1,341,786	549A
1,331,213	340A	1,332,703	321A	1,334,419	375A	1,336,081	415A	1,338,103	402A	1,339,469	823A	1,341,884	507A
1,331,225	321A	1,332,706	322A	1,334,420									

No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.
1,342,602	541A	1,344,141	573A	1,346,343	724A	1,348,578	828A	1,350,343	664A	1,352,216	686A	1,354,719	759A
1,342,603	541A	1,344,153	570A	1,346,344	724A	1,348,600	623A	1,350,359	662A	1,352,241	715A	1,354,725	745A
1,342,606	548A	1,344,163	563A	1,346,365	597A	1,348,642	620A	1,350,408	704A	1,352,271	695A	1,354,726	745A
1,342,651	550A	1,344,165	550A	1,346,385	606A	1,348,687	637A	1,350,419	694A	1,352,272	695A	1,354,731	744A
1,342,668	551A	1,344,215	563A	1,346,473	602A	1,348,688	637A	1,350,433	648A	1,352,322	695A	1,354,806	749A
1,342,694	548A	1,344,220	550A	1,346,515	592A	1,348,689	637A	1,350,440	604A	1,352,328	695A	1,354,809	742A
1,342,701	520A	1,344,241	538A	1,346,571	628A	1,348,722	623A	1,350,472	684A	1,352,331	695A	1,354,818	749A
1,342,711	542A	1,344,258	622A	1,346,624	626A	1,348,724	625A	1,350,473	684A	1,352,338	712A	1,354,822	758A
1,342,737	555A	1,344,270	552A	1,346,642	603A	1,348,731	624A	1,350,474	684A	1,352,367	694A	1,354,824	790A
1,342,741	539A	1,344,330	563A	1,346,728	603A	1,348,736	627A	1,350,475	684A	1,352,377	714A	1,354,838	754A
1,342,743	538A	1,344,372	575A	1,346,797	651A	1,348,741	641A	1,350,482	684A	1,352,399	695A	1,354,889	754A
1,342,790	743A	1,344,392	549A	1,346,798	651A	1,348,757	648A	1,350,483	694A	1,352,534	725A	1,354,900	754A
1,342,798	558A	1,344,436	563A	1,346,802	595A	1,348,775	629A	1,350,591	667A	1,352,541	726A	1,354,903	744A
1,342,804	549A	1,344,437	563A	1,346,803	595A	1,348,804	661A	1,350,600	655A	1,352,580	725A	1,355,003	773A
1,342,809	550A	1,344,438	563A	1,346,819	602A	1,348,843	657A	1,350,621	653A	1,352,623	712A	1,355,008	743A
1,342,829	597A	1,344,461	573A	1,346,830	599A	1,348,873	627A	1,350,624	662A	1,352,648	712A	1,355,009	743A
1,342,834	544A	1,344,566	576A	1,346,833	595A	1,348,874	627A	1,350,627	652A	1,352,653	722A	1,355,009	743A
1,342,839	597A	1,344,585	563A	1,346,839	726A	1,348,889	661A	1,350,709	694A	1,352,696	715A	1,355,103	743A
1,342,842	550A	1,344,671	566A	1,346,854	726A	1,348,933	657A	1,350,714	694A	1,352,700	722A	1,355,105	743A
1,342,846	523A	1,344,673	583A	1,346,874	599A	1,348,966	650A	1,350,740	654A	1,352,710	724A	1,355,107	826A
1,342,910	548A	1,344,681	575A	1,346,959	599A	1,348,974	654A	1,350,750	647A	1,352,763	727A	1,355,107	826A
1,342,911	548A	1,344,691	568A	1,346,967	593A	1,348,977	671A	1,350,756	702A	1,352,768	734A	1,355,107	826A
1,342,917	549A	1,344,705	571A	1,346,974	595A	1,348,990	671A	1,350,798	666A	1,352,797	712A	1,355,107	826A
1,342,947	599A	1,344,719	568A	1,347,026	603A	1,348,999	638A	1,350,814	652A	1,352,834	727A	1,355,107	826A
1,342,953	550A	1,344,745	566A	1,347,071	611A	1,349,000	638A	1,350,820	654A	1,352,912	725A	1,355,107	826A
1,342,956	544A	1,344,752	603A	1,347,074	605A	1,349,040	622A	1,350,824	666A	1,352,916	716A	1,355,107	826A
1,342,993	550A	1,344,753	603A	1,347,083	611A	1,349,053	663A	1,350,825	659A	1,352,917	716A	1,355,107	826A
1,343,034	557A	1,344,770	570A	1,347,088	727A	1,349,056	648A	1,350,858	637A	1,353,035	712A	1,355,107	826A
1,343,037	511A	1,344,788	569A	1,347,089	727A	1,349,113	620A	1,350,865	694A	1,353,126	725A	1,355,107	826A
1,343,040	519A	1,344,830	571A	1,347,103	708A	1,349,114	620A	1,350,876	726A	1,353,166	739A	1,355,107	826A
1,343,063	583A	1,344,850	580A	1,347,158	596A	1,349,134	657A	1,350,877	726A	1,353,166	739A	1,355,107	826A
1,343,077	584A	1,344,851	580A	1,347,159	596A	1,349,150	634A	1,350,891	695A	1,353,167	739A	1,355,107	826A
1,343,109	539A	1,344,869	602A	1,347,160	596A	1,349,156	653A	1,350,892	695A	1,353,169	765A	1,355,107	826A
1,343,135	541A	1,344,878	576A	1,347,181	599A	1,349,159	647A	1,350,893	695A	1,353,182	744A	1,355,107	826A
1,343,138	542A	1,344,893	564A	1,347,189	725A	1,349,185	647A	1,350,907	652A	1,353,194	755A	1,355,107	826A
1,343,139	542A	1,344,905	570A	1,347,200	603A	1,349,200	658A	1,350,932	658A	1,353,220	757A	1,355,107	826A
1,343,153	549A	1,344,950	605A	1,347,358	599A	1,349,238	648A	1,350,959	662A	1,353,268	749A	1,355,107	826A
1,343,182	537A	1,344,951	606A	1,347,401	667A	1,349,265	653A	1,351,047	684A	1,353,283	749A	1,355,107	826A
1,343,224	552A	1,344,962	606A	1,347,473	590A	1,349,294	652A	1,351,089	682A	1,353,316	742A	1,355,107	826A
1,343,249	549A	1,344,977	575A	1,347,481	602A	1,349,322	661A	1,351,091	689A	1,353,358	739A	1,355,107	826A
1,343,250	549A	1,345,034	576A	1,347,501	619A	1,349,334	664A	1,351,092	686A	1,353,362	754A	1,355,107	826A
1,343,255	551A	1,345,050	576A	1,347,518	657A	1,349,362	648A	1,351,144	695A	1,353,384	745A	1,355,107	826A
1,343,260	542A	1,345,051	576A	1,347,543	651A	1,349,382	661A	1,351,151	684A	1,353,385	745A	1,355,107	826A
1,343,285	598A	1,345,077	579A	1,347,544	651A	1,349,393	651A	1,351,205	682A	1,353,419	756A	1,355,107	826A
1,343,313	549A	1,345,082	564A	1,347,567	651A	1,349,411	641A	1,351,266	684A	1,353,448	749A	1,355,107	826A
1,343,314	548A	1,345,083	563A	1,347,568	651A	1,349,417	657A	1,351,363	685A	1,353,463	740A	1,355,107	826A
1,343,315	548A	1,345,132	566A	1,347,619	627A	1,349,445	657A	1,351,385	686A	1,353,500	709A	1,355,107	826A
1,343,317	581A	1,345,133	566A	1,347,664	651A	1,349,446	657A	1,351,404	695A	1,353,521	740A	1,355,107	826A
1,343,387	552A	1,345,134	566A	1,347,713	624A	1,349,447	657A	1,351,451	694A	1,353,532	750A	1,355,107	826A
1,343,390	544A	1,345,144	564A	1,347,719	631A	1,349,497	622A	1,351,458	684A	1,353,571	740A	1,355,107	826A
1,343,400	544A	1,345,192	575A	1,347,734	663A	1,349,530	663A	1,351,460	715A	1,353,599	744A	1,355,107	826A
1,343,401	544A	1,345,253	573A	1,347,741	631A	1,349,599	682A	1,351,483	697A	1,353,638	742A	1,355,107	826A
1,343,441	545A	1,345,305	564A	1,347,796	631A	1,349,597	727A	1,351,489	689A	1,353,641	740A	1,355,107	826A
1,343,443	545A	1,345,306	564A	1,347,798	634A	1,349,598	662A	1,351,494	693A	1,353,642	743A	1,355,107	826A
1,343,446	552A	1,345,323	571A	1,347,801	624A	1,349,681	648A	1,351,495	693A	1,353,654	743A	1,355,107	826A
1,343,447	552A	1,345,341	590A	1,347,822	633A	1,349,707	689A	1,351,521	701A	1,353,685	740A	1,355,107	826A
1,343,457	598A	1,345,373	594A	1,347,823	633A	1,349,713	684A	1,351,522	701A	1,353,685	766A	1,355,107	826A
1,343,468	552A	1,345,404	590A	1,347,845	633A	1,349,714	684A	1,351,652	686A	1,353,818	725A	1,355,107	826A
1,343,469	552A	1,345,429	606A	1,347,870	663A	1,349,750	652A	1,351,672	699A	1,353,931	731A	1,355,107	826A
1,343,470	549A	1,345,430	609A	1,347,895	726A	1,349,756	656A	1,351,673	695A	1,353,937	731A	1,355,107	826A
1,343,482	538A	1,345,431	716A	1,347,896	726A	1,349,798	649A	1,351,693	699A	1,353,980	739A	1,355,107	826A
1,343,509	545A	1,345,437	732A	1,347,897	725A	1,349,802	677A	1,351,755	689A	1,353,995	727A	1,355,107	826A
1,343,575	552A	1,345,452	651A	1,347,927	631A	1,349,810	677A	1,351,801	683A	1,354,056	713A	1,355,107	826A
1,343,585	550A	1,345,476	595A	1,347,928	631A	1,349,867	651A	1,351,811	695A	1,354,057	713A	1,355,107	826A
1,343,619	549A	1,345,605	590A	1,347,940	622A	1,349,881	648A	1,351,835	725A	1,354,058	713A	1,355,107	826A
1,343,656	549A	1,345,620	563A	1,347,979	624A	1,349,899	649A	1,351,859	716A	1,354,059	713A	1,355,107	826A
1,343,662	545A	1,345,632	599A	1,348,036	648A	1,349,908	648A	1,351,865	695A	1,354,068	761A	1,355,107	826A
1,343,674	651A	1,345,656	651A	1,348,068	631A	1,349,918	662A	1,351,877	695A	1,354,081	720A	1,355,107	826A
1,343,715	582A	1,345,657	609A	1,348,099	659A	1,349,919	656A	1,351,888	703A	1,354,122	712A	1,355,107	826A
1,343,716	583A	1,345,680	637A	1,348,129	633A	1,349,947	675A	1,351,962	686A	1,354,123	728A	1,355,107	826A
1,343,725	545A	1,345,694	605A	1,348,159	619A	1,349,956	676A	1,351,977	696A	1,354,154	728A	1,355,107	826A
1,343,737	636A	1,345,695	605A	1,348,175	657A	1,349,983	676A	1,351,990	734A	1,354,223	712A	1,355,107	826A
1,343,797	557A	1,345,714	590A	1,348,227	656A	1,350,009	648A	1,352,014	687A	1,354,293	723A	1,355,107	826A
1,343,833	538A	1,345,715	590A	1,348,264									

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289,746	405A	300,051	800A	302,537	69A	301,387	13A	307,081	627A	310,174	510A	313,965	44A
290,032	28A	300,052	743A	302,543	307A	301,399	311A	307,085	602A	310,176	303A	313,986	37A
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291,229	513A	300,069	429A	302,551	15A	301,179	162A	307,111	59A	310,193	267A	314,015	6A
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297,955	519A	300,635	835A	302,584	398A	305,082	404A	307,152	288A	310,653	327A	314,124	51A
298,012	447A	300,614	59A	302,600	856A	305,083	191A	307,166	182A	310,681	393A	314,131	69A
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299,052	30A	301,684	445A	304,942	437A	305,692	273A	309,160	631A	313,413	44A	314,581	491A
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299,181	409A	301,723	406A	304,986	86A	306,304	426A	309,180	282A	313,578	29A	314,596	52A
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314,868	430A	315,941	313A	317,146	414A	318,350	449A	319,813	662A	321,870	652A	323,845	758A
314,877	281A	315,958	291A	317,165	464A	318,402	414A	319,814	662A	321,878	700A	323,865	779A
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314,943	281A	316,027	303A	317,220	479A	318,462	457A	319,893	565A	321,981	688A	323,973	785A
314,944	183A	316,028	327A	317,254	477A	318,477	649A	319,895	658A	322,019	619A	324,012	883A
314,947	322A	316,048	303A	317,267	377A	318,478	356A	319,933	568A	322,021	637A	324,029	802A
314,957	204A	316,053	302A	317,302	356A	318,503	428A	319,936	619A	322,135	668A	324,053	779A
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314,996	326A	316,111	303A	317,402	376A	318,542	498A	319,992	345A	322,192	696A	324,203	765A
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315,012	480A	316,148	408A	317,412	361A	318,654	421A	320,056	622A	322,347	728A	324,227	766A
315,016	383A	316,149	302A	317,418	419A	318,687	428A	320,065	601A	322,446	728A	324,264	783A
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315,021	206A	316,213	395A	317,448	376A	318,710	500A	320,096	576A	322,462	649A	324,280	742A
315,030	262A	316,214	447A	317,449	380A	318,766	649A	320,139	662A	322,538	719A	324,333	778A
315,035	356A	316,215	364A	317,462	419A	318,772	355A	320,149	568A	322,600	696A	324,334	778A
315,074	270A	316,216	313A	317,493	367A	318,795	464A	320,152	665A	322,610	661A	324,379	827A
315,091	258A	316,217	313A	317,502	375A	318,820	428A	320,178	629A	322,619	719A	324,433	778A
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315,317	255A	316,489	350A	317,702	406A	319,054	658A	320,620	633A	322,845	765A	324,731	744A
315,321	165A	316,491	436A	317,717	522A	319,068	654A	320,629	633A	322,895	815A	324,747	833A
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315,434	343A	316,504	396A	317,825	395A	319,332	459A	320,800	647A	323,155	757A	324,893	798A
315,464	367A	316,511	330A	317,832	491A	319,335	498A	320,807	665A	323,185	744A	324,894	779A
315,497	359A	316,516	500A	317,845	267A	319,336	498A	320,808	665A	323,193	756A	324,919	802A
315,511	323A	316,527	653A	317,874	423A	319,368	467A	320,829	663A	323,205	743A	324,920	802A
315,534	322A	316,535	366A	317,888	457A	319,379	563A	320,846	672A	323,210	761A	324,928	759A
315,536	275A	316,592	483A	317,948	514A	319,384	493A	320,856	565A	323,290	751A	324,945	788A
315,539	296A	316,593	374A	317,962	478A	319,420	517A	320,857	675A	323,295	744A	324,977	821A
315,549	335A	316,594	449A	317,963	497A	319,436	500A	320,893	638A	323,296	744A	325,002	815A
315,554	327A	316,596	405A	317,965	460A	319,450	477A	320,919	634A	323,298	765A	325,072	775A
315,558	394A	316,597	413A	317,977	395A	319,440	475A	320,981	676A	323,339	821A	325,114	831A
315,621	325A	316,604	460A	317,979	355A	319,465	491A	320,996	631A	323,344	829A	325,154	824A
315,622	334A	316,631	326A	317,998	421A	319,473	552A	321,029	659A	323,414	744A	325,155	783A
315,623	291A	316,632	437A	318,033	356A	319,476	502A	321,030	658A	323,416	748A	325,156	802A
315,647	258A	316,633	398A	318,034	396A	319,481	576A	321,119	654A	323,462	744A	325,165	814A
315,656	335A	316,677	404A	318,044	414A	319,505	558A	321,158	659A	323,469	788A	325,230	777A
315,657	295A	316,696	463A	318,134	406A	319,523	650A	321,204	656A	323,474	744A	325,244	815A
315,658	349A	316,702	356A	318,135	406A	319,524	565A	321,232	653A	323,494	743A	325,246	827A
315,659	323A	316,703	393A	318,136	404A	319,541	517A	321,264	698A	323,526	751A	325,298	821A
315,667	340A	316,720	396A	318,141	415A	319,550	439A	321,293	567A	323,588	813A	325,299	821A
315,695	326A	316,748	413A	318,145	424A	319,558	507A	321,304	815A	323,595	815A	325,307	784A
315,700	633A	316,753	418A	318,147	493A	319,559	571A	321,343	666A	323,596	802A	325,313	824A
315,711	271A	316,754	401A	318,151	459A	319,591	525A	321,376	814A	323,607	744A	325,314	787A
315,712	303A	316,757	488A	318,177	456A	319,594	541A	321,382	698A	323,626	749A	325,443	831A
315,715	288A	316,790	437A	318,180	489A	319,613	480A	321,453	654A	323,627	745A	325,444	831A
315,731	307A	316,800	413A	318,200	427A	319,634	553A	321,511	638A	323,643	751A	325,470	817A
315,754	330A	316,858	403A	318,203	490A	319,631	489A	321,524	809A	323,650	761A	325,505	831A
315,773	475A	316,901	393A	318,217	460A	319,656	652A	321,525	631A	323,653	760A	325,571	787A
315,774	366A</												

List of Journals Abstracted, with Abbreviations Used, and Addresses of Publishers and Prices.

JOURNAL.	ABBREVIATION.	ADDRESS OF PUBLISHER AND PRICE.*
Agricultural Bulletin of the Federated Malay States	Agric. Bull. F.M.S. ..	Dept. of Agriculture, Kuala Lumpur, F.M.S. \$1.5.
Agricultural Journal of India	Agric. J. India ..	W. Thacker and Co., 2, Creed Lane, London. 3s.
Agricultural Ledger ..	Agric. Ledger	Reporter on Economic Products to the Government of India. Calcutta.
Agricultural Research Institute, Pusa, Report and Bulletins	Rep. (Bull.) Agric. Res. Inst., Pusa.	Supt. Govt. Printing, Calcutta. Price varies.
Allgemeine Zeitschrift für Bierbrauerei und Malzfabrikation	Allgem. Z. Bierbrau.	Michaelerstrasse 25, Vienna XVIII/1. 150 fr. per annum.
American Journal of Pharmacy.	Amer. J. Pharm. ..	145, North Tenth Street, Philadelphia, Pa., U.S.A. 30 cents.
American Journal of Science	Amer. J. Sci.	New Haven, Conn., U.S.A. 50 cents.
Anales de la Sociedad Española de Física y Química	Anal. Soc. Espan. Fis. Quim.	D. M. T. Gil, Corredera Baja de San Pablo, num. 59, Madrid. 15 pesetas per annum.
Anales de la Sociedad Química Argentina	Anal. Soc. Quim. Argentina	1790, Lavalle, Buenos Aires. 60 c
Analyst	Analyst	Simpkin, Marshall, Hamilton, Kent and Co., Ltd., 2, 4, 6, 8, Orange Street, London, S.W. 3s.
Annalen der Chemie	Annalen	C. F. Winter'sche Verlagshandlung, Leipzig, Germany. M. 60 per annum.
Annales de Chimie	Ann. Chim.	Imprimerie Gauthier-Villars et Cie., Quai des Grands-Augustins, 55, Paris. 40 fr. per annum
Annales de Chimie Analytique	Ann. Chim. Analyt. ..	M. Crinon, 20, Boulevard Richard-Lenoir, Paris. 2 fr.
Annales des Falsifications..	Ann. Falsif.	M. Filaudeau, 42 bis, Rue de Bourgogne, Paris, VIIe. 3 fr.
Annales de l'Institut Pasteur	Ann. Inst. Pasteur ..	Masson et Cie., 120, Boulevard St. Germain, Paris, 6e. 4 fr.
Annales de la Science Agronomique Française et Étrangère	Ann. Sci. Agron. ..	Librairie Berger-Levrault, 5, rue des Beaux-Arts, Paris, 6e. 10 fr. 25.
Annali di Chimica Applicata	Annali Chim. Appl. ..	Title now altered to Giornale Chimica Industriale ed Applicata
Annali della R. Stazione Chimico Agraria Sperimentale di Roma	Ann. R. Staz. Chim. Agrar. Sperm. Roma	R. Stazione Chimico-Agraria Sperimentale di Roma, Rome.
Apotheker-Zeitung	Apoth-Zeit.	Levetzowstrasse 16b, Berlin, N.W. 87. M. 1.20.
Archiv der Pharmazie	Arch. Pharm.	Selbstverlag des Deutschen Apotheker-Vereins, Berlin, Germany. £1 18s. per annum.
Archives des Sciences Physiques et Naturelles	Arch. Sci. Phys. Nat. ..	Rue de Vieux-College, 4, Geneva, Switzerland. 6 fr.
Archivio di Farmacologia Sperimentale e Scienze Affini.	Arch. Farm. Sperm...	Via Depretis, 92, Rome. Lire 2.
Atti della Reale Accademia dei Lincei, Roma	Atti R. Accad. dei Lincei, Roma	Tipografia della R. Accademia dei Lincei, Rome. Lire 10 per annum.
Australasian Pharmaceutical Notes and News	Austr. Pharm. Notes and News	Elliott Bros., Ltd., O'Connell St., Sydney, N.S.W. 3d.
Berichte der Deutschen Chemischen Gesellschaft	Ber.	R. Friedländer und Sohn, Karlstrasse 11, Berlin, N.W. 6, Germany. £2 5s. per annum.
Berichte der Deutschen Pharmazeutischen Gesellschaft.	Ber. deuts. Pharm. Ges.	Verlag von Gebr. Bornträger, Berlin, W. 35, Schöneberger Ufer 12A, Germany. 15s. per annum.
Berichte des Ohara Instituts für landwirtschaftliche Forschungen	Ber. Ohara Inst. landw. Forsch.	Verlag der Ohara Schonokai, Kurashiki Provinz Okayama. Price varies.
Biedermann's Zentralblatt ..	Biedermann's Zentr. ..	O. Leiner, Königstrasse 26B, Leipzig, Germany. M. 36 per annum.
Biochemical Journal	Biochem. J.	Cambridge University Press, Fetter Lane, London, E.C. 14s.
Biochemische Zeitschrift ..	Biochem. Zeits. ..	Verlag von J. Springer, Berlin, W. 9, Linkstrasse 23-24. M. 48 per volume.
Blast-Furnace and Steel Plant	Blast - Furnace and Steel Plant	Thaw Building, 108, Smithfield St., Pittsburgh, U.S.A. 25 cents.
Board of Trade Journal ..	Bd. of Trade J. ..	Messrs. Wyman and Sons, Fetter Lane, London, E.C. 6d.
Bolletino Chimico Farmaceutico	Boll. Chim. Farm. ..	Via Cappuccio, 19, Milan, Italy. 60 cents.

* The price for single copies is given where these are sold separately. In other cases it is only possible to buy the Journals in question in complete volumes. For many of the German and Austrian Journals special prices are charged to foreign purchasers; the actual cost is given where possible.

JOURNAL.	ABBREVIATION.	ADDRESS OF PUBLISHER AND PRICE.
Brennstoff-Chemie	Brennstoff-Chem.	Verlag W. Girardet, Essen. M. 2.50.
Brewers' Journal	Brewers' J.	L. M. Reed, Eastcheap Buildings, Eastcheap, London, E.C. 3. 3s.
British Journal of Photography	Brit. J. Phot.	24, Wellington Street, London, W.C. 2. 4d.
Buletinul Societății de Chimie din România	Bul. Soc. Chim. România	Boulevard Carol, 32, Bucarest.
Bulletin de l'Association des Chimistes de Sucrerie et de Distillerie	Bull. Assoc. Chim. Sucr.	156, Boulevard Magenta, Paris, 10e, France. 4 fr.
Bulletin of Agricultural Intelligence and of Plant Diseases	Bull. Agric. Intell. ..	International Institute of Agriculture, Rome. 2 fr.
Bulletin of the Forest Experiment Station, Meguro, Tokyo	Bull. Forest Exp. Stat., Meguro	Bureau of Forestry, Dept. of Agriculture and Commerce, Tokyo.
Bulletin of the Institution of Mining and Metallurgy ..	Proc. Inst. Min. and Met.	1, Finsbury Circus, London, E.C.
Bulletin of the Kentucky Agricultural Experiment Station	Bull. Kentucky Agric. Exp. Stat.	University of Kentucky, Lexington, Ky., U.S.A.
Bulletin of the Rubber Growers' Association (Incorporated)	Bull. Rubber Growers' Assoc.	38, Eastcheap, London, E.C. 3.
Bulletin of the School of Mines and Metallurgy, University of Missouri	Bull. School Mines and Met., Univ. Missouri	Director of Mining Experiment Station, Rolla, Mo., U.S.A.
Bulletin de la Société de Chimie Biologique	Bull. Soc. Chim. Biol.	Dunod, Successeur de H. Dunod et E. Pinat, 47 & 49, Quai des Grands-Augustins, Paris (VIe). 3 fr.
Bulletin de la Société Chimique de Belgique	Bull. Soc. Chim. Belg.	M. J. Wauters, Palais du Midi (Galerie du Travail 7), Brussels.
Bulletin de la Société Chimique de France	Bull. Soc. Chim. ..	Masson et Cie., 120, Boulevard Saint-Germain, Paris, 6e. 35 fr. per annum.
Bulletin de la Société Française de Photographie	Bull. Soc. Franç. Phot.	51, Rue de Clichy, Paris, 9e. 2 fr.
Bulletin de la Société Industrielle du Nord de la France	Bull. Soc. Ind. Nord..	Rue de l'Hôpital Militaire 116, Lille, France.
Bulletin de la Société Industrielle de Mulhouse	Bull. Soc. Ind. Mulhouse	Berger-Levrault et Cie., 5, Rue des Beaux-Arts, Paris. 60 fr. per annum.
Bulletin de la Société d'Encouragement pour l'Industrie Nationale	Bull. Soc. d'Encour. ..	Secrétariat, Rue de Rennes 44, Paris, France. 36 fr. per annum.
Canadian Chemical Journal	Canad. Chem. J. ..	Westman, Ltd., 72, Queen Street West, Toronto. 25 cents.
Le Caoutchouc et la Gutta-Perecha	Caoutchouc et Gutta-Perecha	A. D. Cillard fils, 49, Rue des Vinaigriers, Paris, X. 36 fr. per annum.
Cellulose-Chemie	Cellulose-Chem. ..	Supplement to Papierfabrikant.
Chamber of Commerce Journal	Ch. of Comm. J. ..	Oxford Court, Cannon Street, London, E.C. 6d.
Chemical Age	Chem. Age	Bean Bros., Ltd., 8, Bouverie Street, London, E.C. 4. 6d.
Chemical and Metallurgical Engineering	Chem. and Met. Eng.	McGraw-Hill Co., 10th Avenue at 36th Street, New York. 25 cents.
Chemical News	Chem. News	E. J. Davey, 97, Shoe Lane, London, E.C. 4. 6d.
Chemical Trade Journal ..	Chem. Trade J. ..	Davis Bros., Danes Inn House, 265, Strand, London, W.C. 6d.
Chemiker-Zeitung	Chem.-Zeit.	Dr. W. Roth, Cöthen, Anhalt, Germany. £1 10s. per annum.
Chemisch Weekblad	Chem. Weekblad ..	D. B. Centen, O.Z. Voorburgwal 115, Amsterdam. 0.25 fr.
Chemische Industrie	Chem. Ind.	Weidmann'sche Buchhandlung, Zimmerstrasse, 94, Berlin, S.W., Germany. M. 50 per annum.
Chemische Umschau auf dem Gebiete der Fette, Öle, Wachse, und Harze	Chem. Umschau ..	Verlag der Chemischen Umschau, Friedrichstrasse 4, Stuttgart. M. 3 (£1 7s. 6d. per annum).
Chemisches Zentralblatt ..	Chem. Zentr.	R. Friedlander und Sohn, Karlstrasse 11, Berlin, N.W. 6, Germany. £2 per annum.
Chemist and Druggist	Chem. and Drug. ..	42, Cannon Street, London, E.C. 9d.
Chimie et Industrie	Chim. et Ind.	49, Rue des Mathurins, Paris. 10 fr.
Collegium	Collegium	K. Schorlenumer, Haltingen, Baden, Germany. 2s.
Communications of the Netherlands Government Institute for advising the Rubber Trade and Rubber Industry	Comm. Netherlands Govt. Inst. for advising Rubber Trade	
Comptes-Rendus hebdomadaires des Séances de l'Académie des Sciences	Comptes rend.	Imprimerie Gauthier-Villars, Quai des Grands Augustins 5 Paris. 140 fr. per annum.
Comptes-Rendus des Travaux de Laboratoire de Carlsberg	Comptes-rend. Trav. Lab. Carlsberg	H. Hagerup, Copenhagen. Price varies.

JOURNAL.	ABBREVIATION.	ADDRESS OF PUBLISHER AND PRICE.
Deutsche Zuckerindustrie ..	Deuts. Zuckorind. ..	Dessauerstrasse, 18, Berlin, S.W.11. M. 30 per vol.
Elektrochemische Zeitschrift	Elektrochem. Zeits. ..	Brandenburgische Buchdruckerei u. Verlagsanstalt Ges., Mühlenstrasse, 9, Berlin-Schöneberg, Germany. M. 2.25.
Engineering	Engineering	C. R. Johnson, 35 and 36, Bedford Street, Strand, London, W.C. 1s. 2d.
Engineering and Mining Journal	Eng. and Min. J. ..	McGraw-Hill Publishing Co., 10th Avenue at 36th Street, New York City. 25 cents.
Farben-Zeitung	Farben-Zeit.	Krausenstrasse 35-36, Berlin, S.W. 19. 15s. per annum.
Färber-Zeitung	Färber-Zeit.	Title now changed to Textilbericht.
Feuerungstechnik	Feuerungstechn. ..	O. Spamer, Täubscheweg 26, Leipzig-R., Germany. M. 10 per quarter.
Gas Journal	Gas J.	W. King, 11, Bolt Court, Fleet Street, London, E.C.4. 1s.
Gas- und Wasserfach ..	Gas- u. Wasserfach ..	R. Oldenbourg, Gluckstrasse, 8, Munich. £1 6s. 8d. per annum.
Gas World	Gas World	8, Bouverie Street, E.C. 4. 8d.
Gazzetta Chimica Italiana..	Gazz. Chim. Ital. ..	La Direzione della "Gazzetta Chimica," Via Tre Novembre, 154, Rome. Lire 70 per annum.
Gerber	Gerber.. ..	Clarystrasse, 4, Teplitz-Schönan. 25s. per annum.
Gesammelte Abhandlungen zur Kenntnis der Kohle	Ges. Abhandl. Kenntn. Kohle	Gebr. Borntraeger, Schöneberger Ufer, 12a, Berlin, W.35. Vols. 1 to 3, M. 80; Vol. 4, M. 85.
Giornale di Chimica Indus- triale ed Applicata	Giorn. Chim. Ind. Appl.	Via S. Paolo, 10, Milan, Italy. L. 5.
Glückauf	Glückauf	Verlag Glückauf, Essen. M. 17.50 per quarter.
Gummi-Zeitung	Gummi-Zeit.	Geschäftsstelle der "Gummi-Zeitung," Krausenstrasse 35/36, Berlin, S.W. 19, Germany. 15s. per annum.
Helvetica Chimica Acta ..	Helv. Chim. Acta ..	Georg & Co., 10, Freiestrasse, Basel. £1 5s. per annum.
Imperial Institute Bulletin..	Bull. Imp. Inst. ..	John Murray, Albemarle Street, London, W. 2s. 6d.
India-Rubber Journal ..	India-Rubber J. ..	Maclaren and Sons, 37 and 38, Shoe Lane, London, E.C. 4d.
International Sugar Journal	Int. Sugar J.	2, St. Dunstan's Hill, London, E.C. 1s. 9d.
Internationale Zeitschrift für Metallographie	Internat. Z. Metallog.	Gebr. Bornträger, Schöneberger Ufer 12a, Berlin, W.35. M. 40 per annum.
Journal of Agricultural Re- search	J. Agric. Res.	U.S. Department of Agriculture, Washington, D.C., U.S.A. Price varies.
Journal of Agricultural Science	J. Agric. Sci.	University Press, Cambridge. 10s.
Journal of the American Ceramic Society	J. Amer. Ceram. Soc.	211, Church Street, Easton, Pa., U.S.A. 60 cents.
Journal of the American Chemical Society	J. Amer. Chem. Soc.	C. L. Parsons, 1709, G. Street, N.W. Washington, D.C., U.S.A. 75 cents.
Journal of the American In- stitute of Metals	J. Amer. Inst. Metals	10, Depot Street, Concord, N.H., U.S.A. \$3.
Journal of the American Leather Chemists' Associa- tion	J. Amer. Leather Chem. Assoc.	American Leather Chemists' Assoc., Easton, Pa., U.S.A. \$12 per annum.
Journal of the Association of Official Agricultural Chem- ists	J. Assoc. Off. Agric. Chem.	Box 744, Eleventh Street Station, Washington, D.C., U.S.A. 27s. per volume.
Journal of Biological Chemis- try	J. Biol. Chem.	Rockefeller Inst. for Medical Research, Mount Royal and Guilford Avenues, Baltimore, Md., U.S.A. \$1.25.
Journal of the Board of Agri- culture	J. Bd. Agric.	Board of Agriculture and Fisheries, 3, St. James's Square, London, S.W. 6d.
Journal of the Chemical Society of London, Trans- actions	Chem. Soc. Trans. ..	Gurney and Jackson, 33, Paternoster Row, London, E.C. 7s. 6d.
Journal of the Chemical, Metallurgical, and Mining Society of South Africa	J. Chem. Met. Soc., S Africa	Old Municipal Buildings, De Villiers Street, Johannesburg 3s. 6d.
Journal de Chimie Physique	J. Chim. Phys.	Gauthier-Villars, 55, Quai des Grands Augustins, Paris. 35fr. per annum; the price of a single copy varies according to the size.
Journal of the College of Agriculture, Tokyo Impe- rial University, Japan	J. Coll. Agric., Tokyo	Director of the College of Agriculture, Tokyo Imperial Uni- versity, Japan. Price varies.
Journal of the College of Engineering, University of Tokyo	J. Coll. Eng., Tokyo	The University, Tokyo.
Journal of the College of Science, Imperial Univer- sity of Tokyo	J. Coll. Science, Tokyo	Director of the College of Science, Tokyo Imperial University, Japan. Price varies.
Journal of the Franklin In- stitute	J. Franklin Inst. ..	The Actuary, The Franklin Institute, Philadelphia, Pa. U.S.A. 60 cents.
Journal für Gasbeleuchtung und Wasserversorgung	J. Gasbeleucht. ..	Now Gas-u. Wasserfach. £1 6s. 8d. per annum.
Journal of General Physiology	J. Gen. Physiol. ..	Rockefeller Institute for Medical Research, Mount Royal and Guilford Avenues, Baltimore, Md., U.S.A. \$1.

JOURNAL.	ABBREVIATION.	ADDRESS OF PUBLISHER AND PRICE.
Journal of the Indian Institute of Science	J. Indian Inst. Sci. ..	Indian Institute of Science, Bangalore. Price varies.
Journal of Industrial and Engineering Chemistry	J. Ind. Eng. Chem. ..	C. L. Parsons, 1709, G. Street, N.W., Washington, D.C., U.S.A. 75 cents.
Journal of the Institute of Brewing	J. Inst. Brewing ..	Harrison and Sons, 45, Pall Mall, London, W. 4s.
Journal of the Institution of Mechanical Engineers	J. Inst. Mech. Eng. ..	Storey's Gate, Westminster, S.W.
Journal of the Institution of Petroleum Technologists	J. Inst. Petroleum Tech.	The Institution, 5, John Street, Adelphi, Strand, W.C. 2. 7s. 6d.
Journal für Landwirtschaft	J. Landwirts.	P. Parey, Hedemannstrasse 10 u. 11, Berlin, S.W. 11. 12s. 6d. per annum.
Journal of the Optical Society of America	J. Opt. Soc. Amer. ..	J. B. Lippincott Co., Philadelphia.
Journal of Physical Chemistry	J. Phys. Chem. ..	W. D. Bancroft, Ithaca, N.Y., U.S.A. \$4 per annum.
Journal de Pharmacie et de Chimie	J. Pharm. Chim. ..	O. Doin et fils, Place de l'Odeon, 8, Paris. £1 per annum.
Journal für praktische Chemie	J. prakt. Chem. ..	J. A. Barth, Dörreinstr. 16, Leipzig, Germany. M. 50 per vol.
Journal and Proceedings of the Royal Society of New South Wales	J. Proc. Roy. Soc. N.S.W.	5, Elizabeth Street, Sydney, N.S.W. 10s. 6d. per vol.
Journal of the Royal Society of Arts	J. Soc. Arts	G. Bell and Sons, York House, Portugal Street, London, W.C. 2 1s.
Journal of the Society of Dyers and Colourists	J. Soc. Dyers and Col.	Pearl Assurance Buildings, Market Street, Bradford, Yorks. 5s.
Journal of the Society of Glass Technology	J. Soc. Glass Technol.	Dr. W. E. S. Turner, The University, Sheffield. 10s. 6d.
Journal of the Society of Leather Trades' Chemists	J. Soc. Leather Trades' Chem.	W. R. Atkin, M.Sc., The University, Leeds. 2s.
Journal of the South African Association of Analytical Chemists	J. S. Afr. Assoc. Anal. Chem.	Box 5254, Johannesburg. 2s.
Journal of the Textile Institute	J. Text. Inst.	J. D. Athey, 16, St. Mary's Parsonage, Manchester. 3s.
Journal of the Washington Academy of Sciences	J. Wash. Acad. Sci. ..	211, Church Street, Easton, Pa., U.S.A. 25 cents; 50 cents for July, Aug. and Sept. numbers.
Keramische Rundschau ..	Keram. Runds. ..	Dreyestrasse, 4, Berlin, N.W. 21.
Kogyo-Kwagaku-Zasshi (Journal of Chemical Industry, Japan)	Kogyo-Kwagaku-Zasshi (J. Chem. Ind. Japan)	Kogyo-Kwagaku-Kwai, Iidumachi, Kojimaehi-ku, Tokyo. 5 yen. (10s. 3d.) per annum.
Kolloidchemische Beihefte ..	Kolloidchem. Beihefte.	T. Steinkopff, Residenzstrasse 12B., Dresden-Blasewitz, Germany. M. 24 per volume.
Kolloid-Zeitschrift	Kolloid-Zeits.	T. Steinkopff, Residenzstrasse 12B., Dresden-Blasewitz, Germany. M. 50 per vol.
Landwirtschaftlichen Versuchs Stationen, Die	Landw. Vers.-Stat. ..	P. Parey, Hedemannstr. 10 & 11, Berlin, S.W. 11. M. 40. per vol.
Mededeelingen van het Central Rubberstation	Mededeel. Centr. Rubberstat.	Javasche Boekhandel & Drukkerij, Weltevreden, Java.
Mededeelingen uit het Geneskundig Laboratorium te Weltevreden	Med. Genees. Lab. Weltevreden	
Memoires et Compte Rendu des Travaux de la Société des Ingénieurs Civils de France	Mem. Compt. rend. Soc. Ing. Civils de France	19, Rue Blanche, Paris. 60 fr. per annum.
Memoirs of the College of Science and Engineering, Kyoto Imperial University	Mem. Coll. Sci. and Eng. Kyoto	Maruzen Co., Ltd., Tokyo, Japan. Price varies.
Memoirs of the Department of Agriculture in India	Mem. Dept. Agric. India	W. Thacker and Co., 2, Creed Lane, London. Price varies.
Metall und Erz	Metall u. Erz	W. Knapp, Mühlweg 19, Halle on Salle, Germany. £1 10s. per annum.
Milchwirtschaftliches Zentralblatt	Milchw. Zentr.	M and H. Schaper, Hanover. M. 27 per half-year.
Mittheilungen des Institutes für Kohlenvergasung und Nebenproduktengewinnung.	Mitt. Inst. Kohlenvergasung	Karl Schweighofergasse, 10, Vienna VII. 4.50 kr.
Mittheilungen aus dem königlichen Materialprüfungsamt zu Gross-Lichterfelde West	Mitt. k. Materialprüf.	Julius Springer, Linkstrasse 23-24, Berlin, W. 9, Germany. £2 10s. per annum.
Monatshefte für Chemie und verwandte Teile anderer Wissenschaften	Monatsh. Chem. ..	A. Hölder, Rothenthurmstrasse 25, Wien I, Austr'a. Kr. 16 per annum.

JOURNAL.	ABBREVIATION.	ADDRESS OF PUBLISHER AND PRICE.
Moniteur Scientifique ..	Monit. Scient. ..	Dr. G. Quesneville, 12, rue de Buci, Faubourg St. Germain, Paris. 2 fr. 25.
Monthly Bulletin of the Canadian Mining Institute	Bull. Canad. Min. Inst.	3-504, Drummond Building, Montreal.
New Zealand Journal of Science and Technology	N.Z.J. Sci. and Technol	The Director, Dominion Museum, Wellington, N.Z. 1s. 6d.
Oesterreichische Chemiker-Zeitung	Oesterr. Chem.-Zeit. ..	Pestalozzigasse 6, Vienna 1. 6 kr.
Oesterreichische Zeitschrift für Berg- und Hüttenwesen	Oesterr. Z. Berg- u. Hüttenw.	Kohlmarkt 20, Wien I, Austria. Kr. 28 per annum.
Oil, Paint, and Drug Reporter	Oil, Paint, and Drug Rep.	100, William Street, New York City, U.S.A. 15 cents.
Paint Manufacturers' Association of the United States, Circulars	Paint Manuf. Assoc., U.S.A., Circ.	Dr. H. A. Gardner, Institute of Industrial Research, 19th and B. Streets, N.W., Washington, U.S.A.
Paper	Paper	251, West 19th Street, New York. 25 cents.
Papier-Fabrikant	Papierfabr.	Otto Elsner, Oranienstr. 140-142, Berlin, S. 42. 6s. 6d. per qr.
Perfumery and Essential Oil Record	Perf. and Ess. Oil Rec.	6, Serle Street, London, W.C. 1s. 6d.
Petroleum	Petroleum	Verlag für Fachliteratur, Courbiestrasse, 3, Berlin, W. 62. £2 per volume.
Petroleum Magazine ..	Petroleum Mag. ..	Steger Building, Chicago, U.S.A. 35 cents.
Pharmaceutical Journal ..	Pharm. J.	Newspaper Buildings, Portugal Street, London, W.C.2. 1s.
Pharmaceutisch Weekblad ..	Pharm. Weeklad ..	D. B. Centen, O.Z. Voorburgwal 115, Amsterdam. 15 fr. per annum.
Pharmazentische-Zeitung ..	Pharm.-Zeit.	Linkstrasse 23-24, Berlin, W. 9. M. 1.40.
Pharmazeutische Zentralhalle	Pharm; Zentralh. ..	T. Steinkopff, Residenzstrasse, 12B, Fresden-Blasewitz. M. 10 per quarter.
Philippine Journal of Science	Philippine J. Sci. ..	Bureau of Science, Manila, Philippine Islands. 50 cents.
Philosophical Magazine and Journal of Science	Phl. Mag.	Taylor and Francis, Red Lion Court, Fleet Street, London E.C. 5s.
Photographie Journal ..	Phot. J.	Harrison & Sons, 44-47, St. Martin's Lane, W.C.2. 2s. 6d.
Proceedings of the American Society of Civil Engineers	Proc. Amer. Soc. Civ. Eng.	33, West 39th Street, New York City, U.S.A. \$8 per annum
Proceedings of American Society for Testing Materials	Proc. Amer. Soc. Test-ing Materials	McGraw Hill Book Co., 239 W. 39th Street, New York. \$5 per annum.
Proceedings of American Wood Preservers' Association	Proc. Amer. Wood Preservers Assoc.	F. J. Angier, Mt. Royal Station, Baltimore, Md., U.S.A. \$2.50 per annum.
Proceedings of the Australasian Institute of Mining and Metallurgy	Proc. Austral. Inst. Min. Met.	48, Queen Street, Melbourne, Victoria, Australia.
Proceedings of British Foundrymen's Association	Proc. Brit. Foundrymen's Assoc.	165, Strand, W.C.
Proceedings of the Engineers Society of Western Pennsylvania	Proc. Eng. Soc. W. Pa.	Union Arcade Building, Pittsburgh, Pa., U.S.A. 50 cents.
Proceedings of the Faraday Society	Proc. Faraday Soc. ..	The Secretary, 10, Essex St., Strand, W.C.2.
Proceedings of the Institution of Civil Engineers	Proc. Inst. Civ. Eng...	The Secretary, Great George Street, London, S.W.
Proceedings of Koninklijke Akademie van Wetenschappen te Amsterdam	Proc. K. Akad. Wetensch. Amsterdam.	J. Muller, Amsterdam.
Proceedings of the Royal Society	Roy. Soc. Proc. ..	Harrison and Sons, 45, St. Martin's Lane, London, W.C. Price varies.
Proceedings and Transactions of Nova Scotian Institute of Science	Proc. and Trans. Nova Scotian Inst. Sci.	Halifax, Nova Scotia. 50 cents.
Pulp and Paper Magazine..	Pulp and Paper Mag...	Industrial and Educational Publishing Co., Ltd., Garden City Press, St. Anne de Bellevue, Canada. 15 cents.
Recueil des Travaux Chimiques de Pays-Bas	Rec. Trav. Chim.	A. W. Sijthoff, Leiden, Holland. 16 fl. per volume.
Revue Générale des Matières Colorantes	Rev. Gén. Mat. Col.	Rue de Rennes, 123, Paris, VIe, France. 5 fr.
Revue de Métallurgie ..	Rev. Mét.	5, Cité Pigalle, Paris, IXe. 7 fr. 50.
Schweizerische Chemiker-Zeitung	Schweiz. Chem.-Zeit	Rascher & Co., Rathausquai 20, Zürich 1, Switzerland. 1.50 fr.
Science Reports of Tohoku Imperial University	Sci. Reports, Tohoku Imp. Univ.	The Maruzen Co., Ltd., Tokyo, Japan. Price varies.
Scientific American	Scient. Amer.	233, Broadway, New York City, U.S.A. 10 cents.
Scientific Proceedings of the Royal Dublin Society	Scient. Proc. Roy. Dublin Soc.	Leinster House, Dublin. Price varies.

JOURNAL.	ABBREVIATION.	ADDRESS OF PUBLISHER AND PRICE.
Sitzungsberichte der Königl. Preussischen Akademie der Wissenschaften	Sitzungsber. Kgl. Preuss. Akad. Wiss.	Verlag der Königlichen Akademie der Wissenschaften, Berlin. M. 12 per annum.
Soil Science	Soil Sci.	Williams & Wilkins Co., 2419-2421, Greenmount Avenue, Baltimore, U.S.A.; Cambridge University Press, Fetter Lane, London, E.C. \$3.25 per volume.
South African Journal of Industries	S. African J. Ind. ..	Government Printer, Box 373, Pretoria. 6d.
Sprechsaal	Sprechsaal	Verlag von Müller und Schmidt, Coburg, Germany. £2 per annum.
Stahl und Eisen	Stahl u. Eisen	Breitestr. 27, Düsseldorf 74, Germany. £2 10s. per annum.
Textilbericht über Wissenschaft, Industrie, und Handel	Textilber.	Melliands Textilber., Mannheim, D6, 3. M. 200 per annum.
Transactions of the American Electrochemical Society	Trans. Amer. Elect. chem. Soc.	Lehigh University, South Bethlehem, Pa., U.S.A. \$3 per vol.
Transactions of the American Foundrymen's Association	Trans. Amer. Foundrymen's Assoc.	R. Moldenke, Watchung, N.J., U.S.A.
Transactions of the American Institute of Chemical Engineers	Tr. Amer. Inst. Chem. Eng.	D. Van Nostrand Co., New York, and F. and N. Spon, Ltd. London.
Transactions of the Ceramic Society	Trans. Ceram. Soc. ..	County Pottery Laboratory, Stoke-on-Trent, Staffordshire. 45s. per volume.
Transactions of the Institute of Metals	Trans. Inst. Metals ..	Institute of Metals, 36, Victoria Street, Westminster, London, S.W.
Transactions of the Iron and Steel Institute	Trans. Iron and Steel Inst.	28, Victoria Street, London, S.W.
Transactions of the North of England Institute of Mining and Mechanical Engineers	Tr. N. Eng. Inst. Min. and Meeh. Eng.	Newcastle-on-Tyne.
Transactions of the Royal Society of Canada	Trans. Roy. Soc. Canada	J. Hope & Son, Ottawa; B. Quaritch, London. Price 5 pence.
United States Bureau of Mines, Bulletins and Technical Papers	U.S. Bureau of Mines, Bull. & Tech. Papers	Bureau of Mines, Washington, D.C., U.S.A. Price varies.
United States Bureau of Standards, Bulletins and Technological Papers	U.S. Bureau of Standards, Bull. and Technol. Papers	Superintendent of Documents, Washington, D.C., U.S.A. Price varies.
United States Commerce Reports, Daily Consular and Trade Reports	U.S. Comm. Rept. ..	Superintendent of Documents, Washington, D.C., U.S.A. \$3.50 per annum.
United States Department of Agriculture Bulletins	Bull. U.S. Dept. Agric.	Department of Agriculture, Washington, D.C., U.S.A. Price varies.
West Indian Bulletin ..	West Ind. Bull. ..	Imperial Dept. of Agriculture, Barbados, W.I. 6d.
Wochenblatt für Papierfabrikation	Wochenbl. Papierfabr.	Güntter-Stall in Biberach a. d. Riss, Württemberg, Germany. M. 1.50.
Wochenschrift für Brauerei	Wech. Brau.	P. Parey, Hedemannstrasse 10, Berlin, S.W., Germany. £1 7s. 6d. per annum.
Zeitschrift für analytischen Chemie	Z. anal. Chem.	C. W. Kriedel's Verlag, Wiesbaden, Germany. £1 per volume.
Zeitschrift für angewandte Chemie	Z. angew. Chem.	Verlag von Otto Spamer, Nürnberger Strasse 48, Leipzig, Germany. £2 16s. per annum.
Zeitschrift für anorganischen Chemie	Z. anorg. Chem.	Verlag von L. Voss, Leipzig, Germany. £1 per volume.
Zeitschrift des Vereins der deutschen Zucker-Industrie	Z. Ver. deut. Zuckerind.	Vereins-Direktorium, Kleistrasse 32, Berlin, W. 62, Germany. £6 5s. per annum.
Zeitschrift für das gesamte Brauwesen	Z. ges. Brauw.	Verlag F. Carl, Nürnberg, Germany. 12s. 6d. per annum.
Zeitschrift für das gesamte Schiess- und Sprengstoffwesen	Z. ges. Schiess- u. Sprengstoffw.	J. F. Lehmann, Paul Heysestrasse 26, München, S.W. 2, Germany. £1 15s. per annum.
Zeitschrift für Elektrochemie und angewandte physikalische Chemie	Z. Elektrochem.	W. Knapp, Mühlweg 19, Halle a.S., Germany. £2 1s. 6d. per annum.
Zeitschrift für Farben-Industrie	Z. Farben-Ind.	Neuenburgerstrasse, 24, Berlin, S.W. 68, Germany. M. 20 per annum.
Zeitschrift für Instrumentenkunde	Z. Instrumentenk.	J. Springer, Linkstrasse 23-24, Berlin, W. 9. M. 28 per annum.
Zeitschrift für öffentliche Chemie	Z. öffentl. Chem.	A. Kell's Buchhandlung, Marktstrasse 15, Plauen i. V., Germany. 10s. 6d. per annum.
Zeitschrift für Physik. ..	Z. Physik.	F. Vieweg und Sohn, Braunschweig, Germany. M. 32 per volume.
Zeitschrift für physikalische Chemie	Z. physik. Chem.	Akademische Verlagsges., Leipzig, Germany M. 24 per volume.

JOURNAL.	ABBREVIATION.	ADDRESS OF PUBLISHER AND PRICE.
Zeitschrift für physiologische Chemie	Z. physiol. Chem. ^a ..	Verlag von K. J. Trübner, Strassburg, Germany. M. 42 per volume.
Zeitschrift für Spiritusindustrie	Z. Spiritusind. ..	P. Parey, Hedemannstrasse 10, Berlin, S.W., Germany £1 5s. per annum.
Zeitschrift für Untersuchung der Nahrungs- und Genussmittel	Z. Unters. Nahr. Genussm.	Julius Springer, Linkstrasse 23-24, Berlin, W. 9, Germany. £1 per annum.
Zeitschrift für wissenschaftliche Photographie, Photochemie, und Photochemie	Z. wiss. Phot. ..	J. A. Barth, Dörrienstr., 16, Leipzig. M. 22.4 per volume.
Zeitschrift für Zuckerindustrie der Czechoslovakischen Republik	Z. Zuckerind. Czecho-Slov.	Die Administration, Havlikovo Nam, Nr. 32, Prag II., Czecho-Slovakia. £1 per annum.

List of Errata, 1920.

No. of Journal.	Page.	Column.	Line from top.	Line from bottom.	Corrigenda.
No. 1—Jan. 15 ..	24A	1	—	38	For "Anderson" read "Andersen."
No. " 2—Jan. 31 ..	35A	1	—	28-29	Delete "(B)(C) Substitutes for celluloid, vulcanite or the like."
" " " ..	19T	2	—	—	In the "Softening" graph, curve C refers to high-grade, not to low-grade balata.
" " " ..	61A	1	12-13 and 17	—	For "anthocyanins" read "anthocyanins."
" " " ..	71A	1	4	—	For "polarising" read "depolarising."
" " " ..	74A	2	2	—	For "1,328,878" read "1,323,878."
No. 3—Feb. 16 ..	97A	2	—	—	Line 16 from top should follow line 13.
No. 4—Feb. 28 ..	170A	1	24	—	For "Manual" read "Menaul."
" " " ..	175A	2	—	4	For "Constantino" read "Constantino."
No. 5—Mar. 15 ..	66T	1	25	—	Replace by "which are relished by cattle, but it is doubtful."
" " " ..	196A	1	—	19	For "Gallet" read "Gallot."
No. 7—Apr. 15 ..	136R	2	22	—	This line should read: "Canadian trade in period Jan. 1919 to Jan. 1920. During this period Canada."
" " " ..	136R	2	34	—	Delete "in 1920."
No. 8—Apr. 30 ..	309A	2	—	24	For "Matthieu" read "Mathieu."
No. 9—May 15 ..	322A	2	20	—	For "1,322,657" read "1,332,657."
No. 11—June 15 ..	197R	1	—	—	In bottom line, for "35" read "115."
" " " ..	203R	2	—	—	In last two columns of table, for "1917-18" and "1918-19" read "1918-19" and "1919-20."
" " " ..	430A	1	—	20	For "Fieldler" read "Fiedler."
No. 12—June 30 ..	215R	2	33	—	For "J. R. McLellan" read "J. C. McLennan."
" " " ..	215R	2	—	29	For "McLellan" read "McLennan."
No. 13—July 15 ..	476A	1	25	—	For "1,324,417" read "1,340,653."
" " " ..	489A	1	—	33	For "Burdick . . . 29.1.29" read (A) 1,339,544 and (B) 1,339,545, 11.5.20. Appl. 9.8.18."
No. 14—July 31 ..	195T	1	—	41	For "all cases" read "many cases."
" " " ..	195T	2	19	—	For "quality" read "gravity."
" " " ..	195T	2	—	19	For "40%" read "50%."
" " " ..	196T	1	—	29	For "100 cb. ft." read "1000 cb. ft."
" " " ..	196T	2	34	—	After "gaseous B.Th.U." insert "This may amount to as much as 1s. per gallon of motor spirit."
" " " ..	224T	2	3-5	—	For "His experience . . . corrosion" read "Lead stored in the open became severely carbonated in a short time after heavy rains, but this was not the case with lead containing perhaps 0.5% Cu."
No. 15—Aug. 16 ..	557A	1	31	—	For "Rappel" read "Ruppel."
No. 16—Aug. 31 ..	563A	1	9	—	For "Waite" read "Wait."
No. 20—Oct. 30 ..	696A	2	7	—	For "on Schlegell" read "Von Schlegell."
No. 22—Nov. 30 ..	396R	1	—	17	For "professional" read "professorial."
" " " ..	765A	1	—	6	For "Boehninger" read "Boehringer."
No. 23—Dec. 15 ..	415R	2	—	13	For "carbonisation" read "carbonatation."
" " " ..	802A	1	7-8	—	For "Trimethyl-m-cresotic" read "Trimethylacetyl-m-cresotic."
" " " ..	804A	1	33	—	For "Z. Anal. Chem., 1920, 59, 297-302" read "Helv. Chim. Acta, 1920, 3, 652-653."
No. 24—Dec. 31 ..	432R	2	17	—	For "G. F. Morrell" read "R. S. Morrell."
" " " ..	813A	2	—	5	For "Allen" read "Allan."
" " " ..	818A	2	17	—	For "Jäcke" read "Jänecké."
" " " ..	830A	2	17	—	For "Basset" read "Bassett."

Journal of the Society of Chemical Industry.

Vol. XXXIX., No. 12.

JUNE 30, 1920.

Vol. XXXIX., No. 12.

INCOME AND EXPENDITURE ACCOUNT FOR YEAR ENDED 31st DECEMBER, 1919.

Corresponding figures for 1918.		EXPENDITURE.		INCOME.		Corresponding figures for 1918.	
£	s. d.	£	s. d.	£	s. d.	£	s. d.
To Journal—Editorial and Sundry Expenses—							
575	0 0	Editor's Salary	575	0 0	By Entrance Fees	565	19 0
83	0 0	Do. Expenses	41	18 1	.. Life Composition Fees	198	10 3
300	0 0	Do. Assistant's Salary	300	0 0	.. Annual Subscriptions	7437	17 6
486	17 11	Do. Abstractors	614	10 7	.. Journal—Advertisements	10,258	9 4
27	11 0	Foreign & Sundry Journals	88	1 7 Sales	3122	12 8
27	11 0	Insurance of Stock	75	13 0	.. Prize for Essay	13,381	2 0
512	10 0	Review—Editor's Salary	537	10 0	.. Dividends and Interest on Investments	579	19 3
435	4 2	Do. Expenses	73	6 5	.. Excess of Expenditure over Income for the year	2353	4 2
		Do. Contributors ..	401	3 0			
2426	10 1		2707	9 8			
10,147	10 4	Publishing Expenses—					
1507	10 4	General	14,904	10 7			
		Postage on Copies 1901 12 1	16,566	2 8			
14,141	19 9		19,273	12 4			
		.. Annual Reports on Applied Chemistry—					
839	5 5	Expenses	1053	7 5			
283	18 7	Leas Sales	610	6 1			
555	6 10		443	1 4			
		.. Sectional Expenses—					
25	0 0	Bristol	35	0 0			
29	0 0	Boston	60	0 0			
85	0 0	Birmingham	103	17 2			
30	0 0	Canada	45	0 0			
40	0 0	Canadian Pacific	45	0 0			
30	0 0	Edinburgh	25	0 0			
109	0 0	Glasgow	100	0 0			
178	14 11	Liverpool	350	0 0			
106	13 8	London	150	0 0			
40	0 0	Manchester	200	0 0			
150	0 0	Newcastle-on-Tyne	53	2 3			
		New York	44	11 10			
		Nottingham	60	0 0			
53	0 2	Sydney	1236	11 8			
858	8 9	Yorkshire	200	0 0			
50	0 0		315	0 0			
8	8 0	Empire Sugar Committee Grant	650	0 0			
156	4 1	Prizes for Essays	650	11 8			
615	0 0	.. Annual Meeting Expenses	613	13 6			
		.. Superannuation (late Editor and Secretary)	388	18 2			
		.. Working Expenses—	539	4 3			
650	0 0	Secretary's Salary	97	5 8			
526	18 4	Clerks in Secretary's Office	100	0 0			
428	18 2	Rent, Lighting and Cleaning	3048	13 3			
109	4 2	Stationery, Binding and Library					
750	15 0	Postages, Telephone and Sundry Expenses					
172	10 8	Back Charges					
2758	6 10	Depreciation of Furniture					
£10,113 14 3			£24,510 18 2				
			19,113 14 3				
			£24,516 18 2				

To Bank Overdraft Secured	£	s.	d.	£	s.	d.
" Sundry Creditors				4616	16	11
" Subscriptions and Entrance Fees to Society and Journal Received by Latham Research Prize Fund and Accumulated Fund				2009	8	7
" Balance December 31st, 1918				1500	13	8
Deduct: Excess of Expenditure over Income, 1919 .. £2553 4 2				341	0	6
Depreciation of Investments at this date .. 1982 4 11						
Loss on Investments sold 136 12 7						
	£	s.	d.			
				14,702	13	4
Add amount included in respect of Stock of Journals and Annual Reports				3772	1	8
				10,930	11	8
Balance December 31st, 1919				413	15	0
				11,344	6	8

By Sundry Debtors	£	s.	d.	£	s.	d.
" Stock of Journals—nominal value				619	16	6
" " Annual Reports Vols. II, & III, as estimated by the Secretary				584	0	0
Subscription in Arrears				413	15	0
Cash in hand (Secretary and Editors) ..				62	0	0
Cash on Deposit, Latham Research Prize Fund				41	7	4
Investments at market value, viz.:—				341	0	6
£ s. d.						
2502 13 11 Metropolitan Consolidated 3% Stock				1601	13	0
1000 0 0 New Zealand 3% Inscribed Stock 1945 ..				610	0	0
1277 4 8 New South Wales 3% Stock 1935				830	3	0
1050 0 0 Funding Loan 4% ..				797	18	0
600 0 0 Gas Light & Coke Co. 3% Consolidated Debenture Stock				309	0	0
1084 13 4 North British Railway Co. 3% Consolidated Lien Stock				520	12	0
900 0 0 Midland Railway Co. 2½% Perpetual Guaranteed Preferential Stock				412	16	0
738 0 0 do. do.						
1509 0 0 2½% Perp. Guar. Preference Stock				316	9	0
673 0 0 Great Eastern Railway Co. Consolidated 4% Irredeemable Guaranteed Stock				973	6	0
1000 0 0 South Eastern Railway Consolidated 4½% Preference Stock				480	18	0
200 0 0 Metropolitan Water Board 3% B. Redeemable Deb. Stock				555	0	0
1250 0 0 Nottingham Joint Station 3% Stock				105	0	0
800 0 0 Great Western Railway Co. 5% Consolidated Guaranteed Stock				1078	15	0
450 0 0 Great Northern Railway Co. 3% Debeture Stock				482	0	0
724 8 0 Nottingham and Grantham Railway Co. 4½% Consolidated Stock ..				310	10	0
834 0 2 India 3½% Stock				445	10	0
700 0 0 War Loan 3½% Stock (inscribed)				442	5	0
1800 0 0 War Loan 5% 1929-47 Bonds				602	0	0
				1647	0	0
Office Furniture				480	3	0
Less Depreciation				100	0	0
				389	3	0
				£10,911	15	4

(Depreciation = 81·2% on cost written off)

To the Members of the Society of Chemical Industry.
We have audited the above Balance Sheet, dated 31st December 1920, and find that the figures therein are correct, and that such Balance Sheet correctly sets forth the financial position of the Society at the date therein stated.
3 Frederick's Place, Gld Jewry, London, E.C. 2.
24 June, 1921.

PRICE, WATERHOUSE & CO.,
Chartered Accountants

London Section.

Meeting held at Burlington House on December 1st, 1919.

MR. JULIAN L. BAKER IN THE CHAIR

THE TECHNICAL APPLICATIONS OF ETHYL CHLORIDE.

BY ALBERT HENNING.

The manufacture of ethyl chloride dates back to 1500 A.D., and the earliest reference which can be traced appears to be that of Basil Valentine, who refers to it in his book, "Wiederholung des grossen Steines der uralten Weisen," ed. Petreus, p. 72. From the description given it is evident that the product, "Spiritus salis et vini," could not have been pure ethyl chloride, and was probably a mixture of spirit with ethyl chloride, containing about 10% of the latter. Reference to ethyl chloride was also made by Glauber (1648), who referred to the fact that spirit digested for a long time with muriatic acid "kills its sal volatile" and a "fine clear oleum vini swims to the top."

The production of ethyl chloride in the pure state was probably first achieved between 1820 and 1830, but in 1871 Genther (Zeits. für Chemie, 1871, p. 147) stated that ethyl chloride was not obtainable as a commercial product, and that for research work he found it necessary to prepare it for himself.

COMMERCIAL PRODUCTION OF ETHYL CHLORIDE.

Whilst, at first, ethyl chloride was prepared almost entirely for medical purposes, the technical application has become so extensive as to demand larger production, and before the war, in one factory alone in Germany, the production of this material was as much as 250,000 kilos. per annum, whilst in another factory the output was 80,000 kilos., and other factories were producing rather smaller quantities. The product was chiefly used for ethylation in the manufacture of dyestuffs and drugs. Further quantities were also produced for use as a refrigerant and as a solvent. In the United States there are four factories making ethyl chloride, and France and Switzerland also produce very large quantities.

Considering the low price of ethyl chloride compared with other ethylating agents, these figures are not in any sense surprising, and although the English manufacturers of ethylated products are persistently demanding ethyl sulphate and other ethylating agents, none of these can compete with ethyl chloride.

The accompanying table shows the comparative cost of various ethylating agents under normal conditions, and the approximate cost per 1 lb. of ethyl radical. From these it will be seen that as an ethylating agent ethyl chloride is the cheapest agent by about 60%, and it is necessary to examine at some length the properties of ethyl chloride, which have a bearing upon its application in manufacture, and to ascertain whether the material possesses such properties as will counterbalance the advantage of cheapness.

	Approx. cost per lb.	C.H ₅ "	Approx. cost 1 lb. C ₂ H ₅ .
--	----------------------------	-----------------------	--

	s. d.		s. d.
Ethyl chloride	1 0	45-0	2 3
Ethyl bromide	6 0	26-0	22 6
Ethyl nitrate	5 0	32-0	15 6
Ethyl sulphate	4 6	37-6	12 0
Sodium ethyl sulphate ..	1 6	19-6	6 0
Ethyl alcohol (absolute)	3 6	63-5	5 6

PROPERTIES OF ETHYL CHLORIDE.

The commonest of all misconceptions regarding ethyl chloride is that it must be stored in high-pressure cylinders, and handled substantially in the same manner as a compressed gas, such as oxygen or hydrogen. As a matter of fact, the material may be siphoned or poured by means of jugs in transferring from one vessel to another.

The scale on which ethyl chloride is manufactured in this country may be judged from the fact that a 950 gallon tank is used for storage of ethyl chloride freed from methyl.

The subjoined table shows the absolute pressure of saturated ethyl chloride vapour between -30° C. and +100° C. In order to estimate the gauge pressure which will be operative in the production of bursting stresses in containing vessels, it is necessary to deduct the pressure of the atmosphere, viz., 14·7 lb. per square inch or 76 cm. mercury column.

Temp.	Absolute Pressure. Cm. Mercury	lb. per sq. in.	Temp.	Absolute Pressure. Cm. Mercury	lb. per sq. in.
-30°C. ..	11-02 ..	2-13	35°C. ..	164-32 ..	31-77
25° ..	14-50 ..	2-80	40° ..	191-96 ..	37-11
20° ..	18-75 ..	3-63	45° ..	223-07 ..	43-18
15° ..	23-96 ..	4-63	50° ..	257-94 ..	49-87
10° ..	30-21 ..	5-84	55° ..	299-00 ..	57-80
5° ..	37-67 ..	7-28	60° ..	340-05 ..	65-74
0° ..	46-52 ..	9-00	65° ..	387-85 ..	74-98
+5° ..	56-93 ..	11-00	70° ..	440-50 ..	85-16
10° ..	70-09 ..	13-55	75° ..	498-27 ..	96-33
12-5° ..	76-00 ..	14-70	80° ..	561-40 ..	108-50
15° ..	83-26 ..	16-10	85° ..	630-16 ..	121-80
20° ..	99-62 ..	19-26	90° ..	704-75 ..	136-30
25° ..	118-42 ..	22-90	95° ..	785-39 ..	151-80
30° ..	139-90 ..	27-05	100° ..	872-28 ..	168-60

Under normal conditions of storing and handling in this country, it is extremely unlikely that the containers would be subjected to a higher temperature than +30° C., and even at this temperature the gauge pressure is only 12 lb. per square inch. It is the common practice to store samples of ethyl chloride for reference in ordinary corked bottles for a period of several years, and several Winchester bottles fitted with ordinary corks and without any other fastenings have shown practically no loss after six years' storage. It is fairly obvious, therefore, that this material should be regarded rather as a volatile liquid than as a liquefied gas, and that elaborate high pressure apparatus is wholly unnecessary. The railway regulations in this country are somewhat elastic. They provide that ethyl chloride should be sent out either in fragile glass containers, packed in cotton-wool and cardboard boxes, or in welded steel drums tested to a pressure of 66 lb. per square inch. From the pressure curve it is fairly obvious that ordinary ether drums capable of resisting a pressure, under test, of 40 lb. per square inch would be quite satisfactory, and, in fact, for ordinary factory purposes these are used in regular practice.

Although ethyl chloride is volatile, owing to its high latent heat, it evaporates only slowly when exposed. The following table shows the actual losses by evaporation of ethyl chloride from an open vessel. A "pony" glass 3½ in. deep and 2 in. diameter at

the mouth was partly filled with 115 grms. of ethyl chloride and allowed to stand in a room at 11° C. and evaporate spontaneously. The b.pt. of this sample was 9°-12° C.

Time in minutes	Gross weight Grms.	Total loss Grms.	Over successive intervals.		
			Time in minutes.	Loss Grms.	Loss per minute Grms.
Start	177.5	—	—	—	—
10	174.5	3.0	10	3	0.30
50	168.5	9.0	40	6	0.15
*150	156.5	21.0	100	12	0.12
310	135.5	42.0	160	21	0.13
360	132.0	45.5	50	3.5	0.07

* For ninety minutes during this period the vessel was kept in a warm room at 14°-15° C. on a mantelpiece directly over a gas fire, during which time it lost 1.4 grms.

After a small proportion of the bulk has evaporated the temperature of the remainder is reduced to +7° to 8° C., when further loss is comparatively slow, and the following quantitative experiment shows how the liquid may be handled in open vessels in the laboratory.

About 2650 c.c. of ethyl chloride was removed from 50 c.c. bottles and filtered through an ordinary 20-cm. paper fitted in a plain funnel. As the corks were cemented in, each one had to be dug out, with consequent mechanical loss. In spite of all factors entering to facilitate losses, the amount collected at the end of the trial was 2100 c.c. This transference took about two hours, and the loss shown is surprisingly small when it is noted that the vials were not cooled in any way, but when opened were at a temperature of 18° C. If they had been cooled by ice before opening the losses would have been negligible.

Similarly, ethyl chloride can be manipulated in ordinary laboratory apparatus without special precautions, and 10 litres of solution has often been extracted with 2 litres of ethyl chloride without the use of cooling agents or special apparatus.

It is commonly stated that ethyl chloride is very readily combustible, and in consequence an undesirable material to deal with in large quantities. Whilst it is undoubtedly inflammable, its high latent heat of vaporisation, combined with the circumstance that the mixture of ethyl chloride and air is only inflammable over a comparatively narrow range of composition renders the material no more dangerous to handle than methylated spirit or benzol. There would be no danger involved in smoking a cigarette near an open vessel containing ethyl chloride, and a lighted cigarette if dropped into a beaker full of ethyl chloride is extinguished. Mention may also be made that liquid ethyl chloride is not ignited by the sparks on the commutator of electrical machines. A spray of the liquid projected from a leak or a small hole will not burn, except in immediate contact with a flame.

The application of ethyl chloride as a refrigerant has become extensive in America, and the experience with the material for these purposes has been such that the National Board of Fire Underwriters has approved its use. This decision was reached after a special investigation had been carried out by Professor Jacobus, of the Stevens Institute of Technology, who was appointed by the Board of Underwriters as their independent expert (see "Ice and Refrigeration," December, 1908, p. 311).

The Navy Department of the United States was furnished with ethyl chloride for making tests on their Proving Ground. The report of these tests was decisive. In fact, the experts at the Proving Ground were inclined to ridicule the idea of a chemical of this kind being sent to them for test of explosive force. We have had 200 cubic feet of ethyl chloride vapour accidentally liberated in an enclosed space within 20 feet of two lighted boilers without accident.

Ethyl chloride, unlike other chlorinated solvents of similar type, is entirely without poisonous properties, and in this respect it possesses a very marked advantage over ethyl sulphate, which is a most treacherous material in works practice. The application of ethyl chloride as an anæsthetic has, however, given rise to the impression that soporific effects are liable to be produced in workpeople handling it, but this is an entirely mistaken impression. For the production of anæsthesia, the application must be made under very definite and carefully regulated conditions, and by means of an airtight face-piece or mask. A small leakage of air past the face-piece is sufficient to prevent anæsthesia, and, as a result, complaints are occasionally received that material supplied for anæsthetic purposes had been found unsatisfactory in practice. Such complaints are generally traceable to leakage. In the course of handling, by means of a staff of girls, hundreds of thousands of tubes, and filling these with ethyl chloride, no single case of accidental anæsthesia has ever been observed. The use of ethyl chloride as a local anæsthetic constitutes sufficient proof of this statement. A surgeon will spray ethyl chloride on to the chin or lip of the patient and yet general anæsthesia is not produced.

In the case of a staff of twenty girls sitting in one room of about 2500 cubic feet, handling ethyl chloride freely, the vapour being constantly present in the atmosphere, no complaint has ever been received; but, on the contrary, newcomers generally state that the work has a bracing effect and induces a better appetite. An open fireplace is used in this room.

Ethyl chloride holds but the very slightest trace of water, and, compared with ether, this advantage is most marked for solvent or extraction purposes. The statement that 10 parts of ethyl chloride will dissolve in 100 parts of water is often taken as meaning parts by weight. Actually this refers to volumes of ethyl chloride *gas*, which makes the solubility only 1 part in 3000 by weight. This is negligible for most purposes. It must not be taken that the best ethyl chloride on the market contains even this amount, since every care is taken to ensure the product being anhydrous and free from ether. Such a product, as stated later, can be safely used in contact with any metal without fear of corrosion.

TECHNICAL APPLICATIONS OF ETHYL CHLORIDE.

The principal applications of ethyl chloride are:—

- (1) In the synthesis of dyestuffs and drugs as an ethylating agent.
- (2) As a working substance in refrigerating machinery.
- (3) As a solvent for extraction purposes.
- (4) In research work and in laboratory practice.

In addition, ethyl chloride is, of course, used for medical purposes, but these applications do not come within the scope of this paper.

Application as an ethylating agent.

The importance of the ethyl group is not generally realised, and there is found in literature and

also in research work a persistent tendency to refer to the more expensive methyl derivatives as intermediate products. It is obvious, from the point of view of cost of raw materials alone, that a methyl derivative must be more expensive than the corresponding ethyl derivative, unless there is some special difficulty in the manufacture of the latter.

The import statistics of the United States show that the value of typical ethylated colours (including only those colours the constitution of which is known) imported before the war into the U.S. amounted to over £75,200 per annum. In addition to this amount, very considerable allowance must be made for ethylated colours included under the unclassified list, and also for a proportion of the direct colours, which are generally sold under names indicating that they are derivatives of diaminidine, but which are really derivatives of the cheaper ethoxybenzidine. For the production of these colours two outstanding processes are involved: (a) Ethylation of the amino group. (b) Ethylation of the hydroxyl group.

(a) The first type of transformation can often be produced by heating the salt of the amine in an autoclave with pure ethyl alcohol. In this reaction the salt must be a haloid salt, and therefore generally the use of an enamel-lined autoclave is necessary. In the application of other ethylating agents the difficulty experienced is, in general, the purification of the ethylating agent; and, in fact, if even comparatively small proportions of methyl compounds be present, the shade of the colour produced is considerably modified. Ethyl chloride may be used for this purpose, and has a distinct advantage in that it is a material which can be produced commercially in an exceptionally high state of purity, and whereas the ordinary commercial ethyl chloride contains a high percentage of methyl chloride, a special quality for ethylation is now available in large quantities in this country which contains less than 0.1% of methyl chloride. As to the method of ethylating amino compounds with ethyl chloride, it is, of course, impossible to generalise; but it may be stated that with suitable apparatus the reaction may be carried out in ordinary cases in about four hours, in an autoclave capable of withstanding a working pressure of about 150 lb. per square inch. The advantage is very marked when compared with the procedure necessary in the case of ethylation with ethyl alcohol. In the latter case a much more substantial autoclave is necessary, an enamel lining is essential, and the speed of operation will bear no comparison.

(b) The ethylation of hydroxy compounds is carried out by substantially the same methods, and in this case ethyl chloride is the standard agent in technical use. The process of ethylation of hydroxy compounds has received special attention as a means of increasing the fastness to alkalis of hydroxyazo colours, particularly *p*-hydroxyazo colours, in which the deficiency in fastness is very noticeable; as an example of this transformation the conversion of Brilliant Yellow into Chrysophenine may be cited. In reactions of this kind it is always necessary to use an ethylating agent, and the price of ethyl chloride has naturally rendered its application universal.

In this country, the necessity for using an autoclave with ethyl chloride has been considered a serious objection to its application, and leanings towards ethyl sulphate have been noticeable, under the impression that this material would overcome all the chemical engineering difficulties involved. In Germany, before the war, we find that the sulphate was not in anything like general use, the chloride having been easily able to hold its own. The information given below regarding the tech-

nique of ethylation with ethyl chloride will show that the product is perfectly safe and simple to handle upon the large scale, provided reasonable intelligence is exercised.

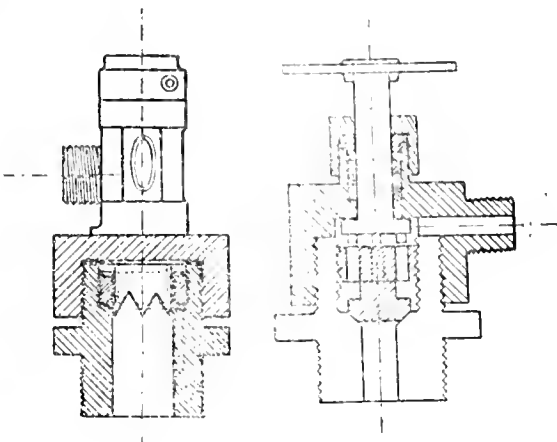


FIG. 1.

Pure ethyl chloride is now issued in drums provided with the special fastening shown in Fig. 1. By means of the devices shown, a valve may be screwed into position as soon as the drum is ready for use, this valve being provided with a serrated edge, which cuts through the lead sealing disc shown. The ethyl chloride may then be transferred either direct into the autoclave or into a measuring vessel (see Fig. 2). In this vessel a charge of ethyl chloride may be measured off, and by manipulation of the taps shown the contents transferred to an autoclave, which need not be cooled prior to the introduction of the charge. By means of these devices leakage of ethyl chloride is completely obviated, and cooling of the autoclave, such as is necessary in the ordinary methods of manipulation in using alcohol and other agents, is avoided.

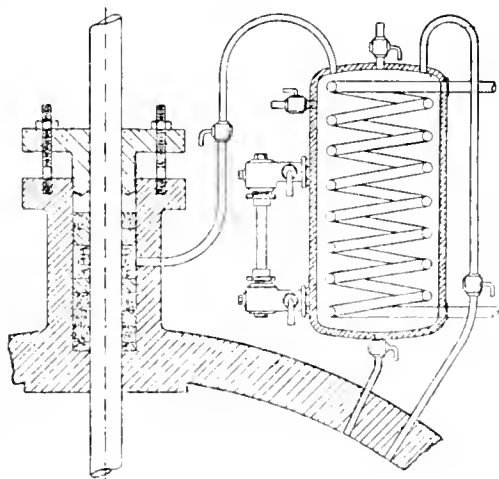


FIG. 2.

It has been stated that ethyl chloride presented unusual difficulties in autoclaves, owing to leakage through the stuffing-box. This difficulty is nearly always due to the wrong kind of packing having

been employed. Since ethyl chloride is a solvent for every kind of oil, wax, or grease, ordinary paraffined asbestos packing cannot remain in good condition for more than a few minutes. If, however, the packing be filled with fibre impregnated with glycerin the same difficulty is not experienced, as ethyl chloride does not dissolve glycerin.

An arrangement whereby leakage of ethyl chloride through the stuffing-box may be entirely counteracted consists (see Fig. 2) of a simple packing-box having double packing, the space between these being connected with the condensing vessel shown, so that the pressure on the outer packing may be reduced to practically zero, *i. e.*, to the difference in pressure between the vapour pressure of the ethyl chloride in the condensing vessel and the atmospheric pressure. In the arrangement shown the condensing vessel also serves as a filling vessel. These devices are the subject of a patent application.

For some operations it is possible to use an autoclave of the rotating type, and since nearly all the ethylations with ethyl chloride take place below 100° C., it is possible to use a simple stationary jacket type such as that shown in Fig. 3. In this case, since there is no stuffing-box under pressure, no leakage can occur. When the ethylation is finished, it will generally be found that the pressure has fallen to a low value, and that there is practically no ethyl chloride left, so the emptying of the autoclave will present no difficulties whatever.

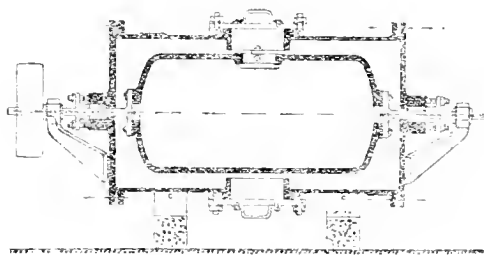


FIG. 3.

By means of the devices mentioned above ethyl chloride may be used without the slightest leakage occurring, and with far less difficulty than would be experienced in using an autoclave with alcohol. In the case of alcohol, fairly elaborate precautions are necessary in order to avoid loss, owing to the high temperature necessary to induce reactions, and in the case of ethyl sulphate the poisonous character of the material constitutes in itself a difficulty, which may not at first be realised.

The great advantage of ethyl chloride above all other ethylating agents is that, owing to its low boiling point, it can be secured commercially in a very high state of purity, and, whereas nearly all other materials for the same purposes are liable to contain considerable percentages of methyl compounds, ethyl chloride is now marketed practically free from methyl chloride. The influence of methyl chloride upon the shade of the dyestuff produced is marked, and in the case of synthetic drugs the methyl compounds are generally regarded as more toxic.

Application as a solvent.

Ethyl chloride possesses marked advantages, shared to some extent by methyl chloride, for use in the extraction of various natural products. Ethyl chloride dissolves nearly all organic compounds with great readiness, and, in the case

of comparatively volatile compounds, the low boiling point of ethyl chloride renders its application particularly advantageous. Ethyl chloride of ordinary commercial quality is anhydrous, and where it is necessary that the dry solute should be extracted its use is indicated, especially if the solute is sensitive to heating.

This matter of temperature may be of great importance where a substance is easily decomposed, and it may be instanced that nitrosamines prepared from secondary amines can be easily extracted and recovered from ethyl chloride solution without decomposition.

As to its solvent power, the following indications may be given:—Monohydroxy compounds such as phenol are soluble, as are acids like benzoic acid, but when both hydroxyl and carboxyl groups are present, as in salicylic acid, the solubility is greatly diminished. A second hydroxyl group has the same effect, resorcinol and quinol being practically insoluble. The carbinol group follows the rule for the hydroxyl groups, for, although propyl alcohol is easily soluble, glycerol is not. Introduction of successive halogen groups into a compound does not decrease the solubility, but tends to increase it. The presence of nitro groups diminishes the solubility of a compound in ethyl chloride.

Oils and fats are readily dissolved. In fact, experiments have been made successfully with flowers, herbs, seaweed, mosses, and even seeds and nuts, in extracting the oil and other substances.

In many operations in which extraction processes are used, it is necessary to freeze, usually by means of cooled brine, in order to cause crystallisation of certain constituents of the extract. When ethyl chloride is used as a solvent this procedure is quite unnecessary, because the extracted material may be obtained at a temperature as low as -15° to -20° C. by simply evaporating off the ethyl chloride under a slightly reduced pressure. The application of this principle to the isolation of pure compounds from essential oils is specially advantageous, and the ethyl chloride lends itself to recovery more easily than any other solvent. In this direction it would appear that considerable development is likely, and that where products of a high degree of purity are required it may become almost universal. For extraction purposes ethyl chloride possesses a distinct advantage over all other chlorinated solvents, in that it does not tend to become acid on storage if properly stabilised in manufacture. A sample after several years' storage under the action of light developed no trace of acid.

Application in refrigeration.

Before discussing the application of ethyl chloride refrigeration, it is necessary to review the physical data available, and in this paper will be given for the first time a complete set of figures suitable for use in the design of refrigerating machinery.

The accompanying table has been specially calculated from the meagre physical data available; it shows the vapour pressure, latent heat, specific volume, etc., for this purpose. The figures in the next table have been calculated from these to show the general order of magnitude of the essential features of various classes of refrigerating machinery, using carbon dioxide, ammonia, sulphur dioxide, methyl chloride, and ethyl chloride. The upper line of the table gives the relative cylinder dimensions based upon the assumption that piston compressors are used, operating at the same speed for a definite amount of refrigeration, and the figures in the lower portion indicate the working

Thermal properties of ethyl chloride.

Temperature.		Absolute temperature, T.	Saturated vapour. Gauge pressures.		Saturated vapour. Absolute pressures.		Saturated vapour. Specific volumes.		Saturated vapour. Density.		Saturated vapour. Specific volumes.		Liquid. Specific volumes.	
°F.	°C.		Lb. per sq. in.	Kilos. per sq. cm.	Cm. mercury.	Lb. per sq. in.	Kilos. per sq. cm.	Cm. mercury.	Cub. ft. per lb.	Lb. per cub. ft.	Kilos. per cub. m.	Cub. ft. per lb.	Cub. m. per kilo.	
—22°	—30°	243	435.7	—0.833	—64.98	2.13	0.1498	11.02	34.2	0.029	0.47	0.0163	0.00102	
—13°	—25°	248	447.7	—0.836	—61.50	2.80	0.1971	14.50	26.5	0.038	0.67	0.0164	0.00103	
—4°	—20°	253	456.7	—0.778	—57.28	3.63	0.2348	18.75	20.9	0.048	0.77	0.0164	0.00103	
—1°	—15°	258	465.7	—0.707	—52.04	4.63	0.3256	23.06	16.7	0.061	0.96	0.0167	0.00104	
—14°	—10°	263	474.7	—0.622	—45.79	5.84	0.4106	30.21	13.5	0.084	1.19	0.0169	0.00105	
—3°	—5°	268	483.7	—0.521	—39.33	7.28	0.5119	36.67	11.0	0.091	1.49	0.0169	0.00105	
32°	0°	273	492.7	—0.401	—32.45	9.00	0.6322	46.82	9.1	0.110	1.75	0.0170	0.00106	
38°	3°	278	501.7	—0.259	—23.91	11.00	0.7337	56.93	7.6	0.132	2.13	0.0172	0.00107	
41°	5°	283	510.7	—0.089	—7.26	13.55	0.8325	70.09	6.25	0.160	2.57	0.0173	0.00108	
60°	15°	288.5	519.2	0.009	0.00	16.70	1.035	83.26	5.6	0.179	2.86	0.0175	0.00109	
54°	12°	293	528.7	0.021	23.42	18.00	1.132	93.42	4.55	0.187	3.03	0.0176	0.00110	
77°	25°	298	537.7	0.076	42.42	22.90	1.354	109.62	3.90	0.220	3.58	0.0177	0.00110	
86°	30°	303	546.7	0.068	57.05	27.05	1.609	129.90	3.23	0.241	4.17	0.0178	0.00111	
95°	35°	308	555.7	0.200	83.22	31.77	2.235	164.32	2.89	0.299	4.78	0.0178	0.00111	
104°	40°	313	564.7	1.576	115.06	37.11	2.609	191.96	2.57	0.374	9.25	0.0182	0.00115	

pressures on the evaporation and compression sides of each system. From this it will be noticed that whereas ethyl chloride, for a given amount of refrigeration, requires a much larger compressor, the range of pressures required is extremely low. This pressure range is, in fact, so low that for all ordinary purposes the disadvantage of the large displacement is entirely counteracted, because instead of using an elaborate reciprocating compressor, one may employ a simple rotary pump, not very different in construction from the well-known gas exhausters.

	Carbon dioxide	Ammonia	Methyl chloride	Sulphur dioxide	Ethyl chloride
Comparative vol. displacements per unit of refrigeration,	1.00	5.77	11.6	15.1	37.0
Pressure ranges † between -10° and 20°C . Difference	58.1 27.1	8.79 2.63	4.82 1.72	3.35 1.04	0.410 1.355
	31.0	5.86	3.10	2.31	0.945

† Absolute pressures in atmospheres.

Refrigerator cars fitted with ethyl chloride machines are not by any means a recent innovation. The machine is operated from the car axle by means of a belt drive, arrangements being provided for compensation when the car passes over curved portions of the track.

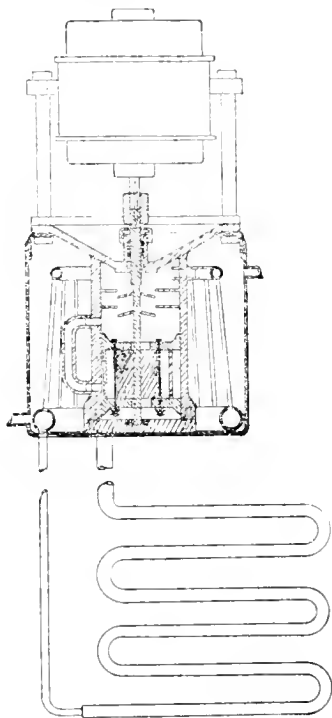


Fig. 4.

An additional advantage for small machines is that the pump may be driven directly from a small motor without the use of any reduction gear, and this cannot be said for small plant using other refrigerants. By way of giving a general idea of dimensions of ethyl chloride machines, it has been

found in practice that with cooling water at 65°F . an 18-in. diameter rotary pump having a rotor 12 in. wide, running at 150 to 300 revolutions per minute, is equivalent to a five-ton ice machine; in operating such a machine the highest pressure in the system is 15 lb. per square inch, and the pumps will even give a sufficient pressure to operate the machine using cooling water above 90°F ., and yet maintain a sufficiently low pressure on the evaporation side of the system to cool down as far as -15°C . Whilst in this country ethyl chloride is practically unknown as a refrigerant, in America this business has been developed very considerably—more than 150 plants are working—and, in fact, the U.S. naval authorities are using ethyl chloride machines on their warships. On the Hanover Bank Building of New York an ethyl chloride refrigerating plant has been working over nine years, and on the U.S.S. "Dakota" over eight years. The French naval authorities have also had several vessels fitted with similar plants. Other countries likewise have fitted their naval vessels with ethyl chloride refrigerating machines, and are increasing the number of installations of this type.

A very promising development in ethyl chloride refrigeration is found in its application to very small plant, suitable for ordinary household use, and having in general a refrigeration capacity of about 500 kilo-calories per hour. A machine of this description is readily driven directly coupled

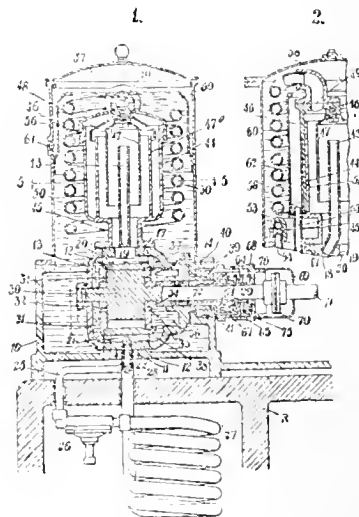


Fig. 5.

to a quarter H.P. motor; Figs. 4 and 5 show the general construction of two types of machine. In using other refrigerants in small machines, it is found that the small size of the various connecting pipes, and particularly of the expansion valves, renders regulation almost out of the question, whereas, in the case of the ethyl chloride machine, in which much larger volumes are handled for a given performance, the fittings may be made of convenient practical size, and regulation is easily within the capacity of inexperienced people. For household use, an ammonia or a carbon dioxide machine would be dangerous, owing to the high working pressure necessary, and the consequent skilled supervision required. In the case of ethyl chloride the highest pressure encountered will be about 15 lb. per square inch, and consequently the machine is safe to use. All the connecting pipes, condenser tubes, etc., may be of the lightest material, and may be made of steel or copper.

Owing to the low pressure involved, the necessity for brine circulation does not occur, and if we consider the application of ethyl chloride refrigeration to ordinary chemical purposes, it may be seen that, whereas in certain cases where corrosion of cooling coils is likely to occur, the direct circulation of ammonia or carbon dioxide would not be attractive, for example, in a cooling vessel for some corrosive liquid, ethyl chloride, by virtue of its low pressure range, might well be circulated without danger direct in the cooling coils of the vessel, and there would be no difficulty in using lead coils for this purpose in practice. In the case of ammonia or carbon dioxide this would be impossible. It may be thought, at first sight, that no advantage could accrue from the direct circulation of the working substance in such coils, but since a refrigerating machine is a reversed heat engine, the efficiency, *i.e.*, the number of calories absorbed for a given amount of power put into a machine, depends upon the value of the factor $(T_2 - T_1)/T_1$, approximately, where T_2 = absolute temperature of the condenser coils and T_1 = absolute temperature of the refrigerator coils. This is a consequence of Carnot's theorem.

Such coefficients of performance are shown in the table; it will be seen that by maintaining a temperature of, say, 20° F. it is only possible to get half the output obtained with a working temperature of -10° F.

Coefficients of performance of a perfect refrigerating machine.

Lower limit. of temperature, °F	Upper limit of temperature, ° F.					
	50°	60°	70°	80°	90°	100°
-10°	7.5	6.4	5.6	5.0	4.5	4.1
0°	9.2	7.7	6.6	5.8	5.1	4.6
10°	11.7	9.4	7.8	6.7	5.9	5.2
20°	16.0	12.0	9.6	8.0	6.8	6.0
30°	24.5	16.3	12.2	9.8	8.2	7.0
40°	50.0	25.0	16.7	12.5	10.0	8.3

Now, in order to maintain a temperature of 20° F. by brine circulation it would be necessary to use brine which had been cooled before pumping into the cooling vessel at 10° F., or possibly even 0° F., and it is obvious, therefore, that the heat is being absorbed under conditions which do not compare favourably with the direct circulation system. No doubt for some purposes direct circulation of ammonia or carbon dioxide would be possible, but in chemical works, where corrosion is likely to occur and liability to disaster must be avoided, the properties of ethyl chloride render its application worthy of serious consideration. It is noteworthy that the direct circulation of ethyl chloride may be made in coils or jackets constructed in acid-resisting materials which are deficient in mechanical strength.

Ethyl chloride for laboratory use and in research work.

Few efforts have formerly been made to supply ethyl chloride in convenient packages for laboratory purposes. It is now, however, issued in such a manner as to be generally available for research work, and it is hoped that greater confidence in using this material will be the result. Ethyl bromide, iodide, and sulphate are expensive materials for use as laboratory reagents, and the results obtained with them are not representative of good technical practice.

The application of ethyl chloride in the laboratory as a solvent possesses many favourable

features, and for laboratory purposes in which small quantities are required at a time, ethyl chloride is now issued in glass siphons similar to the well-known sulphur dioxide siphon, and in cheap and handy metal cylinders. These inexpensive containers are most convenient for laboratory work, as the piece of lead tubing may be cut off and a rubber fitted with a screw clip fixed over the open end. These kind are not liable to breakage as are glass containers, and are inexpensive in every way.

For experimental purposes in ethylation, ethyl chloride is now issued in hermetically sealed tubes containing 2, 5, or 7 c.c. of ethyl chloride; and the 7 c.c. tubes contain 0.1 gm.-mol. of pure ethyl chloride. These tubes may be dropped into an autoclave unbroken, and when the temperature rises they will burst, or the capillary tubes may be broken off before they are put in the autoclave, provided the tubes are cooled slightly before this is done. By means of these special tubes an experimental ethylation, or several simultaneous experiments, may be conveniently carried out, very much after the same fashion as analytical work is now often done with compressed tablets of the various reagents.

The low boiling point of ethyl chloride makes it a very convenient cooling agent for laboratory work. When allowed to emerge through a capillary jet, or an injection needle, it is capable of producing a very low temperature, and small objects may readily be reduced to as low as -30° C. For cooling small quantities of reaction mixtures, such as those obtained in diazotising, or inducing crystallisation, or in the preservation of biological specimens, the laboratory vessels containing ethyl chloride will be found convenient.

The use of ethyl chloride in hygrometers is also convenient, as the current of air which is necessary in the case of ether is not required, and simple and accurate determinations may be made without fitting up a complicated apparatus.

In conclusion, it may be mentioned that for medical application it still continues in considerable demand. Its use as a medium for spraying-on drugs in dermatological and surgical work is also becoming popular. Ethyl chloride containing 7% of phenol has been used successfully against vermin, and for killing mosquitoes and preventing bites.

At the commencement of the reading of the paper two glasses filled with ethyl chloride were exhibited on the table; at the end of the meeting the volume of the liquid was found to be practically unaltered, although the temperature was 65° F. (18° C.), which is considerably above its b. pt. (12.5° C.).

DISCUSSION.

The CHAIRMAN asked how the cost of ethyl chloride as a refrigerant compared with that of other materials.

Captain L. M. NASH asked whether oil or fat extracted by ethyl chloride was in a pure state or whether other substances were extracted. Was the solvent more in the nature of ether or petroleum spirit in regard to the extraction of matter other than fat?

The AUTHOR said there were other by-products as well, but they could be got rid of. He could not compare it with petroleum spirit because he had made no comparative tests.

Mr. S. B. TALLANTYRE said that in using ethyl chloride as an ethylating agent he believed it was usually dissolved in alcohol. Under such conditions, at 150° C. for instance, a fair amount of acidity was often developed, and this attacked iron

slightly. This appeared to show the unstability of ethyl chloride at temperatures above the normal.

Mr. HENNING said that he would ask Mr. Bishop to reply to the question as to the comparative cost of the ethyl chloride and the ammonia processes of refrigeration. There were probably 200 or 300 ethyl chloride machines at work, but they did not enable a very exhaustive statement to be made as to comparative cost. Ethyl chloride could be used instead of ether in connection with crystallisation.

Mr. C. BISHOP said that in his experience the capital cost of ethyl chloride plants was from 5 to 7½% more than that of carbon dioxide plants. These plants, however, had only been built for covering a certain small range, and they probably, owing to their range of size, cost more than either ammonia, carbon dioxide, or sulphur dioxide systems, but there were the advantages of simplicity, safety and automatic operation. The cost of running the various kinds of plant was practically the same.

Communication.

THE ESTIMATION OF ANILINE IN COMMERCIAL ANILINES.

BY WILLIAM JAMES SANDERSON AND WILLIAM JACOB JONES.

WHEN the aniline content of a commercial aniline is in question, methods of determination based on the reactions of aromatic amines are not permissible, since the toluidines are among the commonest impurities. Again, the boiling points of the substances present in commercial anilines are so near together that a method based on separation by fractional distillation is precluded. A method depending on the relation between the freezing point of the sample and its aniline content may, however, be applied, and such a method is described in this paper.

of the three phases, solid sodium hydroxide, saturated aqueous sodium hydroxide solution, and the aniline holding in solution a definite amount of water, comes to equilibrium, and the aniline layer, on separation from the other two phases, freezes at a definite temperature, which is dependent only on the aniline content of the sample under examination. For example, pure aniline, after having been subjected to this treatment, still holds in solution an amount of water which causes it to freeze at -6.30°C ., namely, 0.30° lower than the freezing point of pure, dry aniline.

Method.—To the aniline to be examined about 3% of its weight of water is added, and about 20% of solid sodium hydroxide. The mixture is allowed to stand with occasional shaking for 12 hours in a stoppered vessel. At the end of that time the aniline is separated and the freezing point determined by the usual Beckmann method. The thermometer used should be graduated to fifths or tenths of a degree and should be tested by means of melting ice (0°) and of freezing, pure, dry aniline (-6.00°), prepared as later described. The temperature will first fall and then suddenly rise. The highest temperature then attained is taken as the freezing-point, $t^{\circ}\text{C}$., of the aniline. If there be x parts by weight of aniline per 100 parts of organic matter in the aniline under examination, and if the chief impurity in the aniline is nitrobenzene, as, for example, in "aniline oils for blue," then $x = 113.3 + 2.11t$. If the chief impurities in the aniline are the toluidines, as, for example, in "aniline oils for red," $x = 110.7 + 1.70t$.

When the freezing point is below -10°C . it is advisable to mix a known weight or volume of the aniline with a suitable amount of purified aniline of known freezing point, so that the freezing point of the mixture comes above -10°C . Simple calculation then gives the percentage of aniline in the original aniline oil.

Purification of aniline.—The aniline used in the present work had been purified by repeated recrystallisation from itself, followed by dehydration over sodium hydroxide, and by final distillation. The freezing point of the aniline thus purified and dried was -6.00°C . on the international gas-thermometer scale. Ampola and Rimatori (*Gazzetta*, 1897, 27, A, 35), give -5.96°C .

Freezing points of dry, binary mixtures of aniline with :

Weight % of aniline	Benzene	Phenylhydroxylamine	Nitrosobenzene	<i>p</i> -Aminophenol	Nitrobenzene	<i>o</i> -Toluidine	<i>p</i> -Toluidine	<i>m</i> -Phenylenediamine	Nyldine
100	-6.00°	-6.00°	-6.00°	-6.00°	-6.00°	-6.00°	-6.00°	-6.00°	-6.00°
99	-7.18°	-6.47°	-6.58°	-6.58°	-6.53°	-6.66°	-6.65°	-6.60°	-7.00°
98	-8.00°	-6.94°	-7.16°	-7.16°	-7.03°	-7.18°	-7.25°	-7.18°	-7.75°
97	-8.70°	-7.41°	-7.74°	-7.74°	-7.50°	-7.75°	-7.85°	-7.75°	-8.48°
96	-9.30°	—	—	—	-7.97°	-8.33°	-8.45°	—	-9.00°
95	—	—	—	—	-8.43°	-8.95°	-9.05°	—	—
94	—	—	—	—	-8.85°	-9.53°	-9.63°	—	—
93	—	—	—	—	-9.27°	-10.11°	—	—	—

Owing to the solubility of water in aniline it is necessary, before determining the freezing point to bring the aniline to a definite state of dehydration or of hydration. In the case of impure anilines it is not permissible to effect complete dehydration by distillation, since part of the least volatile impurities would remain in the undistilled residue. It has, however, been found that the aniline may be brought to a fairly definite state of hydration by the following treatment. The aniline is just saturated with water, and about one-fifth of its weight of solid sodium hydroxide added to it. On being allowed to stand for twelve hours or longer, with occasional shaking, the resulting system consisting

Freezing points of aniline mixtures.—Mixtures containing pure dry aniline and the substances, carefully purified and dried, which are commonly associated with it in commercial anilines, were prepared, and their freezing points taken. The table, which shows the results obtained with binary mixtures, gives the percentage by weight of aniline present for various freezing points. Thus, a dry mixture consisting of 95% of aniline and 5% of nitrobenzene freezes at -8.43°C . It was found, as with pure aniline, that the freezing points of the mixtures, after they had been subjected to the treatment with water and sodium hydroxide already described, were in each case lowered by 0.3°C .

American Section.

Meeting held at Chemists' Club on November 21, 1919.

MR. C. E. SHOLES IN THE CHAIR.

INDUSTRIAL USES FOR THE SHARK AND PORPOISE.

BY DR. ALLEN ROGERS.

For several years the author has been interested in the utilisation of shark and porpoise skins for leather manufacture and has investigated the methods of handling this class of raw material. Through this work he became acquainted with Mr. Alfred Ehrenreich, President of the Ocean Leather Company, who has also spent many years in the study of this subject, and through whose efforts the establishment of an entirely new industry has been made possible.

During several recent visits to the Everglades of Florida and to Morehead City, North Carolina, the shark and porpoise have been studied in their native haunts, and the fact demonstrated beyond a doubt that it is possible to handle these animals in a commercial manner. As a result the Ocean Leather Company has now in operation at Morehead City a modern plant where the hides are removed and salted. In addition, at this plant the livers are rendered for their oil and the flesh converted into a high-grade fertiliser stock. At Sanabal Island, Florida, another factory is in the process of construction which will be a duplicate of the one at Morehead City.

The sharks are caught in nets of 8-inch mesh, 360 yards long and about twelve feet deep, made of heavy twine. As the boat picks up the buoy, one man standing in the stern pulls in the cork line, while another hauls up the lead. When the shark is brought up a rope is placed over the tail, or, in some cases, a hook is placed in the eye. If the fish is alive it is killed by a sharp blow with an axe, and then lifted on board by means of a block and fall. In an average haul from ten to twenty sharks are taken from each net.

On arriving at the dock the fish are unloaded, and the work of dressing at once started. The first operation is to remove the fins and tail. The fins are tacked on a rack and allowed to dry in the sun; they are used by the Chinese for making soup. The fish is then cut down the back, and a circular cut made over the neck and around the gills. The skin is finally removed in such a manner that only the hole of the pectoral fins and rectal opening remain in the pelt. The flayed skins are placed in salt for 24 hours, fleshed on the beam or machine, and then pickled with salt and sulphuric acid. The pickled skins are drained and placed in bundles for shipment.

The livers are thrown into barrels, where they are allowed to remain for several days to disintegrate, and then placed in steam-jacketed kettles and heated to boiling for about one hour.

From the kettles the oil is run into washing and settling tanks, where the gurry is separated from the oil. The oil is then drawn into a second tank, again washed, and finally run to a third tank, where it is stored until ready for shipment. Exposure to the sun and rain effects more or less bleaching.

The carcase of the shark may have possibilities as a food product, but at present is being used in the manufacture of fish scrap for fertiliser stock. The carcase is thrown into the hopper of a specially-designed mill, where the flesh and bones are ground to a fine pulp, which is dried in a rotary dryer. This grade of fish scrap contains on an average 15-17% of nitrogen expressed as ammonia.

The shark skins as they arrive at the tannery at Newark, N.J., are soaked in water to remove the salt and again fleshed. The skins for bag leather are limed and bated in the usual manner and tanned in bark. The tanned skins are then treated with strong hydrochloric acid by the Kohler process to remove the shagreen, and after washing and colouring are ready for the finishing process.

The skins to be used for shoe leather are not limed or bated, but are at once treated for the removal of the shagreen. This so-called shagreen is a very hard layer which covers the whole fish; it has the appearance of very coarse sand paper and is almost impossible to remove by mechanical means. The elimination of shagreen from the raw skins is a problem which until recently has remained unsolved. A process discovered by the author which consists in soaking the skins in a salt solution of 10° B. (sp. gr. 1.074) mixed with an equal volume of hydrochloric acid. The removal of the shagreen is accomplished in about two hours. By leaving skins in this solution for a week no damage occurs. The skins are neutralised in a salt solution of 10° B., to which soda ash is added from time to time. When free from acid the stock can be tanned by any of the methods now in vogue.

For several years the Bureau of Fisheries has endeavoured to interest the public in using shark meat for edible purposes with a certain amount of success. In fact, that species of shark known as dogfish is being canned in large quantities and sold under the name of grayfish. Some of the larger sharks are sold in certain markets as deep sea sword fish. In some places shark meat as such is offered to the trade and finds a ready market. When popular prejudice has been overcome it must be admitted that shark flesh is a fair rival of the halibut, better than the cod, and equal even to the much-prized sword fish.

The Ocean Leather Company alone expect to bring their catch to 1000 sharks per day, and with an estimated catch by other fishermen of 1000 daily we would have 2000 sharks averaging 100 lb., representing a supply of edible material to the amount of 75,000,000 lb. annually. If it should prove impossible to get this material to the market in a fresh condition, it is easily cured with salt and borax, and then compares favourably with any good grade of salted fish. It may also be smoked or kippered.

During numerous trips to North Carolina and to Florida a vast amount of data has been accumulated which is of value in determining yields and cost of production. Included in these data are many individual records, some of which

may be of interest as showing the size of fish and products secured. They are as follows:—

	Length.	Girth.	Weight.	Weight of liver.	Size of skins.
	ft. ins.	ft. ins.	lbs.	lbs.	sq. ft.
Tiger shark, male	7 8	3 0	128	24	9
female†	15 4	6 8	—	—	28
Cub shark, male	10 6	4 8	337	82	14
female	11 6	5 2	360	33	18
Sand shark, female	8 2	3 8	271	28	10
Hammerhead shark male	5 8	2 3	56	6	4

†Oil rendered, 21 gallons.

It will be seen that the ratio of liver to body varies in the different species, and also that in the same species there is a difference between male and female.

Although the porpoise are not so plentiful as sharks they deserve brief mention. Porpoise travel almost exclusively in schools, and are, therefore, usually caught in the purse seine. The method consists in surrounding the school and then landing them on the beach. These fish vary in size from 3 to 30 ft. Their skin carries a heavy layer of blubber, which is split off and rendered to obtain lubricating oil. On the face of the fish is an extra heavy blubber known as junk, which, when rendered separately, yields a more valuable oil than ordinary blubber oil. In the jaw cavity is a small amount of oil, about 1 oz. to a fish, known as jaw oil. This oil is carefully rendered and brings from \$30.00 to \$80.00 per gallon.

The flesh of the porpoise is very dark in colour and resembles liver in flavour. When cooked like beef it is very tender and delicious. The same prejudice is held for porpoise as for shark, so time must be allowed for the public to become educated to this food material. In the meantime the body is utilised for making fertilisers.

Liverpool Section.

Meeting held on December 19, 1919.

DR. E. F. ARMSTRONG IN THE CHAIR.

THE MECHANISM OF THE ADDITION OF HYDROGEN TO UNSATURATED GLYCERIDES IN THE PRESENCE OF FINELY-DIVIDED NICKEL.

BY R. THOMAS, M.Sc., A.I.C.

PART I.

THE MECHANISM OF THE ADDITION OF HYDROGEN TO UNSATURATED GLYCERIDES.

The use of finely-divided reduced nickel as a catalyst in chemical reactions involving the addition of hydrogen to unsaturated organic compounds has of recent years been the subject of several technical applications. Although its suitability for such reactions was first established by Sabatier and Senderens as far back as 1897, and numerous investigations dealing with it have since been published by them and other authors, none of these, as far as I am aware, deal very exhaustively

with the subject from a dynamical standpoint, and of course, from the manufacturing point of view, the velocity of the reaction is a most important consideration. These earlier discoveries of Sabatier and Senderens have received their widest application in "fat hardening," i.e., the conversion of liquid oils or fatty acids into more valuable solid products.

The results embodied in this communication were obtained by the author in 1914 during the course of some technical investigations undertaken on the subject.

The unsaturated glyceride chosen as a starting point for these experiments was olive oil, as it was the nearest approach to a chemical entity among either vegetable or animal oils. In order to avoid the possibility of introducing traces of catalyst poisons it was considered better to use the naturally occurring product, after purification, than to synthesise olein, $C_3H_5(OC_{18}H_{33}O)_3$, as synthesis involves the use of materials which are well known to be "poisons" even when occurring in very minute quantities.

Purification of the oil.

Caustic soda solution, in amount a little in excess of that required for the neutralisation of the free fatty acids present, was added to the olive oil. The liquid was separated from the soap which was thrown out, washed several times with water, and dried *in vacuo*. Analysis by the method described by Lewkowitch* showed that the liquid glyceride was composed of 93% of olein, $C_3H_5(OC_{18}H_{33}O)_3$, and 7% of linolin, $C_3H_5(OC_{18}H_{31}O)_3$. According to Ubbelohde and Goldschmidt† the percentage of linolin varies from 7% to 10% of the liquid glycerides of various samples of olive oil.

Preparation of the catalyst.

The finely-divided nickel used as catalyst in these experiments was obtained by reducing precipitated nickel hydroxide in a current of pure hydrogen at 360° C., with the usual precautions necessary to obtain a product of high catalytic activity.‡ Enough catalyst was prepared to last through the whole series of experiments, care being taken that its "activity" remained unimpaired by keeping it out of contact with air.

The hydrogen employed both for reducing the catalyst and carrying out the hydrogenation of the unsaturated glycerides was obtained by electrolysis, and was of a very high degree of purity.

Theoretical considerations.

The presence of 7% of linolin (iodine value 173) mixed with olein (iodine value 85) makes it necessary in any accurate investigation of velocity of hydrogenation to find mathematical expressions for the transformation of both glycerides, as the former is responsible for 13–14% of the total hydrogen absorption of olive oil. The presence of two sets of double bonds in linolin gives rise to three distinct possibilities, either a direct saturation to stearin or the formation of olein (or its isomer) as an intermediate product. From several considerations I am inclined to think that the saturation of a compound containing two sets of double bonds may follow one or all of the above alternative courses according to conditions, the determining factors being (a) the nature of the catalyst, (b) its amount, and (c) its activity, together with (d) the temperature at which the reaction is carried

* Lewkowitch, "Chemical Technology and Analysis of Oils, Fats, and Waxes," Vol. 1, p. 560.

† "Handbuch der Oele und Fette," Vol. 3, p. 524.

‡ Sabatier, "La Catalyse en Chimie Organique," pp. 54–55.

out. It is quite conceivable, for instance, that a large amount of catalyst and high temperature would favour direct transformation of linolin to stearin. What actually happens in normal practice is that the three reactions:—Linolin \rightarrow stearin; linolin \rightarrow olein \rightarrow stearin; and linolin \rightarrow iso-olein \rightarrow stearin, occur simultaneously, the comparative extent depending on the above-mentioned conditions. Ubbelohde and Svanoe³ conclude that the clupanodonic acid in whale oil adds on four hydrogen atoms simultaneously, forming a linolic acid without the intermediate formation of linolenic acid. In the case of cottonseed oil, however, the same authors state that the linolic acid is first transformed into iso-oleic acid. It is not intended in the present communication to enter into a fuller discussion of this phase of the subject, as it does not materially affect the immediate object in view, namely, to show as a basis for the rest of this paper, that the addition of hydrogen at constant pressure to olive oil is a unimolecular reaction. The following equations were deduced on this assumption, and the agreement between the observed values of the

where k_2 is the velocity constant of the reaction linolin + hydrogen \rightarrow stearin.

From (i) we have in the usual way $k_1 = \frac{1}{t} \log \frac{a}{a-y}$ or

$$y = a(1 - e^{-k_1 t}) \dots \dots (iii.)$$

Similarly from (ii)

$$z = b(1 - e^{-k_2 t}) \dots \dots (iv.)$$

If x be the total hydrogen absorbed at the time t , then

$$x = y + z = a(1 - e^{-k_1 t}) + b(1 - e^{-k_2 t})$$

$$\text{or } ae^{-k_1 t} + be^{-k_2 t} = (a+b) - x \dots \dots (v.)$$

This expression enables us to calculate k_1 and k_2 .

For at intervals of time t_1 and t_2 we have:—

$$ae^{-k_1 t_1} + be^{-k_2 t_1} = (a+b) - x_{t_1}$$

$$ae^{-k_1 t_2} + be^{-k_2 t_2} = (a+b) - x_{t_2} \dots (vi.)$$

Putting $e^{-k_1 t_1} = M \dots (vi.)$ and $e^{-k_2 t_1} = N \dots (vii.)$

and choosing t_1 and t_2 so that $t_2 = 2t_1$ we obtain

$$aM + bN = (a+b) - x_{t_1} \dots \dots (viii.)$$

$$aM^2 + bN^2 = (a+b) - x_{t_2} \dots \dots (ix.)$$

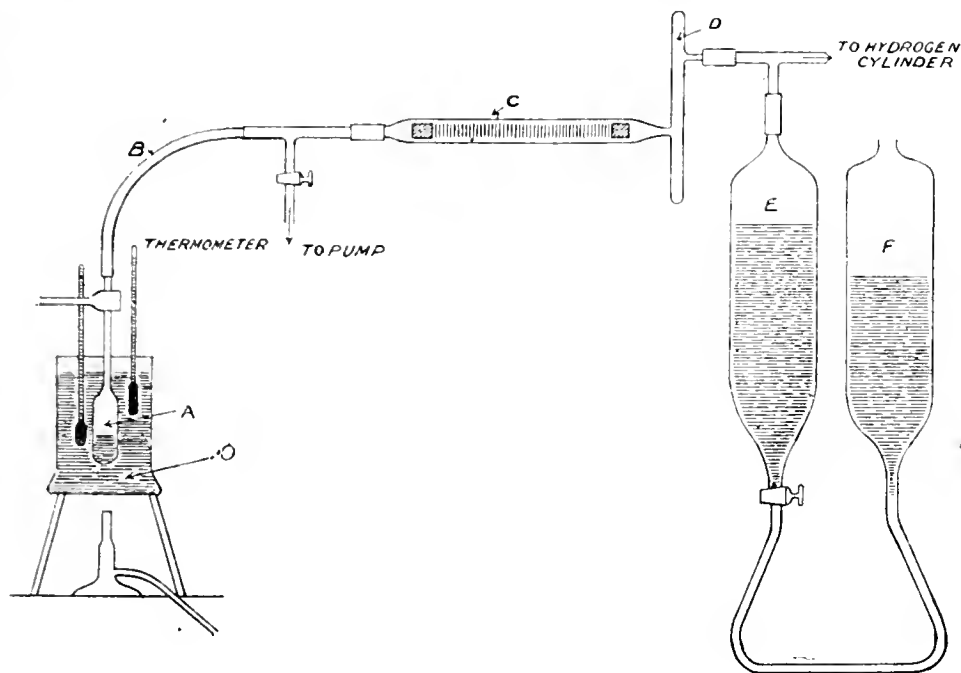


Fig. 1.

hydrogen absorption and those calculated from the equations justifies this assumption:—

If a = amount of hydrogen necessary to saturate completely the olein in olive oil.

y = amount of hydrogen absorbed by the olein in time t .

b = amount of hydrogen necessary to saturate completely the linolin in olive oil.

z = amount of hydrogen absorbed by the linolin in time t .

then $\frac{dy}{dt} = k_1(a-y) \dots \dots (i.)$

where k_1 is the velocity constant of the reaction olein + hydrogen \rightarrow stearin.

$$\frac{dz}{dt} = k_2(b-z) \dots \dots (ii.)$$

a and b are known from the iodine value of the oil and the relative proportions of olein and linolin present. x_{t_1} and x_{t_2} are determined experimentally.

Solving the equations (viii) and (ix) in the usual way we get M and N , and hence k_1 and k_2 from (vi) and (vii).

In order to test the validity of the above expression k_1 and k_2 are calculated for values of, say, $t_1 = 10$ minutes and $t_2 = 20$ minutes. With the values of k_1 and k_2 so obtained, x_t is calculated for the remaining values of t , and the values of x compared with those determined experimentally. Figure 1 shows the apparatus employed for the determination of the rate of absorption of hydrogen by olive oil.

Into A, which is conveniently made out of a 100 c.c. pipette, 10 grms. of oil and the requisite amount of nickel are introduced. B is a piece of

rubber tubing connecting to C, which contains anhydrous calcium chloride to dry the hydrogen. D contains glass wool to stop any liquid water being drawn into C during the evacuation of E. The latter is of about 1000 c.c. capacity and graduated. It is connected, as shown, to a hydrogen reservoir, and also to F containing water. The apparatus is evacuated and filled with hydrogen, these two operations being repeated several times to ensure the entire absence of air. When the apparatus is finally full of hydrogen at atmospheric pressure, A is shaken at a constant rate (400 double strokes per minute) by an electrical shaking arrangement in a bath, O, containing oil or other suitable material and maintained at a constant temperature. The absorption at atmospheric pressure after definite intervals of time is observed by reading the level of the water in E in the usual way.

The arrangement described enables the rate of absorption of hydrogen to be measured with considerable accuracy. Several repetitions of an experiment yielded results in which the maximum difference in the absorption in a given time was only 2%.

Experimental results.

The iodine value of the purified olive oil used in these experiments was 86, corresponding to a hydrogen absorption at 20° C. and 760 mm. of 815 c.c. The values of a and b in equations viii and ix are therefore 708 and 107. Two typical experiments carried out at 180° C. using 1.0% of nickel as catalyst (calculated on the oil) gave the following results:—

I.

Time (mins.).	x (observed).	x (calc.).	k_1 (olein).	k_2 (linolin).
	c.c.	c.c.		
5	67	64	—	—
10	118	118	—	—
20	203	203	0.00880	0.068
30	265	269	—	—
60	428	416	—	—
90	539	519	—	—

II.

Time (mins.).	x (observed).	x (calc.).	k_1 (olein).	k_2 (linolin).
	c.c.	c.c.		
5	66	65	—	—
10	120	120	—	—
20	204	204	0.00884	0.066
30	270	274	—	—
60	433	423	—	—
90	646	530	—	—

The observed and calculated values of x at 10 mins. and 20 mins. are, of course, identical, as the observed values in these two cases were employed for calculating k_1 and k_2 as already described. The agreement between these two sets of values for other values of t is very satisfactory.

With the same concentration of nickel at 150° C. and 120° C. the values obtained were:—

Temp. = 150° C.

Time (mins.).	x (observed).	x (calc.).	k_1 (olein).	k_2 (linolin).
	c.c.	c.c.		
5	39	38.5	—	—
10	71	71	—	—
20	132	132	0.00587	0.030
30	185	176	—	—
60	323	313	—	—
90	461	444	—	—

Temp. = 120° C.

Time (mins.).	x (observed).	x (calc.).	k_1 (olein).	k_2 (linolin).
	c.c.	c.c.		
5	25	25	—	—
10	47	47	—	—
20	89	89	0.00355	0.021
30	131	130	—	—
60	226	225	—	—
90	294	299	—	—

The results detailed above show that the addition of hydrogen at constant pressure to a mixed unsaturated glyceride such as olive oil in the presence of nickel as catalyst is a reaction of the first order. Fokin* showed that the reaction between oleic acid and hydrogen is a unimolecular re-

action, according to the equation $k = \frac{1}{t} \log \frac{a}{a-x}$

PART II.

THE FUNCTION OF THE CATALYST.

Sabatier† considers the action of the catalyst to be due to the alternate formation and decomposition of an unstable nickel hydride. Variations in the activity of the catalyst are ascribed to its power of forming different hydrides, the higher hydrides being more active than the lower. No physical or chemical evidence exists, however, which shows the possibility of the formation of a compound of nickel and hydrogen. On this theory, moreover, it would be difficult to explain either the influence of small amounts of catalyst poisons or of catalyst promoters. Armstrong and Hilditch‡ compare the action of nickel in fat hardening to that of an enzyme in the hydrolysis of glucosides.

A good deal of light can be obtained on the mechanism of the catalytic action of nickel in fat hardening from a consideration of (i) the influence of the pressure of hydrogen on the velocity of hydrogenation and (ii) the temperature coefficient of the reaction.

Influence of pressure.

This was determined by means of a simple modification of the apparatus described in figure 1, as shown in figure 2.

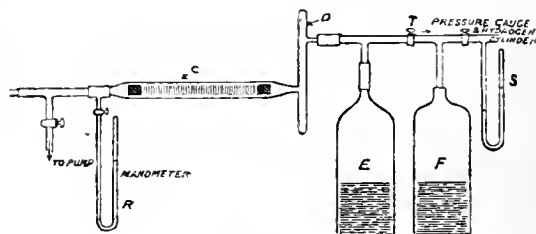


Fig. 2.

The apparatus is filled in the usual way (T being left open) with hydrogen at the required pressure from the hydrogen cylinder (the highest pressure which could be safely employed with the glass apparatus was 2 atmospheres). The pressure is indicated on the closed manometers, R and S, as

* Fokin, J. Russ. Phys.-Chem. Soc., 1908, 40, 276.

† "Die Hydrierung durch Katalyse." "La Catalyse en Chimie Organique," p. 254.

‡ Proc. Roy. Soc., 1919, A, 96, 137; this J., 1919, 780A.

well as on a pressure gauge attached to the hydrogen cylinder. When the apparatus has been finally filled at the pressure indicated on R and S the pressure tap, T, is closed, leaving only the reservoir, F, in communication with the cylinder. During the progress of the reaction the water rises in E and falls in F. The pressure in E at any time as registered by the manometer, R, is the pressure in F (registered on S and kept constant by communication with the cylinder) minus the difference in level in the water in E and F. If the diameters of E and F are about 2½—3 inches, then the final pressure at the end of the absorption is only about 5 or 6 inches of water less than at the start. This is a very small variation on a pressure of, say, 2 atmospheres, being only about 0.75%. The results obtained at 180° C. and pressures of 1.9, 1.0, and 0.8 atmospheres were as follows:—

Pressure = 1.9 atmospheres.

Time (mins.).	x (observed).	x (calc.).	k_1 (olein).
5	139	139	—
10	223	223	0.0254
20	345	350	—
40	523	541	—
60	626	645	—

Pressure = 1 atmosphere.

Time (mins.).	x (observed).	x (calc.).	k_1 (olein).
10	106	104	—
20	182	183	—
40	306	308	0.0095
60	394	402	—
80	468	474	—

Pressure = 0.8 atmosphere.

Time (mins.).	x (observed).	x (calc.).	k_1 (olein).
10	93	94	—
20	160	150	—
40	257	248	0.0070
60	320	331	—
80	384	400	—

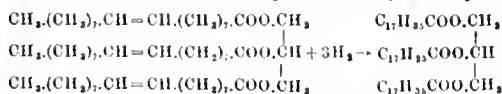
The values of x in the foregoing table have been corrected for pressure and represent the absorption at 20° C. and 760 mm. k_1 is the velocity constant for olein, calculated from equations viii and ix, the calculated values for x being derived from the same expressions. (In these remarks on the influence of pressure and likewise in the forthcoming consideration of the temperature coefficient, attention is confined to olein. k_2 (linolin) was calculated in each case, but naturally its value is not of the same degree of accuracy as that of k_1 (olein) on account of the preponderance of the latter constituent).

The next table shows the relation between the pressure of hydrogen and the velocity constant k_1 .

Pressure (atm.).	k_1 .	$k_1 \div p^{1.5}$
0.8	0.0070	0.0097
1.0	0.0095	0.0095
1.9	0.0254	0.0096

Thus the velocity constant of the reaction is proportional to $p^{1.5}$ where p is the pressure under which the hydrogen reacts. That this is in accordance with the requirements of the law of mass action the following consideration will show:—

The hydrogenation is represented by the equation



For the velocity of the reaction we have therefore:—

$$\frac{dx}{dt} = k C_{\text{olein}} \times (\text{C}_{\text{H}_2})^3$$

i.e., it is proportional to the third power of the concentration of the hydrogen actually at the seat of the reaction, i.e., in the oil or catalyst. To evaluate this we must know the relation existing between it and the pressure of hydrogen in the rest of the apparatus.

Sieverts, in a series of papers in the *Zeitschrift für physikalische Chemie* and *Zeitschrift für Elektrochemie*, has made a very exhaustive investigation of the solubility of hydrogen in various metals. He gives figures* for the absorption of hydrogen by nickel for temperatures from 200° C. to 1600° C. and pressures up to 1.5 atmospheres. At constant temperature the solubility of hydrogen in nickel is proportional to the square root of the pressure. The hydrogen absorbed is thus probably in an atomic condition for in the equilibrium $\text{H}_2 \rightleftharpoons 2\text{H}$, $\text{C}_{\text{H}} = K\sqrt{\text{C}_{\text{H}_2}}$. For C_{H_2} in the expression for the rate of hydrogenation we can therefore substitute $\sqrt{P_{\text{H}_2}}$ where P is the pressure of the hydrogen, and we obtain:—

$$\frac{dx}{dt} = k C_{\text{olein}} \times (\sqrt{P_{\text{H}_2}})^3$$

in other words, the velocity is proportional to $P^{1.5}$, which is in agreement with our experimental results.

The first function of the catalyst is therefore to absorb the hydrogen, the absorbed or occluded gas being in a highly "active" condition, in all probability in the atomic condition. The absorption occurs of course through the medium of the oil, in which hydrogen has a solubility of 5.5% by volume, at 180° C.†

Having thus seen how the hydrogen molecules become "active" it will now be of interest to consider the manner in which the olein molecule—the other participant in the reaction—becomes reactive. A consideration of the temperature-coefficient of the reaction will help to elucidate this point.

The temperature-coefficient of the reaction velocity (k_1).

Arrhenius explains the accelerating effect of temperature on the velocity of a reaction by assuming a mass action equilibrium between "active" and "inactive" molecules, an increase of temperature favouring the formation of a greater number of "active molecules."‡ This conception is criticised by Lewis and Lambell,§ who put forward arguments in favour of assuming the existence of physically active molecules rather than chemically active molecules in mass action equilibrium as suggested by Arrhenius. On either of these views, and on any reasonable theory of catalysis, it is clear that in the case of a catalysed reaction the temperature coefficient must vary to a certain extent with the concentration of the catalyst. The greater the concentration of the catalyst, the smaller should the temperature coefficient be. As long as the number of "active" molecules is small the lowering effect of catalyst

* Z. physik. Chem., 1911, 591.

† Ubbelohde and Svanoe, Z. angew. Chem., 1919, 32, 257; see this J., 1919, 870A.

‡ Z. physikal. Chem., 1889, 4, 226.

§ Chem. Soc. Trans., 1915, 197, 235.

concentration on the temperature coefficient will be a slight one, but if for any reason, such as the presence of a very large amount of catalyst, the number of "active" molecules is large compared with the number of "inactive" ones, then the accelerating effect of temperature is much smaller.

As shown in the first part of this communication, the values of k_1 (the velocity constant for olein) using 1% of nickel at 120°, 150°, and 180° were 0.00355, 0.00587, and 0.00884 respectively, which are in the ratio 1:1.66:2.5. Experiments were conducted at these temperatures using 0.5% of nickel, the velocity contents being 0.00180, 0.00305, and 0.00490 respectively, which are in the ratio 1:1.7:2.7; with 0.2% of nickel the corresponding ratios were 1:1.7:2.8. These results are summarised in the following table:—

Conc. of Ni.	$\frac{k_{150^\circ}}{k_{120^\circ}}$	$\frac{k_{180^\circ}}{k_{120^\circ}}$
1.0	1.66	2.5
0.5	1.70	2.7
0.2	1.70	2.8

Thus the temperature coefficient is slightly diminished as the concentration of the catalyst increases. This diminution is, however, only small even when the catalyst concentration is increased five-fold (from 0.2 to 1.0), and thus the total number of "active" molecules present is small in comparison with the total number of molecules.

A glance at the above figures will show that the temperature coefficient of the hydrogenation of an unsaturated glyceride like olein is a small one, an increase of temperature of 60° C. only increasing the value of the velocity constant 2.8 times, and indeed the real value of the temperature coefficient is even smaller than would at first sight appear, since at the higher temperature nickel absorbs more hydrogen, and as the velocity is proportional to the third power of the concentration of the absorbed or "active" hydrogen, an appreciable fraction of the increase in value of the velocity constant is due to this factor. In order to arrive at the real temperature coefficient of the reaction, it becomes necessary therefore to allow for this increased hydrogen concentration. For the whole range of temperatures investigated by Sieverts (*loc. cit.*) up to the melting point of nickel I find that the influence of temperature on the solubility of hydrogen in nickel can be represented with considerable accuracy by the expression:—

$$\frac{1}{m} \frac{dm}{d\theta} = \alpha - \beta \theta \quad \dots \dots (x)$$

where m is the amount of hydrogen dissolved, the temperature in degrees centigrade and α and β are constants. This equation on integration gives:—

$$\log \frac{m_2}{m_1} = \alpha (\theta_2 - \theta_1) - \frac{\beta}{2} (\theta_2^2 - \theta_1^2) \dots \dots (xi.)$$

from which it is calculated that an increase of temperature of 60° in the neighbourhood of 200° C. increases the solubility of hydrogen in nickel 1.14 to 1.15 times.

It is interesting to note in this connection that the solubility of hydrogen in oil also increases with temperature. Thus at 180° C. it is about 1.3 times as great as the solubility at 120° C.,* so that the relative increase in the solubility of hydrogen in nickel is equal to the square root of the relative increase of the solubility in oil ($\sqrt{1.3} = 1.14$), which is additional evidence in favour of the view put

forward in this paper as to the mechanism of the reaction. The increase in the value of k , therefore, on account of the increased solubility of hydrogen in nickel with temperature is 1.5 times ($= 1.15^2$) for an increase of 60° C. The true value of $k_{180^\circ}/k_{120^\circ}$ is therefore only 1.9 ($2.8 \div 1.5$), while $k_{150^\circ}/k_{120^\circ}$ is practically 1.45. These figures give a value for $(k_{t+10})/k_t$ of about 1.13. A low temperature coefficient of this order of magnitude is generally associated either with diffusion catalysis or with a photochemical reaction. On account of the very vigorous shaking of the oil with the catalyst the former possibility is excluded, and it would appear therefore that the reaction is of the nature of a photochemical reaction, due of course in this case to infra-red radiation. For the photochemical decomposition of SO_3 to SO_2 and O_2 under the influence of ultra-violet rays, Coehn and Bacher* found that for the temperature range 50°—160° C. the value of k_{t+10}/k_t was 1.2, which is of the same order as the value 1.13 found above.

The photochemical equivalent law as developed by Einstein† postulates that a photochemical reaction takes place by the absorption of quanta of radiant energy, one molecule being decomposed by one quantum E equal to $h\nu$, where h is Planck's universal constant and ν is the vibration frequency of the radiant energy absorbed. Bodenstein‡ applied this law to several photochemical reactions, and found that a small number (one to four) of quanta per molecule are absorbed to bring about the reaction (in one case the number was as high as nine). In recent years the application of Einstein's photochemical law has been extended to include radiation carried by long waves (infra-red), thermal reactions being regarded as occurring through the absorption of infra-red radiation, the energy transfer occurring in terms of quanta (in this connection see Trautz, Z. wiss. Phot., 1906, 4, 160; Richardson, Phil. Mag., March, 1914).

Lewis§ has carried out an exhaustive investigation of catalysis in homogeneous systems, and was the first to suggest that "Catalysis is essentially a radiation phenomenon."†† a suggestion which is supported by a considerable amount of experimental evidence published in a series of papers by Lewis and others during the years 1914—1918. For the effect of temperature on the velocity constant of a reaction, Marcelin** has deduced thermodynamically the expression $\frac{d \log k}{dt} = \frac{E}{RT^2}$, where E is

the energy absorbed by the molecule in excess of the average energy of all the molecules before it becomes active. He calls E the "critical energy," a more suitable term being "critical increment," ‡‡ as suggested by Lewis and Lamble (*loc. cit.*), who utilise this expression for the hydrolysis of methyl acetate, the inversion of sucrose, etc., applying at the same time the radiation theory of catalysis.

If we apply Marcelin's equation (above) to the hydrogenation of olein we obtain by integration:—

$$\log \frac{k_{180^\circ}}{k_{120^\circ}} = \frac{E}{R} \left(\frac{1}{393} - \frac{1}{453} \right) = \log 1.9;$$

hence E is equal to 3820 calories per grm.-mol. or 16.2×10^{11} ergs. If the reaction is photochemical, occurring through the absorption of one quantum

* Z. Elektrochem., 1907, 13, 545.

† Ann. Physik, 1912, 37, 832.

‡ Z. physik. Chem., 1913, 85, 329.

§ Chem. Soc. Trans., 1914, 105, 2330; 1915, 107, 233; 1916, 109, 55; 1917, 111, 389, 457, 1086; 1918, 113, 471.

** Comptes rend., 1914, 158, 116.

†† Loc. cit., 1914, 2336.

‡‡ Rice gives a mathematical conception of this in the equation $\frac{d \log k}{dt} = \frac{V_c - V_m + \frac{1}{2} RT}{RT^2}$

where V_c denotes the critical value of the internal energy of a number of molecules and V_m the mean potential energy of the molecules.

* Ubbelohde and Svanoe. Z. angew. Chem., 1919, 32, 257, 280; see this J., 1919, 870A.

($=h\nu$) of radiant energy per molecule, this figure (16.2×10^{11} ergs) should be equal to $Nh\nu$, where N is the number of molecules per gram. molecule (6.15×10^{23}), and h (Planck's constant) is equal to 7×10^{-27} . Substances containing CH_2 and CH groups show a characteristic absorption band at about 6.86μ . If, therefore, we take the value 7μ for λ , and hence

$$\frac{3 \times 10^{10}}{7 \times 10^{-4}} \text{ for } \nu \left(= \frac{\text{velocity of light}}{\text{wave length}} \right)$$

we get for $Nh\nu$ the value 1.81×10^{11} ergs. Thus E and $Nh\nu$ are in fair agreement.

Let us now consider the radiation emitted by the catalyst. Reduced nickel is a "full radiator," and we may therefore apply to it Wien's displacement law, i.e.:—

$$\lambda_{\text{max}}. T = b \text{ and } E\lambda_{\text{max}} = aT^5$$

where λ_{max} is the length of the particular waves which carry most of the radiant energy and $E\lambda_{\text{max}}$ the energy carried by the waves of length λ_{max} . Taking Lummer and Pringsheim's value for b , i.e., 2940×10^{-4} , it is seen that at the temperatures of these experiments λ_{max} is in the neighbourhood of 7μ . Thus at 150°C . we have:—

$$\lambda_{\text{max}} \times (273 + 150) = 2940 \times 10^{-4} \\ \text{or } \lambda_{\text{max}} = 7\mu.$$

It would appear therefore that the unsaturated glyceride becomes reactive by the absorption of one quantum (per molecule) of the radiant energy emitted by the catalyst, the vibration frequency being that corresponding to the maximum radiation. Or again, if this is the case, then since the maximum energy radiated is proportional to the fifth power of the absolute temperature ($E\lambda_{\text{max}} = aT^5$), then we should expect:—

$$\frac{k_{180}^\circ}{k_{120}^\circ} = \left(\frac{273 + 180}{273 + 120} \right)^5 = 2.0$$

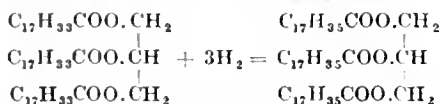
the figure found by experiment being 1.9.

The cause of the reactivity of the hydrogen has already been explained when dealing with the effect of pressure on the velocity of the reaction, as due to absorption by the catalyst, accompanied by a splitting into atoms. By extending Lewis's radiation hypothesis of homogeneous catalysis to catalysis in a heterogeneous system as shown above, the reactivity of the olein molecules can be explained. We thus have all the conditions necessary for a reaction to occur between the unsaturated glyceride and the hydrogen, both being in an active condition through the instrumentality of the catalyst.

PART III.

THE INFLUENCE OF FOREIGN GASES ON THE CATALYST (CATALYST POISONING) AND ON THE VELOCITY OF HYDROGENATION.

It has already been shown that the first function of the catalyst is to absorb hydrogen, and that the power of absorption is proportional to \sqrt{p} where p is the hydrogen pressure. Further, it was proved that the velocity of hydrogenation of olein is proportional to the third power of the "active" hydrogen in accordance with the equation:—



and therefore proportional to $p^{1.5}$. On this account alone the presence of gaseous impurity will have an appreciable retarding effect on the rate of a heterogeneous reaction of this type, since there is an accumulation of the impurity as the hydrogen is

absorbed, there being little, if any, diffusion of the gaseous impurity against the current of absorbed hydrogen. In order to prevent any possibility of backward diffusion a "trap" was inserted in the apparatus described in figure 1, between the calcium chloride tube and the reaction vessel. The "trap" consisted of a "bubbling tube" containing any suitable liquid through which the hydrogen passed on its way to the oil. The apparatus was filled with hydrogen containing a certain percentage of the gaseous impurity under investigation, and from the dimensions of the entrapped space the percentage of impurity present at any stage of the absorption could be accurately estimated.

If the volume of the "entrapped" space is V and the hydrogen contains n parts per unit volume of the gaseous impurity under consideration, then $V - nv$ is the volume of pure hydrogen originally present in this space. Put $V - nv = V_0$, then when x c.c. of hydrogen have been absorbed by the oil, the volume of pure hydrogen in the entrapped space is $V_0 - nx$. The partial pressure of the hydrogen at any time, t , in which x c.c. has been absorbed is therefore proportional to this quantity.

$$Px = \frac{1}{V} (V_0 - nx) \\ \text{when the total gas pressure is 1 atmosphere.} \\ = \frac{n}{V} (V_0/n - x) \\ = \phi (c - x) \text{ where } V_0/n = c, \text{ a constant for a given} \\ \text{initial fraction of impurity.} \\ \text{and } n/V = \phi = \text{constant.}$$

It was shown earlier that the addition of hydrogen at constant pressure to an unsaturated glyceride follows the unimolecular law, and therefore for olein we have:

$$\frac{dx}{dt} = kp^{1.5} (a - x)$$

p being the pressure of hydrogen. With the pure gas at constant pressure, this of course resolves itself into $k(a - x)$.

For hydrogen containing a gaseous impurity, therefore, if this impurity has no further action than lowering the partial pressure of the hydrogen the reaction velocity is represented by

$$\frac{dx}{dt} = k \phi (a - x) (c - x)^{1.5} \\ = k_1 (a - x) (c - x)^{1.5} \quad \dots \quad (\text{xii.})$$

where ϕ and c have the significance mentioned above. Thus we have

$$\int_0^x \frac{dx}{(a - x) (c - x)^{1.5}} = k_1 \int_0^t dt \quad (\text{xiii.})$$

In order to integrate this expression it is transformed thus:

$$\int_0^x \frac{dx}{(a - x) (c - x) \sqrt{c - x}} = k_1 \int_0^t dt$$

Substituting y for $\sqrt{c - x}$, enumerating the other factors containing x in terms of y , and resolving into partial fractions, we obtain

$$-\frac{2}{a - c} \left[\int \frac{dy}{y^2} - \int \frac{dy}{y^2 - (c - a)} \right] = k_1 \int dt$$

Integrating and re-substituting $\sqrt{c - x}$, etc., we have

$$\frac{2}{c - a} \left[-\frac{1}{\sqrt{c - x}} - \frac{1}{2\sqrt{c - a}} \cdot \log \frac{\sqrt{c - x} - \sqrt{c - a}}{\sqrt{c - x} + \sqrt{c - a}} \right]_0^x = k_1 \left[t \right]_0^t$$

and therefore

$$k_1 = \frac{1}{t} \cdot \frac{2}{c-a} \cdot \left[\frac{1}{\sqrt{c}} \frac{1}{\sqrt{c-x}} \frac{1}{2\sqrt{c-a}} \right. \\ \left. \log \frac{(\sqrt{c-x} - \sqrt{c-a})(\sqrt{c} + \sqrt{c-a})}{(\sqrt{c-x} + \sqrt{c-a})(\sqrt{c} - \sqrt{c-a})} \right] \dots (\text{xiv.})$$

This expression holds good, of course, only for pure tri-olein. When another unsaturated glyceride is present as well, such as linolin in olive oil, allowance has to be made for this. The velocity expressions for olive oil using pure hydrogen, were deduced in Part I. (equations viii. and ix.), the experimental results agreeing well with the theoretical deductions. The deduction of a similar perfectly valid expression for the transformation of olive oil in the presence of hydrogen containing foreign gases would become very complicated, while, on the other hand, treating olive oil as if the liquid glyceride consisted only of olein gives rise to a considerable error, as will be seen from the following table for olive oil and pure hydrogen:

Time (mins.).	x (absorption).	$k = \frac{1}{t} \log \frac{a}{a-x}$
10	92	0.0119
20	155	0.0105
40	254	0.0066
60	347	0.0044
80	419	0.0092

Thus k is by no means constant. The drop in the value of k is most marked in the initial stages of the reaction, as would be expected from the considerably greater velocity of transformation of linolin than of olein (see Part I.). When the absorption has reached 150 c.c. practically all the linolin has disappeared, and the fall in value of k becomes considerably smaller and more gradual. By confining our calculations therefore to absorptions above this figure, equation (xiv.) will express to a first approximation the course of the reaction with olive oil and hydrogen containing a foreign gas which acts only as a diluent. Experiments were conducted with hydrogen containing several different foreign gases. We will confine our attention to three of them, which are typical of the rest namely:—

(a) *Nitrogen*, as typical of gases which undergo no chemical action with or under the influence of either the catalyst, glyceride, or hydrogen.

(b) *Carbon monoxide*, as typical of gases which undergo a transformation with hydrogen in the presence of the catalyst.

(c) *Hydrogen sulphide* as typical of gases which unite with the catalyst.

Influence of nitrogen.

The nitrogen used was passed several times over heated copper to ensure the complete absence of oxygen, and then mixed with the hydrogen in the required proportion. The rate of absorption by olive oil was compared with that of pure hydrogen under the same conditions (1% of nickel and 180° C.). The volume of the "trapped" space was 95 c.c., i.e., as the absorption occurs the nitrogen accumulates in this space. Using hydrogen containing 3% of nitrogen, the absorption proceeded as shown in the next table, compared with pure hydrogen.

For hydrogen containing 3% nitrogen the value of c in equations (xii.), (xiii.), and (xiv.) is 3072, since $d=0.03$, $V=95$, and $V_0=92.15$. The value of a in equation (xiii.) is, of course, the total hydrogen absorption corresponding to the iodine value of the

oil (=815). On substituting the values of x found above in equation (xiv.) it was evident that the

Concentration of nickel=1.0%.

Pure hydrogen.		Hydrogen containing 3% of nitrogen, x =absorption (c.c.).		
Time (mins.).	Absorption (c.c.).	Time (mins.).	x First expt.	x Second expt.
5	50	5	48	49
10	92	10	89	87
20	155	20	147	138
40	254	40	217	205
60	347	60	259	246
80	419	80	296	280

absorption fell much more rapidly than is represented in that equation. Thus, the relative value of k dropped from 60 to 40 in the period from 20 to 80 minutes, which is a much greater fall for the same range of absorption than the error introduced by regarding olive oil as olein, and which amounts for the range of absorption under consideration to a fall of about 10%. It has been tacitly assumed by many investigators on catalysis that nitrogen is merely a diluent, but it is quite clear from the above results that it has some further action than mere dilution; (fig. 3 shows this quite clearly). All the nitrogen could be recovered at the end of the experiment, and no trace of ammonia could be detected, showing that there is no chemical action of nitrogen either with the catalyst or with the hydrogen. Its action must therefore be physical, probably diminishing the power of the catalyst for occluding hydrogen* (over and above, of course, the obvious diminution of the amount of hydrogen occluded owing to a diminution in the partial pressure of the gas). The activity of the catalyst will therefore diminish as the nitrogen accumulates, i.e., as x increases. In order to follow the course of the reaction therefore, a term must be introduced into equation (xii.) to denote the falling activity of the catalyst, i.e.,

$$\frac{dx}{dt} = k(f)b.(a-x)(c-x)^{1.5}$$

Where $(f)b$ is a function of the activity of the catalyst, this function is of the form $b-x$, since the activity diminishes as the nitrogen increases and nitrogen increases as x increases. We have therefore:—

$$\frac{dx}{dt} = k(a-x)(b-x)(c-x)^{1.5}$$

$$\text{or } \int_0^x \frac{dx}{(a-x)(b-x)(c-x)\sqrt{c-x}} = k \int_0^t dt$$

Integrating the expression in the same manner as equation (xiii.) we obtain:—

$$k = \frac{2}{(a-b)(c-b)(c-a)} \frac{1}{t} \left[\frac{b-a}{\sqrt{c}} - \frac{b-a}{\sqrt{c-x}} + \frac{c-b}{2\sqrt{c-a}} \right. \\ \left. \log \frac{(\sqrt{c-x} - \sqrt{c-a})(\sqrt{c} + \sqrt{c-a})}{(\sqrt{c-x} + \sqrt{c-a})(\sqrt{c} - \sqrt{c-a})} + \frac{a-c}{2\sqrt{c-b}} \right. \\ \left. \log \frac{(\sqrt{c-x} - \sqrt{c-b})(\sqrt{c} + \sqrt{c-b})}{(\sqrt{c-x} + \sqrt{c-b})(\sqrt{c} - \sqrt{c-b})} \right] \dots \dots (\text{xv.})$$

* Bancroft (Amer. Electrochem. Soc., Oct., 1917) comes to an analogous conclusion in an article on "Poisoning of catalytic agents." He refers to the synthetic ammonia process (Haber), the decomposition of hydrogen peroxide in presence of platinum, the Sabatier hydrogenation process, etc., and concludes that actions of "poisons (solid, liquid, or gaseous) on catalysts is due to the occurrence of an adsorption process, which interferes with the normal adsorption of the reagents."

The value of b is thus found to be 500, and for 3% of nitrogen we have the following results:—

Time (mins.).	x (absorption in c.c.).	$\frac{1}{2}k(a-b)(c-b)(c-a)$ (from equation xv.).
20	147	0.115
40	217	0.104
80	296	0.104

Similarly, with 0.5% of nickel the rate of absorption was:—

Conc. of catalyst = 0.5%.

Pure hydrogen.		Hydrogen containing 3% of nitrogen.		$\frac{1}{2}k(a-b)(c-b)(c-a)$ (from equation xv.).
Time (mins.).	Absorption (c.c.).	Time (mins.).	Absorption (c.c.).	
20	89	20	74	—
40	142	40	121	—
50	162	50	136	0.046
100	263	100	197	0.041
200	411	225	319	0.043

The value of b was thus found to be independent of the concentration of nickel, but varies with the nature of the gas and its concentration. This is the result that one would expect from the manner in which b was deduced. It was suggested that some nitrogen was adsorbed or occluded on the surface of the catalyst, and the amount so occluded would, *ceteris paribus*, be proportional to the surface, which, in its turn, is proportional to the concentration of the catalyst. When the catalyst concentration is reduced therefore the amount of occluded nitrogen is reduced in the same proportion.

Similarly, a and b are, of course, independent of the catalyst concentration, while c varies with the concentration of nitrogen in a manner easily calculated from equation (xii.).

Thus the explanation given on the foregoing page of the mechanism of the "poisoning" action of an indifferent gas is in entire accordance with the experimental evidence.

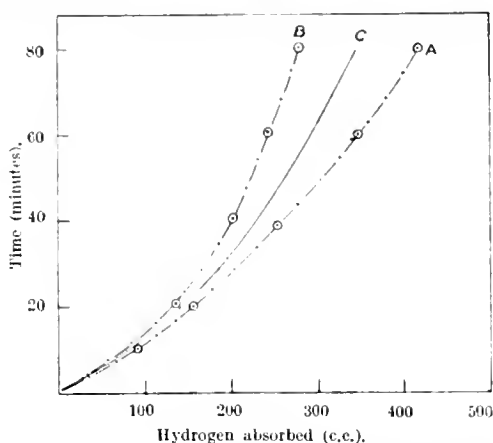
Influence of carbon monoxide.

The carbon monoxide was prepared by the action of sulphuric acid on sodium formate and was freed from carbon dioxide by washing with caustic potash solution. The "poisoning" effect of carbon monoxide is very considerably greater than that of nitrogen. In these experiments a catalyst of higher activity was employed in order to carry out the reaction in reasonable time. The comparison between the rate of absorption of hydrogen when pure and when containing various proportions of carbon monoxide will be seen from the following figures:—

Concentration of nickel = 0.5%.

Pure hydrogen.			Hydrogen containing 0.2% of CO.		Hydrogen containing 0.5% of CO.		Hydrogen containing 1.0% of CO.		Hydrogen containing 2.0% of CO.	
Time (mins.).	Absorption Observed	Calculated*	Time (mins.).	Absorption (c.c.).	Time (mins.).	Absorption (c.c.).	Time (mins.).	Absorption (c.c.).	Time (mins.).	Absorption (c.c.).
10	121	121	10	100	10	90	10	79	10	41
20	187	187	20	140	20	124	20	107	20	77
40	281	293	40	202	40	166	40	141	40	103
80	449	461	80	304	80	222	80	174	80	128
120	579	581	120	383	120	262	140	216	104	142
—	—	—	—	—	233	346	287	294	225	195
—	—	—	—	—	293	371	—	—	264	206

* From equations viii. and ix.



A. Absorption curve for pure hydrogen.
B. Absorption curve for hydrogen containing 3% nitrogen.
C. Theoretical curve for 3% diluent gas (drawn from equation xiv.).

Fig. 3.

The above results are shown graphically in fig. 4.

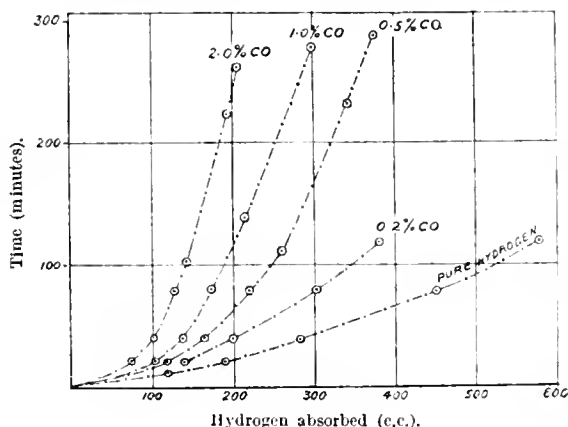


Fig. 4.

In the experiments of the first series, with 0.2, 0.5, and 1.0% CO, the carbon monoxide was almost completely converted into CH_4 , while with 2.0% of CO 85% of it had been so converted. Armstrong and Hilditch* obtained an almost identical result.

The products of the reaction of carbon monoxide and hydrogen, *i.e.*, methane and water, depress the

* Proc. Roy. Soc., 1919, A. 95, 144.

activity on the same grounds as nitrogen, and, in addition, the carbon monoxide, in order to undergo reaction, absorbs some of the energy emitted by the catalyst, which would have been used for the hydrogenation of the oil. Hence the very great "poisoning" effect of carbon monoxide.

Concentration of nickel = 1.0%.

I. Pure hydrogen.		II. Hydrogen containing 2.0% of CO	
Time (mins.).	Absorption (c.c.)	Time (mins.).	Absorption (c.c.)
5	100	5	72
10	163	10	91
20	235	20	114
30	287	40	145
40	341	90	202
50	389	218	218

Other gases investigated were found to fall naturally into one or other of the three groups mentioned, which furnish a convenient classification of gaseous catalyst poisons:—

(a) Gases of which the "poisoning" action is purely a physical one, consisting of two factors, namely, the lowering of the partial pressure of the hydrogen and the diminution of the power of the catalyst for absorbing or occluding hydrogen. Nitrogen is a typical example of this class.

(b) Gases which, in addition to having the physical action of the former class (either themselves or in the form of their transformation products) undergo a chemical change in the presence of the catalyst. The "poisoning" influence of this group is, speaking generally, much greater than that of the former group, a possible explanation of this being that not only do they diminish the "absorptive" power for hydrogen of the catalyst, but also, being occluded on the immediate surface of, or even dissolved in the catalyst, they absorb some of the "energy" emitted by the catalyst, becoming themselves reactive. Carbon monoxide is typical of this class.

(c) Gases which are able to react chemically with the catalyst itself, forming stable compounds at the temperatures employed. The mode of action of these poisons is self-explanatory. Hydrogen sulphide was found to belong to this class.

SUMMARY.

(1) Equations are derived for the rate of addition of hydrogen to mixtures of unsaturated glycerides, using nickel as a catalyst. The reaction between pure hydrogen maintained at a constant pressure and an unsaturated glyceride is of the first order. The more highly unsaturated glyceride (linolin) takes up hydrogen at a very considerably greater rate than olein.

(2) The rôle played by the catalyst is discussed from the results of experiments on the influence of the pressure of hydrogen and of temperature on the velocity of the reaction. It was found that the rate of saturation of olein (containing three double bonds) was proportional to $p^{1.5}$ where p is the pressure of hydrogen, which is in accordance with the view that the hydrogen becomes active through its absorption by the catalyst—Sieverts having shown that such absorption is proportional to \sqrt{p} —with a dissociation of the hydrogen molecules into atoms. The temperature coefficient of the velocity constant was found to be small, an increase of 10° in the temperature for the range investigated (120° – 180° C.) only increasing the velocity 1.13 times, thus suggesting a photochemical reaction, the molecules of olein being brought into an active

condition by absorption of infra-red radiation emitted by the catalyst—a conception of catalysis first put forward by Lewis and Lambie (*loc. cit.*) for the hydrolysis of methyl acetate in a homogeneous system, the catalyst being the hydrogen ion.

(3) The mode of action of "gaseous catalyst poisons" is illustrated and discussed. Such action may be a purely physical one, or it may be chemical in the sense that the "poison" is capable of reacting either with or in the presence of the catalyst.

In conclusion the author desires to express his thanks to Messrs. Lever Bros., Ltd., for permission to publish these results, and in particular to Mr. Buchanan and Mr. Tainsh for the great interest they showed in the progress of the investigation.

Research Laboratories, Port Sunlight.

Communication.

THE SOFTENING OF PLASTIC MATERIALS DETERMINED BY THE WIDNEY RESILIOMETER AND EXPRESSED GRAPHICALLY.

BY ALAN SPEEDY.

It has long been customary to compare plastic materials by such tests as melting point, twisting point, penetration, etc. These tests in the author's opinion are valuable up to a certain point, but are not easily reproducible and do not give any indication of the various changes taking place under heat. It is for this reason that the following method has been adopted.

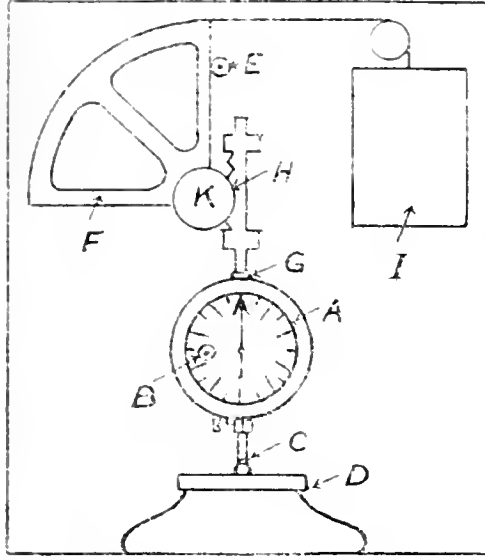
The instrument used for carrying out this test was the Widney Resiliometer† (Fig. 1), which was adapted so that with a presser foot, C, of $\frac{1}{8}$ in. diameter, the weight, I, gave a pressure of 200 lb. per sq. in. The metal base, D, was replaced by ebonite, in order that loss of heat through conduction should be as little as possible. The details of the machine can be seen from Fig. 1; the large dial, A, is graduated in 100 divisions, each 1-1000 in., so that one revolution of the large pointer or hand is equivalent to an elevation of 1-10 in. travel by the presser foot. When the material is more than 1-10 in. thick the hand will revolve more than once around the dial, the number of revolutions being indicated on the small dial B.

The operations are as follows:—The thickness is first determined by placing the sample between C and D, the normal thickness being read off on the dial. The spring-catch, E, is now released, holding the weight, I, and the quadrant, F, allowed to swing until the pressure is put on the upper end of presser foot, G, at the top of dial. This forces the foot into material until it reaches the limits of compression as the result of the weight applied; the reading on the dial at that time is taken and compared with the normal or original thickness. For example, a sample having a normal thickness of 100 mils (a mil = 1/1000 in.) gave a reading at 200 lb. per sq. in. of 25, i.e., at this

* Lewis has recently begun to consider heterogeneous reactions from the point of the radiation hypothesis (Chem. Soc., Trans. 1919, 115, 182).

† Manufactured by the Widney Co., 322, South Jefferson Street, Chicago.

pressure the material would show a compression of 25%.



A. Dial. B. Revolution counting dial. C. Presser foot. D. Base. E. Spring catch. F. Quadrant. G. Upper end of presser foot. H. Transmission plunger. I. Weight. K. Toothed pivot.

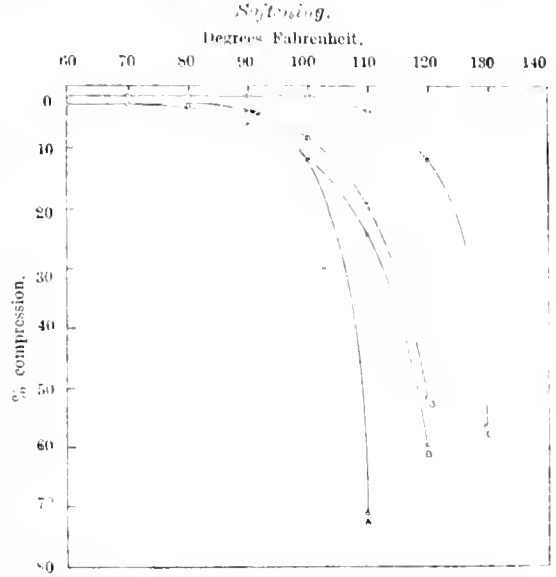
Fig. 1.

Preparation of test piece.—A sample of convenient size for testing is 2.5"×0.5"×0.100" thick and is easily obtainable by aid of a small mould, the shape of the sample permitting several tests to be determined throughout its length.

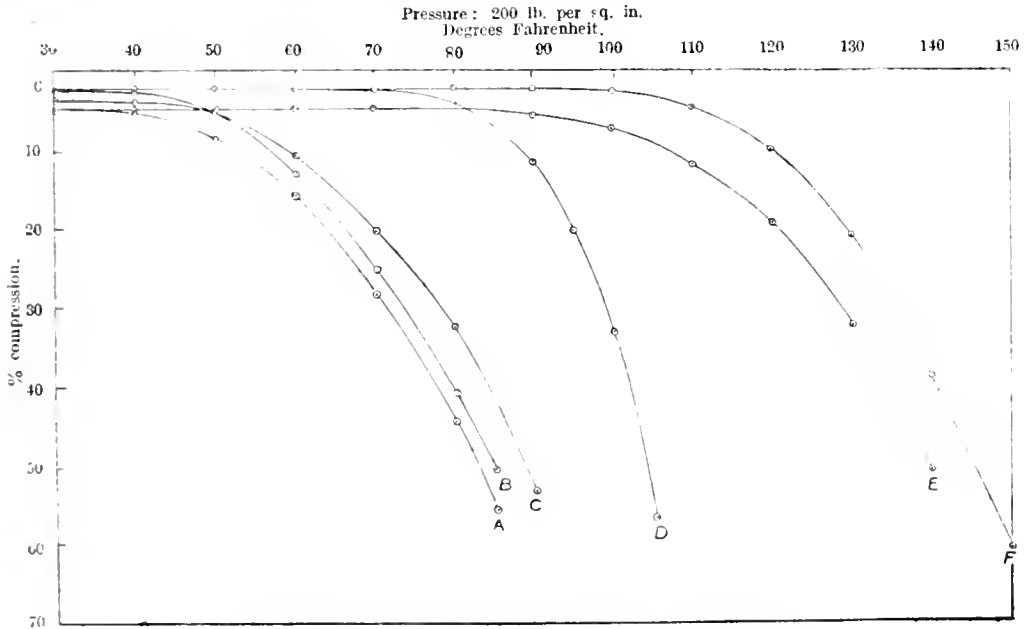
"Softening."—The procedure is as follows:—

tained at the required temperature for a few minutes to ensure that the sample is uniform throughout. It is then quickly taken out, its compression taken and replaced in the bath, the temperature of which is then raised at the rate of about 4° F. a minute to the next reading, the sample being tested at every 10° F.

When dealing with complex substances which have no definite constant, the value of these curves will be at once apparent. The author has found the curves most instructive and of great assistance in



A. Siak gutta percha (low grade). B. Balata (low grade). C. Balata (low grade). D. Leaf gutta percha (high grade).

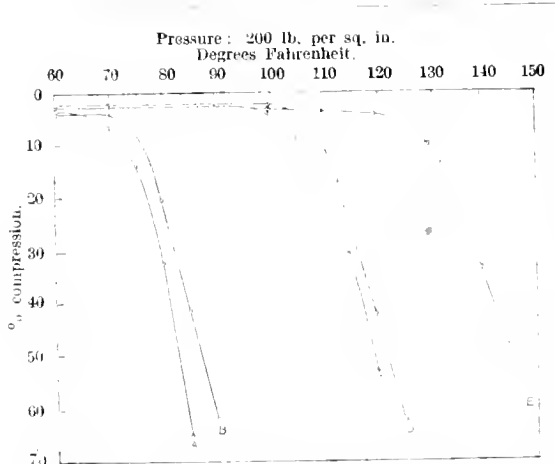


A. Mexican oil pitch. B. Tough oil pitch. C. Asphaltum. D. Medium coal tar pitch. E. M.R.X. (bitumen). F. Trinidad pitch.

A test piece, which should be moulded about twelve hours before testing, is placed in water at 60° F. or lower, depending on the sample, and main-

controlling running batches of material.

In conclusion, he desires to thank Mr. A. Manning for assistance in carrying out tests.



Softening.
WAXES.

Temp. ° F.	Compression, %.				
	Paraffin, 120°.	Earth.	Ceresin (Brown).	Paraffin, 135°.	Montan.
60	3.5	4.0	2.5	2.0	2.0
65	4.0	—	—	—	—
70	6.5	4.0	2.5	2.0	2.0
75	14.0	—	—	—	—
80	32.5	20.5	2.5	2.0	2.0
85	65.0	41.5	—	—	—
90	92.6	62.0	3.0	2.0	—
100	—	88.0	4.5	2.5	3.5
110	—	—	20.0	10.0	4.0
115	—	—	31.0	—	—
120	—	—	53.5	42.5	4.5
125	—	—	—	63.0	—
130	—	—	—	—	10.5
135	—	—	—	—	20.0
140	—	—	—	—	34.0
145	—	—	—	—	50.0

See Graph 1

A B C D E

GUTTA PERCHA AND BALATA.

Temp. ° F.	Compression, %.			
	Siak G.P.	Balata (low grade).	Balata (high grade).	Leaf G.P.
60	2.5	2.5	2.5	1.0
70	2.5	2.5	2.5	1.0
80	2.5	3.0	3.0	1.0
90	4.0	6.5	3.5	1.0
100	12.0	12.0	8.0	1.0
110	71.5	24.5	19.5	4.0
120	—	60.0	52.0	12.0
130	—	—	—	57.0
140	—	—	—	—

See Graph 3

A B C D

PITCHES.

Temp. ° F.	Compression, %.					
	Mexican oil.	Tough oil.	Asphal- tum.	Medium coal tar.	M.R.X. (bitu- men) mineral rubber.	Trini- dad.
30	5.5	3.0	4.0	2.5	5.0	2.5
40	5.5	3.0	4.5	2.5	5.0	2.5
50	9.0	5.5	5.5	2.5	5.0	2.5
60	16.0	13.5	11.0	2.5	5.0	2.5
70	28.5	25.5	20.5	3.0	5.0	2.5
80	44.5	41.0	32.5	4.5	5.0	2.5
85	56.0	51.0	44.0	—	—	—
90	70.3	67.6	53.5	12.0	6.0	2.5
95	—	—	67.5	20.5	—	—
100	—	—	—	33.5	7.5	3.0
105	—	—	—	57.0	—	—
110	—	—	—	—	12.5	5.0
120	—	—	—	—	19.0	10.0
130	—	—	—	—	32.5	21.0
140	—	—	—	—	51.0	39.0
150	—	—	—	—	67.5	61.5

See Graph 2

A B C D E F

American Section.

Meeting held at Chemists' Club on Nov. 21, 1919.

MR. C. E. SHOLES IN THE CHAIR.

INDUSTRIAL FILTRATION.

BY HENRY B. FABER.

The first important step in the approach to a problem of filtration is the determination whether or not the solids may be separated from the liquid content by the agency of a filter medium, and for this purpose a preliminary settling test should be made. A graduated vessel filled with the slurry in question and maintained at a given temperature will give much useful information, which may be supplemented by a microscopical examination of the various particles. If there are particles in suspension which exhibit Brownian movement, I would be inclined to classify the problem as one not adapted to the ordinary methods of filtration, viz., the separation of the solid from the liquid by means of a porous medium.

Following this test, a very simple apparatus may be used in order to obtain data as to the character, size, and, in many cases, method of operation of the filter capable of handling the problem. It consists of a frame of brass tube $\frac{1}{2}$ in. in diameter, the lower side of which is perforated. Over this frame is sewn canvas or some other common filtering medium, forming a paddle or filter leaf. This leaf is approximately $3\frac{1}{2}$ in. square, having $\frac{1}{2}$ sq. ft. of active filtering surface. This test filter leaf, connected to a Woulff's bottle exhausted by a vacuum pump, is introduced into the slurry, which has been brought to the temperature at which it will be filtered, kept thoroughly agitated. The solids form as a cake upon the filter leaf, the liquid being drawn through and caught in the Woulff's bottle. After a cake of the desired thickness has been formed the leaf with the adhering cake may be removed and introduced into water, which is drawn through the cake, expelling the filtrate and washing the solids. After washing has been completed the leaf with the adhering cake is suspended in the air with the suction still on to remove excess of moisture. The cake may now be discharged from the leaf by disconnecting it from the Woulff's bottle and subjecting the leaf to a puff of air blown through its hollow frame. This simple test will supply data from which to calculate the area of active filtering surface required to handle a given problem under the prescribed conditions.

Filtering problems fall naturally into three distinct groups:—

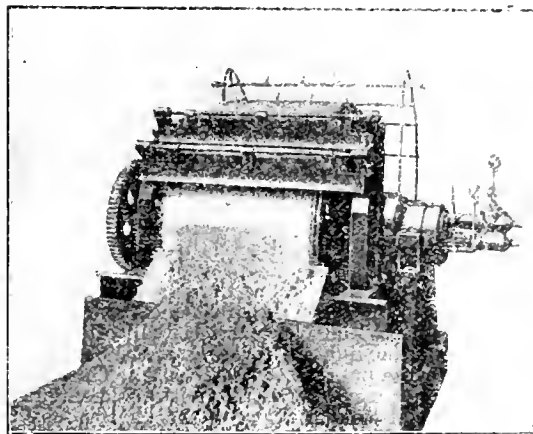
The first group embraces those slurries in which the suspensions are fine, but not of a colloidal character; those slurries which contain slow-filtering suspensions in large or small bulk; and those slurries which by reason of a relatively small amount of suspended matter require merely clarification.

The second group embraces those slurries containing relatively free-filtering suspensions—slurries which permit the liquid content to be separated readily from the solids, where the solid content is of considerable bulk, crystalline, or definite in its form.

The third group embraces those slurries which are principally mother liquors, containing relatively large crystals, in which the liquid content can be drained readily and the crystals washed easily, the principal problem being the automatic and easy handling of the solids.

In the first group, in which the suspensions are fine and the slurries slow in filtering, the engineer has two problems confronting him—the problem of large area of filter surface and that of keeping the filter surface clean. The filter press is probably the best known type of filter equipment in use.

A process of filtration invented by George Moore has been extensively employed in metallurgical work. This process involves the use of filter leaves, operating in an open tank. These leaves are frames of pipe, usually rectangular in shape, over which are sewn filter cloth completely encasing the frames and forming a hollow shallow chamber with ribs spaced equidistant across the face of the leaf to prevent the two faces of the cloth from touching. The pipe constituting the lower edge of the frame is perforated. When the leaf is submerged in the slurry to be filtered and suction applied, this suction is translated throughout the entire space within the leaf, causing the liquid to pass rapidly through the cloth, leaving the solids deposited upon the outside surface in the form of an adhering cake. Many of these leaves are attached to a common header, and may be enclosed in a container for use with either suction or pressure. The simplicity of this construction permits the use of very large areas of filtering surface at a relatively low cost.



Rotary Filter—Zenith Type.
(In operation.)

In reference to the handling of problems in the second group, viz., slurries which are relatively free-filtering and build a substantial firm filter cake quickly, the rotary filter is the most desirable type to use. The rotary filter made its first appearance in Belgium about fifty years ago, and consisted of a headed barrel rotating about its long axis. The face of the drum was perforated and covered with a filtering medium held in place by winding cords. This machine was used for the filtration of salts and proved very effective. Suction was applied to the interior of the drum by means of a goose-neck passing through the head of the drum and dipping down to within a short distance from the bottom. The drum rotated in a shallow container,

dipping into the slurry through an arc of about 40°. Later this type of machine was built of substantial metal construction. The perforated surface was replaced by bars of iron, forming a grating. In all cases a goose-neck dipping within the drum downward to within a short distance from the bottom, served as an exhaust and also to carry away the liquid from the inside of the drum. When the drum was rotated the filter cake, which adhered to the surface, was removed by means of a scraper.

George Moore has also invented a multiple-compartment or rotary filter, in which the peripheral surface of the drum is composed of a number of shallow hollow compartments, over which the filtering medium is stretched. Each of these compartments has its own separate pipe line connected to a valve hub, by means of which suction or pressure may be applied at will through any desired arc. These machines have had wide usage in the mining field for the handling of concentrates, and are extensively used in industrial chemical work, where automatic and continuous filtration is rapidly replacing the older methods.

In handling slurries of the character mentioned in the third group, viz., crystals from mother liquor or coarse granular suspensions, the centrifuge or draining boards have been widely used.

The rotary filter has failed in many cases by reason of the fact that the solids will form too heavy a cake and will not adhere firmly enough to prevent the cake breaking away from the drum and thickening in the container. The "rotary hopper de-waterer" is a type of filter machine built especially for this class of work and consists of a series of elongated hoppers arranged radially about a central axis, each hopper having a filter bed, below which a pipe line connects with the central valve hub, similar in design to that used in the construction of the rotary filter. As the filter beds are set quite a distance below the rim of the hoppers, they form a series of filter trays, revolving slowly about a central axis. The slurry is fed into these trays at a point about 30° from the zenith and de-watered by means of suction as they rotate through an arc of about 40°. Suction is then automatically cut off and the product discharges either by gravity or by a puff of air, making the operation continuous and automatic.

In washing the filtered solids it is important to provide against the channelling of the filter cake. In the filter press, as well as the self-dumping type, it is difficult to provide against a classification of the solids, as the heavier particles will often settle away from the lighter, forming areas of less resistance to the passage of the filtrate. This classification is not met with to any noticeable extent in the open tank type of filter, as the slurries may be kept in constant and thorough agitation during the process of filtration, thus ensuring a homogeneous slurry. Consequently, it has been my experience that washing on the open tank leaf type of filter can be accomplished with less wash-water and a more thoroughly cleansed product obtained than by either the press or self-dumping type. It seems to be a prevailing custom among engineers to determine the degree of wash that a filter-cake has been subjected to by merely testing the wash-water coming from the filter. It is much better to sample the filter cake itself, taking a number of samples from different parts in the chamber or on the filter leaf. It is also desirable, wherever possible, when extreme degrees of washing are required with a minimum amount of wash-water, to discharge the cake after the mother liquor has been replaced to a reasonable extent by wash-water, and to churn up or cream the product, thus getting the advantages of diffusion, and then to re-filter. This method of washing is particularly advisable in the

case of the rotary filter where the period of washing is relatively short, the filtration, washing, and automatic discharge being accomplished in one revolution of the filter drum. Very satisfactory results have been obtained on difficult problems of washing where only sufficient wash-water is sprayed upon the cake to replace the mother liquor held by the cake, volume for volume, and the cake discharged into an agitating tank in which it is mixed with wash-water in sufficient amount to permit the easy handling of the slurry, and re-filtered. In dealing with free alkalis the passage of steam through the cake prior to washing with water will often show very beneficial results. Many filtration problems will be simplified if more care and attention is given to the form in which the solids appear at the time that filtration is to be attempted. In other words, the chemical reactions should have run their course and the suspensions should have been allowed to form themselves in a physical condition as far as practicable, giving the best separation possible having regard to their nature and crystalline structure.

Birmingham Section.

*Meeting held at Birmingham University on
Thursday, December 11, 1919.*

MR. L. P. WILSON IN THE CHAIR.

ON THE INFLUENCE OF THE TEMPERATURE, CONCENTRATION, DURATION OF THE MASH, AND SLACKNESS OF MALT ON THE STARCH PRODUCTS OF THE EXTRACT OF MALT.

BY JAMES O'SULLIVAN, F.I.C.

In a communication to the Institute of Brewing "On an Improvement in the Method of Malt Analysis" (J. Inst. Brew., 1911, 17, 35; see this J., 1911, 230) I showed how by the standard method of mashing the starch of malt could be estimated and the relative quantities of its transformation products determined in the mash extract. The mashing conditions in that work were mainly in accordance with the standard method of malt analysis (J. Inst. Brew., 1906, 12, 1; see this J., 1906, 236). I now purpose showing the influence which variations in the conditions of mashing have on the composition and behaviour in a fermentation of the transformation products of the starch.

It must be clear to anyone who has followed the method of working described in the paper above mentioned that if the sucrose, always present in malt, were inverted during the time allowed in the standard method of mashing an error would be introduced to the extent of the invert sugar produced. Although I then pointed out that during the time of mashing none of the sucrose was inverted, and that slightly more sucrose was found in the mash extract than in the alkaline water extract, yet I consider it useful to give further proof of this. When malt is extracted with water at 20° C. for three hours, with or without agitation, I have invariably found that the extract is greater and has a higher cupric-reducing power than when the extraction is made with alkaline water, as in the standard method of malt analysis.

That the higher cupric-reducing power of the

water extract is not due to inversion of the sucrose of the malt during the extraction was proved by heating a volume of that extract to 100° C. and estimating its sucrose content by the method described by me (*loc. cit.*), when it was found that it contained the same amount of sucrose as the alkaline water extract. The cupric-reducing power of the water extract, or that of the alkaline water extract, is not altered by boiling either of these extracts. These facts prove that invertase is not present in malt.

By simply grinding a quantity of malt in the presence of water, in a specially-constructed apparatus, I found that it yielded to the water an amount of maltose equal to 63% of the starch it contained, allowance being made for the cupric-reducing power of the sugars present in the malt.

By grinding in a similar manner potato starch, and also malt starch, with prepared diastase, in the former case for two hours and in the latter for fourteen hours, it was found that the cupric-reducing power of the portion that went into solution—calculated as maltose—amounted respectively to 17.4 and 70.4% of the starches employed. (See also J. L. Baker and H. F. E. Hulton, *Chem. Soc. Trans.*, 1914, 105, 1529; this J., 1914, 760.)

The results of many similar experiments with starches showed that the optical activity of the starch products so obtained is much less than that calculated from their apparent maltose and dextrin content, after correcting for the quantity of diastase and its very slight levo-rotatory power.

I conclude from the foregoing that the greater extract and the higher cupric-reducing power of the water extract of malt over that obtained by the alkaline water method of extraction is due to solution of the starch granules and not to the inversion of the sucrose.

At present I am prepared to state that apparent maltose and dextrin are not the sole products of the action of diastase in the cold on either malt or potato starch "granules." However, I find no evidence of the presence of dextrose in what goes into solution, as I failed to obtain an insoluble osazone. The osazone test for small quantities of dextrose in presence of relatively large quantities of starch products, or of maltose, is, I find, a complete failure.

In order to establish the absence of invertase from ordinary malt I give in Table 1 the amount of sucrose found in 10 malts obtained by the two methods of extraction stated in that Table.

Table 1.
Sucrose per cent. in malt.

Malt.	Extraction by		
	Standard method of malt analysis. Alkaline water at 20° C.	Mash 3 hours.	
		Sucrose %.	Sucrose %.
1	4.5	64°	4.5
1	4.5	64°	4.5
2	5.0	59°	4.7
2	5.0	59°	4.5
3	5.5	65°	4.7
4	5.5	65°	5.4
5	6.2	65°	5.5
6	6.5	65°	7.0
7	4.2	65°	7.3
8	4.4	65°	4.2
9	5.3	65°	5.2
10	4.9	65°	4.7
			4.6

On comparing the numbers in columns 2 and 4 above there can be no other conclusions than:— (1) That sucrose is not inverted in the process of mashing, and (2) that invertase is not present in malt. I have already proved (*Trans. Laboratory Club*, 1890, 3, 5; this J., 1890, 535) that invertase is present in germinating barley, and (*J. Chem. Soc.*, 1900, 77, 69; this J., 1900, 386) have established its presence in all parts of mature plants of the gramineae. Invertase and sucrose are present in malt culms, but invertase cannot be to any extent extracted by water from them. Malt culms contain as much sucrose as malt.

The absence of invertase from malt, and also lipase as proved by me (*loc. cit.*), makes the way clear for determining the influence of varied conditions of mashing on the starch transformation products of malt.

In Tables 2, 3, and 4 the numbers given, other than those in columns 9, 10, and 11, were obtained by the mode of procedure already described (*loc. cit.*).

Cold water extract was obtained by digesting 25 grms. of the ground malt for three hours at 20° C. in 250 c.c. of alkaline water containing 20 c.c. of N/10 potassium hydroxide. The specific gravity of the filtrate from this digestion was determined, and also its cupric-reducing power, gravimetrically, by using 10 c.c. and calculating this as maltose per 100 c.c.

Mash extract.—50 grms. of the ground malt was introduced directly into a 515 c.c. (Heron's) flask and placed in a water bath kept at a temperature 2° C. above that of the required mashing temperature. The malt in the flask was heated to 60°–61° C., and when the quantity of mashing water used was 360 c.c. this was added to the malt at 3° above the required mash temperature; when 134 c.c. of mashing water was used, this was added at 6° above the required mash temperature. The temperature in column 3 in the Tables was actually that of the mash for the time stated until it was heated to 75° C. for 10 minutes.

The mash in the flask was cooled and made up to 515 c.c. at 15.5° and filtered. The specific gravity of the filtrate and its cupric-reducing power, employing 3 c.c., were determined and the latter calculated as maltose per 100 c.c.

We have so far four factors: *a*, the specific gravity of the mash extract; *b*, that of the alkaline water extract; *c*, the cupric-reducing power, as maltose per 100 c.c. of the former; and *d*, that of the latter. From these factors we arrive at the composition of the starch transformation products at the temperature of the mash, and also at the amount of starch in the malt in the following manner.

$a-b$ =the specific gravity of the mash extract which is due to the starch products, and $c-d$ =the grms. of maltose in the starch products of the mash extract.

The specific gravity above water of $(a-b) \div 4$ equals the grms. of starch product per 100 c.c. in the mash extract. For the strength of this extract the divisor 4 is nearer the truth than any other divisor. There is, however, a slight, unimportant correction to be made here. From numerous determinations of the quantity of soluble albuminoids in the cold water extract and in the mash extracts of malts I find that the latter contains an average of 0.04 gm. of albuminoids ($N \times 6.25$) per 100 grms. of malt more than the former, and therefore the correct number for the starch products is $\frac{1}{4}(a-b) \div 4 - 0.04$ grms. of starch products per 100 c.c. (*e*).

Then $(c-d) \times 100 \div e$ = the percentage of maltose in the starch products (column 8).

The dextrin is obtained by deducting the grams of maltose from the starch products, and the maltose divided by 1.055 gives the starch equal to the maltose which, when added to the dextrin, gives the starch in 10 grms. of malt (column 6). One grm. of starch = 1.055 grms. of maltose.

The "transforming power" of the malt (column 7) is expressed by the percentage of starch which is converted into maltose.

As an example, the numbers obtained for a malt were: *a* mash extract sp. gr. 1028.70, *c* = 4.833 grms. maltose per 100 c.c.; *b* cold extract sp. gr. 1007.49, *c* = 0.734 grm. maltose per 100 c.c.; due to starch products $1021.21 = 4.099$ grms. maltose per 100 c.c. Then $(21.21 \div 4) - 0.04 = 5.26$ grms. of starch products per 100 c.c., and $(4.099 \times 100) \div 5.26 = 77.8\%$ of maltose on starch products, $5.26 - 4.099 = 1.161$ grms. of dextrin, and $4.099 \div 1.055 = 3.885$ grms. of starch; and 1.161 grms. of dextrin + 3.885 grms. of starch = 5.046 grms. of starch in 10 grms. of malt, or 50.46%. The transforming power of the malt is $(4.099 \times 100) \div 5.046 = 81$.

The extract of 10 grms. of malt in 100 c.c. was fermented with yeast at 22° C. for 5 days and shaken every day. On the sixth day—fermentation having apparently ceased on the third—the fermented extract was evaporated, cooled, and its volume (yeast in) made up to the original volume at 15.5°. In order to avoid filtering out the yeast the extract, when made up to the original volume, was transferred to a beaker and allowed to rest, covered overnight; the yeast having subsided in that time, the supernatant liquid was poured off and its specific gravity determined. The degrees of gravity before fermentation, less those of the residue, give the degrees fermented, from which the percentage of extract fermented is obtained, which is given in the tables (column 10).

In carrying out the fermentation of the extracts it was immaterial whether primary yeast (*Saccharomyces cerevisia*) or secondary (*S. pastorianus*) was employed, or whether an excess of these ferments, or a growth of either was employed, the amount fermented being the same in all cases. By a growth of yeast I mean starting the fermentations with a small quantity.

The fermentation of the alkaline water extract (cold-water malt extract) after neutralisation and boiling was carried out as in the mash extract; and although the whole of the cupric-reducing power of this extract disappears on fermentation, there was a slight phosphate precipitate obtained from the unfermented residue, which was filtered off and treated as if it were cuprous oxide. Allowance was made for this in all determinations of the cupric-reducing power of the extracts.

The degrees of gravity fermented of the cold-water malt extract, together with the degrees corresponding to the maltose found in the mash extracts, give the total, which, for my purpose, is calculated as fermentable, and from which the percentage fermentable given in column 9 is deduced.

The amount of the starch products fermented (column 11) is obtained by deducting the degrees of gravity fermented of the cold-water malt extract from the total degrees fermented of the mash extract; the difference is due to the degrees of gravity corresponding to the starch products fermented.

I have allowed—though this is unimportant—for the gravity of the albuminoids in the mash extracts over those in the cold-water malt extract by deducting 0.16 from the difference between the gravities of the two extracts.

The percentage of starch products fermented (column 11) is equal to (degrees of gravity fermented in mash extract—degrees of gravity fermented in cold-water malt extract) $\times 100 \div$ degrees of gravity due to starch products in the mash extract.

Table 2.

Mash.				Extract.						
1	2	3	4	5	6	7	8	9	10	11
Series.	Water, c.c.	Temperature, °C.	Time of stand, hrs.	Solids % on malt D 4.	Starch % on malt.	Transforming power, grms. maltose 100 grms. starch.	Composition of starch products, M = maltose % D = dextrin %.	Percent. fermentable maltose + fermentable of C.W.M.E.	Percent. fermented.	Percent. fermented of starch products.
1	360	65-66	1	71.9	48.8	75.5	M = 72.7 D = 27.3	63.8	69.8	80.3
				71.8	48.6	77.5	M = 76.6 D = 23.4	65.8	70.7	83.3
				71.8	49.8	76.3	M = 75.5 D = 24.5	64.3	69.6	80.1
				71.8	49.0	76.4	M = 74.9 D = 25.1	64.6	70.0	81.2
				71.6	49.6	80.7	M = 77.4 D = 22.6	67.1	71.8	85.0
				71.0	47.9	82.5	M = 79.3 D = 20.7	68.2	71.7	83.1
2	360	62.5 63.5	1	72.0	50.0	81.4	M = 78.3 D = 21.7	67.7	72.5	81.4
				71.5	49.1	81.5	M = 78.4 D = 21.6	67.6	72.0	83.1
				71.8	48.7	87.2	M = 85.0 D = 15.0	71.7	72.5	85.4
				71.9	49.0	85.0	M = 84.0 D = 16.0	68.5	72.9	85.0
				71.6	49.3	86.5	M = 81.7 D = 18.3	70.2	73.0	87.3
				71.7	49.3	86.2	M = 83.6 D = 16.4	70.1	72.8	85.9
3	360	60.5 61.5	1	70.7	48.8	76.0	M = 73.0 D = 27.0	63.6	62.2	70.0
				70.7	48.8	78.8	M = 77.0 D = 23.0	66.5	63.3	71.5
				70.7	48.8	78.6	M = 76.5 D = 23.5	65.0	63.9	72.0
				70.2	48.0	86.4	M = 82.7 D = 17.3	70.6	75.8	89.5
				70.2	48.0	89.0	M = 85.2 D = 14.8	72.3	74.3	87.5
				70.2	48.3	87.0	M = 83.5 D = 16.5	72.3	75.7	88.9
4	134	65-66	1	70.2	48.1	87.4	M = 83.8 D = 16.2	71.7	75.2	88.6
				69.5	47.6	75.0	M = 72.9 D = 27.1	63.6	67.2	77.1
				60.8	47.8	77.0	M = 74.4 D = 25.6	62.8	66.6	76.3
				69.5	47.7	74.0	M = 70.9 D = 29.1	62.9	65.4	74.9
				69.6	47.7	75.3	M = 72.8 D = 27.2	63.1	66.4	76.1
5	134	65-66	3							
6	134	69-70	3							

On examination of the average results in Table 2, where 360 c.c. of water to the 50 grms. of malt was used—thin mash—and the temperature varied, it will be seen that as the temperature diminishes the transforming power increases, and, as a matter of course, the calculated fermentable matter increases, but the amount fermented does not in-

crease in proportion. It will also be noticed that the amount of starch products which ferments is greater than the maltose they contain, and that this is not so marked in the low temperature series of experiments. The inference is, as we know of old, that apparent dextrin is fermented.

When the results in series 4 of experiments—the variation being in the volume of water (thick mash)—are compared with series 1 we find that the transforming power and the fermentable are, for all practical purposes, the same, but the fermented is 6.9, and the fermented of the starch products is 10.1% less than in series 1, and the fermented of the starch products is less than the maltose they contain.

A comparison of the results in series 5, in which the stand of mash was 3 hours, with those of series 4 of 1 hour shows that the transforming power is 8.8, the fermentable 6.7, and the fermented 12.1% higher than in series 4, and also that the starch products fermented are 17.4% higher than in series 4 and greater than the maltose they contain.

The results in series 6, in which the mashing temperature was 69°–70° C., are much lower than in series 5, and the starch products fermented is above the maltose they contain.

In all of the unfermented residues both maltose and dextrin were present, consequently it must not be inferred where the amount of starch products fermented is less than the maltose contained in them that only maltose, and not all of that, is fermented. Apparent dextrin and apparent maltose fermented in all, but I am only concerned with the amount fermented of the malt extract.

Table 3.

Mash.				Extract.						
1	2	3	4	5	6	7	8	9	10	11
Malta.	Water, c.c.	Temperature, °C.	Time of stand, hrs.	Per cent. on malt D 4.	Starch % on malt.	Transforming power, grms. maltose 100 grms. starch	Composition of starch products, M = maltose % D = dextrin %.	Per cent. fermentable maltose + fermentable of C.W.M.E.	Percent. fermented.	Percent. fermented of starch products.
1	134	65-66	1 3	81-5 64-2	43-4 45-7	63-4 78-8	M = 61-4 D = 38-6 M = 75-9 D = 24-1	56-4 66-9	50-0 59-4	51-3 64-6
2	134	65-66 60-61	3 3	63-5 66-5	49-2 47-2	85-0 93-0	M = 81-1 D = 18-9 M = 88-8 D = 11-2	71-5 77-0	68-8 70-3	77-7 80-0
3	134	65-66 60-61	3 3	63-2 65-0	48-4 45-2	85-1 95-0	M = 81-5 D = 18-5 M = 94-6 D = 5-4		65-5 67-1	
4	134	65-66	3	67-9	48-2	73-7	M = 70-4 D = 29-6	64-2	60-2	64-8

The results in Table 3 are from four malts according to the condition of series 5 of experiments, and it will be seen how greatly they differ from those of that series. It will also be noticed that in mashing at 60°–61° C. the starch is not all dissolved in malts 2 and 3. These were foreign, thin and steely, low-class malts, and were chosen to see if the starch products yielded by them, and the behaviour of these on fermentation, would show

any great variation. The results are shown to differ, and there is no analytical method yet devised, other than that here described, by which the difference, on mashing, in the starch products of these malts could be revealed. It will be observed that a fermentation of the extract is necessary in all cases in order to disclose if a malt when mashed will yield high, not easily fermented, or low, easily fermented, types of starch products.

Matthews and Auty (J. Inst. Brew., 1907, 13, 685) in a very exhaustive work on the influence of the temperature of the mash on the composition of the extract, experimented at mashing temperatures of 60°–65.6° C. with a 10% malt-to-water mash and a two hours' stand.

The results I have given are on the basis, in the thin mash, of practically 14% and in the thick mash of 37% of malt to water, and at the expiration of the time of stand the entire mash was heated to 75° C. for 10 minutes.

Matthews and Auty did not raise the temperature above that of the mash before filtering, consequently active diastase would be left. As the time taken to filter the volume of extract they dealt with would be very much longer than that of the stand of the mash, I infer that although under their conditions of working their results are in themselves practical and comparative, yet they cannot be looked upon as showing the influence of the temperature of the mash on the composition of the wort. The higher the temperature of the mash the less would be the diastatic action during its filtration when not heated to a temperature sufficiently high to stop the diastatic action.

The quantity of moisture in malt has, from time immemorial, been looked upon with suspicion by the brewer. Excessive moisture in malt indicates long and imperfect storage, and, in my opinion, it is absurd to ascribe this, under ordinary conditions of transit, to be due to moisture absorbed during transit.

The following experiments were made in order to answer the question: Has slackness of malt any influence on the composition and character of the starch transformation products of the extracts when such a malt is mashed?

A weighed quantity of malt containing 3.9% of moisture was placed in a wide-mouthed bottle and kept stoppered; another weighed quantity of the malt was placed in a similar bottle and kept unstoppered. From time to time the malt in the open bottle was well mixed and weighed. After the lapse of six months this malt was found to have absorbed only 6.8% of moisture, making a total moisture content of 10.7%. The malt kept in the closed bottle for the same time had only absorbed 0.17%. I will call the former "dry malt" (D), and the latter "slack malt" (S). To aim at the same amount of extract from the slack malt 53.4 grms. is required where 50 grms. of the dry malt is used.

Table 4 gives the results obtained in experiments S 1 and S 2 for the slack malt, and in D 1 and D 2 for the dry malt; and in D 3 the results for the dry malt 19 months after the results S 1 and 2 and D 1 and 2 were obtained.

A second malt containing 3.9% of moisture on March 20, 1911, was used, and the results, D 4 and D 5, were obtained for it at the date. Some of this malt at this date was put into a wide-mouthed bottle and a ball of moist cloth was suspended over it in the bottle.

The cloth was kept moist, and from time to time the malt was taken out of the bottle and mixed. On May 11, 1911, the moisture was found to be 8%, or an increase of only .41% in spite of ex-

posure to a moist atmosphere for nearly two months. The malt at this date was sealed up in a bottle, as was the original malt on March 20, 1914.

The results obtained for these malts on November 9, 1914, are given in Table 4, experiments S 6 and S 7 and D 8 and D 9. The results of S 1 and D 1 one hour stand show a difference in the transforming power and, consequently, a difference in the composition of the starch products, and although there is less fermentable in D 1, more of the extract ferments and of the starch products than in S 1, but in S 2 and D 2 the results are practically the same. In D 3 the results obtained for the same malt kept for 19 months show the transforming power to be practically the same as in S 2 and D 2, but the fermented of the extract and of the starch products is less. The average of the two experiments with the second malt on March 20, 1914, D 4 and D 5, and the two experiments on Nov. 11, 1914, D 8 and D 9, are practically the same, but the results on Nov 11, 1914, S 6 and S 7, show the transforming power and extract to be less, but practically the other figures do not differ from those of the dry malt.

The malts were ground in a Seck mill set at 25; it was, however, found that for the determination of the alkaline cold water extract of the slack malt this method of grinding gave low results unless the malt was passed many times through the mill. There is little doubt that the difference between the extracts of the slack and dry malts is also due to the fact that the former does not grind as fine, under like conditions, as the latter.

Table 4.
"Slack" and "dry malt" compared.

Mash.					Extract.							
Experiment	1	2	3	4	5	6	7	8	9	10	11	
	Water, c.c.	Temperature, °C.	Time of stand, hrs.		Solids % on malt D 4.	Starch % on malt.	Transforming power, grms. maltose 100 grms. starch.	Composition of starch products' M = maltose %, D = dextrin %.	Percent, fermentable maltose + fermentable of C.V.M.E.	Percent, fermented.	Percent, fermented of starch products.	
S.	{	1	360	65	1	70.0	48.8	79.9	M = 77.7 D = 22.3	67.8	63.2	71.2
		2	360	65	3	71.0	49.3	85.0	M = 81.9 D = 18.1	71.2	69.8	79.6
D.	{	1	360	65	1	70.0	48.6	77.0	M = 73.6 D = 26.4	64.9	65.0	73.8
		2	360	65	3	71.0	49.2	84.0	M = 80.1 D = 19.9	70.0	68.9	78.3
D.	3	360	65	3	71.0	48.9	83.5	M = 79.2 D = 20.8	68.8	66.5	76.0	
D.	{	4	134	65	3	71.0	47.8	91.8	M = 87.5 D = 12.5	76.2	71.1	80.3
		5	134	65	3	71.0	47.9	92.8	M = 88.5 D = 11.5	76.9	69.3	77.8
S.	{	6	134	65	3	69.5	47.0	90.0	M = 86.2 D = 13.8	76.9	73.0	80.9
		7	134	65	3	70.0	47.4	90.4	M = 86.3 D = 13.7	76.8	73.2	81.2
D.	{	8	134	65	3	71.1	48.3	92.6	M = 88.4 D = 11.6	78.4	71.7	77.0
		9	134	65	3	71.0	48.1	91.0	M = 86.9 D = 13.1	77.3	72.4	78.9
D.	10	134	65	3	71.0	49.3	89.0	M = 85.1 D = 14.9	76.3	74.7	83.0	
S.	11	134	65	3	70.0	48.4	88.3	M = 84.4 D = 15.6	75.0	74.0	81.9	

The malts—slack and dry—the results of which on Nov. 9, 1914, are given in Table 4, Expts. 4

to 9, were sealed up in bottles on that date. On June 26, 1916, they were mashed exactly as on the previous date. The results then obtained are given in Expts. D 10 and S 11. It will be seen that the transforming power in each case is less than on the previous date; the composition of the starch products is altered, less apparent maltose is present, the amount fermentable is the same, but the amount which actually fermented of the extracts is greater.

It will also be noticed that all the numbers for transforming power obtained for the second malt in the Expts. 4 to 11 are much above what is considered to be a resting stage in starch transformations; that is, 84% of maltose on the starch, and in Table 3 for the malts 1 and 4 it is less.

I observed in this work that on keeping the malts the cupric-reducing power of their alkaline water extracts diminished considerably, while the quantity of sucrose remained unaltered.

DISCUSSION.

The CHAIRMAN, alluding to the statistics, enquired whether the author had made a graph of the increase of the rate of hydrolysis with the increase of temperature or concentration. If so, what was the type of reaction indicated? Of course, there was only a small range of temperature, as there was not much activity, or fermentation, outside certain narrow limits, the reaction being quite different from the ordinary chemical hydrolysis of carbohydrates.

Mr. DUNCAN said it would appear that if the Lintner method, which had become so standardised and misinterpreted, were improved upon, a substantial advance had been made. Apparently the paper turned on the fermentation test on a miniature basis, and he imagined that subsequent to the fermentation a gravity estimation was made of the residual extract, and the result was divided by a factor. What factor was adopted? Had the author studied the question of the influence of acidity of the mash in connection with the fermentability of the products secured? There appeared possibilities of the lactic acid treatment of mashes proving effective with so-termed "stubborn malts."

Mr. J. RACE enquired if the refractometer had been used for the estimation of solid matter in mash worts instead of the specific gravity method. Did the author conclude from his experiments on slack malts that moisture had no influence on the starch transformation compounds?

Mr. F. R. O'SHAUGHNESSY said the results obtained would appear to indicate that there were considerable differences between the various portions of the dextrin. In the acid hydrolysis of starch there was invariably an appreciable discrepancy between the reducing power of the product and the rotation figures. If one fermented out the product there was always a residue which contained apparently non-fermentable matter. Did that apply in the present case?

Mr. O'SULLIVAN, in reply, said it was not possible to make the comparison as suggested by the Chairman, because instead of the action increasing with the temperature it decreased. He did not value the Lintner method except perhaps from the maltster's point of view; it merely afforded an index as to how malt was cured. It gave no information whatever as to the behaviour of the malt when mashed, and that opinion had been shared, he believed, by the late Professor Adrian Bawn. He did not think it was practicable or useful to study the question of the influence of the acidity of the mash, and he had not done any experiments in that direction. He had not used the refractometer;

he did not know that anything could be more reliable than the specific gravity for determining the amount of extract yielded by malt. There were only slight differences between slack and dry malts, but it must not be inferred that he was speaking of beer. The best quality beer, in point of flavour and character, would be made with dry malt. The acid hydrolysis of starch did not apply, as the products by acid were quite different.

THE ACTION OF WATER ON LEAD.

BY J. F. LIVERSEEGE, F.I.C., M.I.C. AND A. W. KNAPP, B.S.C., F.I.C.

It is well known that many waters act on lead. The subject was investigated by Dr. A. C. Houston, who devised the erosion test with which this paper largely deals (Moorland Waters in regard to their Action on Lead. *Vide* Supplements to Reports of Local Government Board, 1900-2).

The question is of considerable importance from the point of view of public health, and the conditions under which lead is eroded are of interest in relation to the uses of lead in the arts. The waters which Dr. Houston examined were acid in character, and notable for their power of dissolving lead. On the other hand our experiments deal with a faintly alkaline natural water which, like distilled water or pure rain-water, dissolves very little lead, but which has, in the untreated condition, the power of eroding the lead, so that scales formed on it fall away, leaving a fresh bright surface open to attack. The erosive ability was not considered by Dr. Houston to be a dangerous quality, save under special conditions and in the presence of bright lead.

The water used in the tests.

The gathering-ground of the Birmingham water supply, which provides for the needs of a million persons, is about seventy square miles of uncultivated land in Wales. Geologically it consists of Silurian conglomerate, shales, and flagstones. The water is brought by an aqueduct 73 miles long to the large storage reservoir at Frankley. The aqueduct is composed of 24 miles of "cut and cover" conduit, 13 miles of tunnels, and 36 miles of iron pipes. During the passage of the water through the tunnels a certain proportion of spring water forces its way in and is taken up, and so the water becomes less soft during its passage from Wales to Frankley. In order to avoid any risk of so soft a water acting on lead, a small proportion of chalk is added to the water at the beginning of the aqueduct; for some time about three parts of ground chalk per 100,000 was added to the water, but it was found that this was a larger quantity than the water would dissolve, and the usual addition in recent years has been about two parts per 100,000.

The following figures show the proportions of certain constituents in the waters:—

Parts per 100,000.

	Total solids.	Alkalinity as CaCO ₃ .	Chlorine in chlorides.
Water at Welsh reservoir	4	0.3	0.9
Tunnel water	21	13.0	1.2
Water at Frankley (untreated)	5	1.7	1.1
Water at Frankley (treated)	6	2.3	1.0

The following is a complete analysis of the water as supplied to the consumer (parts per 100,000):—Carbonates as CO₃, 1.44; sulphates as SO₄, 0.10;

chlorides as Cl, 0.95; silicates as SiO₃, 0.43; calcium (Ca), 1.03; magnesium (Mg), 0.18; iron (Fe), 0.08; potassium (K), 0.14; sodium (Na), 0.53; undetermined, 1.02—total solid matter, 5.90; free ammonia, 0.000; albuminoid or organic ammonia, 0.005; nitrogen in nitrates, 0.0; oxygen consumed in 4 hours at 27° C., 0.11; total alkalinity as CaCO₃, 2.1.

The alkalinity of the water was determined as follows:—To 500 c.c. of water in a porcelain basin phenolphthalein is added, and if a red colour is produced the liquid is titrated cold with N/10 hydrochloric acid. Lacmoid is then added and the titration completed whilst boiling. As calcium bicarbonate is neutral to phenolphthalein a red colour with that indicator shows that some carbonate is present, and the term "actual CaCO₃" is used to indicate this fact. The total amount of acid used is described as "total alkalinity as CaCO₃."

By the expression "the alkalinity was 2.8" we mean that the total alkalinity, expressed as parts CaCO₃ per 100,000 of water, was 2.8.

The erosion test.

This test is an estimation of the action of the water on completely immersed sheet lead. The value of the test lies in the fact that a high erosion figure is an advance warning that the water in its fluctuations may be approaching a dangerous condition. The erosion test is performed with bright lead in water with free access to air, and under these conditions a water will erode lead when it is unable to act on lead service pipes to any extent.

The kind and position of the erosion will be shown to depend on a number of conditions, the chief of which is the alkalinity of the water. Three kinds of action occur:—

1. With waters of alkalinity 0.2 to 0.6 (*i.e.*, untreated Welsh waters), and generally with treated waters the alkalinity of which does not exceed 1.5, the whole of the surface of the lead is eroded. A flocculent precipitate forms, which falls to the bottom of the tube and leaves the lead surface quite bright but mottled.
2. With treated waters of alkalinity 1.5 to 2.5, the action is usually *corrosion* rather than erosion; a loosely-held, opaque, white crust forms here and there irregularly on the lead surface. This crust appears on the upper surface of the inclined lead in the ordinary erosion test, on the under surface if the lead is placed horizontally and almost touching the glass, and on both sides if the lead stands vertically.
3. With waters of alkalinity 2.5 and over the lead almost invariably becomes covered with a dull whitish film, which appears to act as a protective coating.

Method of performing the erosion test.—The method adopted in all experiments where the details are not specified is that outlined by Dr. Houston:—

Commercial sheet lead in strips $\frac{1}{2}$ inch wide and about $\frac{1}{8}$ inch thick (one square foot weighs 13 lb.) is scraped bright with a plumber's shave hook, and cut into lengths of one inch. Each piece is rubbed with a clean duster kept for the purpose and dropped into the water without touching with the fingers. Test-tubes 6 inches by $\frac{3}{4}$ inch, reserved for this purpose only, are washed with tap water and allowed to drain.

10 c.c. of the water is poured from a cylinder into the test-tube and the strip of lead polished

with a cloth and dropped into it. The lead is now totally immersed in the water. The tube is shaken to remove air-bubbles from the surface of the lead and allowed to stand in a cupboard for 1, 3, 7, or 14 days.

(a) *If there is no evidence of erosion* the water is decanted into a 50 c.c. Nessler cylinder and the lead strip shaken with about 10 c.c. of distilled water, which is poured into the cylinder. The lead is then removed and the tube washed with 5 c.c. of dilute acetic acid (roughly decinormal) and with water until there is 50 c.c. in the cylinder.

(b) *If erosion is doubtful* the water is decanted into a 50 c.c. Nessler cylinder, the lead dropped into a funnel, and any eroded lead washed off with a jet of water into the cylinder. Dilute acetic acid is used as before.

(c) *If there is evident erosion* the turbid water is decanted into a 100 c.c. flask. The lead is shaken out into a funnel and washed with a jet of distilled water. 10 c.c. of the dilute acetic acid is put into the tube and allowed to stand a short time before it is emptied into the flask. The tube is washed with more distilled water and the flask filled up to the mark. When the solution is quite clear 50 c.c. or a smaller measured quantity is put into a Nessler cylinder and made up to 50 c.c.

Comparison is made in 50 c.c. cylinders. 5 c.c. of the dilute acetic acid (or a quantity sufficient to make the amount present up to 5 c.c.) and 3 c.c. of saturated hydrogen sulphide solution are added to every cylinder. The colour produced by the colloidal lead sulphide is matched by standards prepared at the same time. The standards are made with distilled water, except in the case of very dark waters, when it is advisable to include 10 c.c. of the original water.

The standard lead solution used is of such a strength that 1 c.c.=0.0001 gm. Pb, and is prepared from a solution a hundred times its strength, which contains 1.831 grms. of lead acetate and 10 c.c. of N/1 acetic acid per 100 c.c. Standards may be increased by 0.1 or 0.2 c.c. of the lead solution after the hydrogen sulphide has been added, but these standards are not so reliable as those to which the hydrogen sulphide is added last.

Alteration of duration of the erosion test.—At the beginning of the investigation the times of exposure of the lead to the water were 7 and 14 days, but as the figures obtained were almost identical, the 14 days' test was discarded. Thus of the 56 samples of alkalinity 2.5 to 5.1 examined during Dec., 1905, Jan., Feb., and March, 1906, 24 gave the same figure for both 7 and 14 days; the greatest difference obtained was: 7 days 0.3, 14 days 0.7 (alkalinity 3.0). The average figures for the 56 samples were alkalinity 3.1, 7 days' erosion 0.33, 14 days' erosion 0.37.

Dr. Houston suggested at least a week (Vol. 2, p. 444) as the period for this test. This is a long time to wait before determining if a water possesses high erosive ability, and experiments were performed to find if one day would not serve the same purpose. The waters used were mixtures of an untreated water (alkalinity 0.5) and a treated water (alkalinity 2.8).

The average of the results obtained from 166 samples of Frankley water in 1909 was 0.28 for

one day and 0.45 for seven days. We conclude from an examination of our figures that the one-day test gives as good an indication of erosive ability as the 7-days' test, and since the 1-day test enables us to put on repetitions whilst the water is fresh, we have adopted it for general use.

Table 1.—Comparison of 1- and 7-day erosion.

Alkalinity as parts CaCO ₃ per 100,000	Erosion (lead per 100,000)	
	1 day.	7 days.
1.4	4.4	9.0
1.7	1.7	2.0
1.9	0.3	0.3
2.1	0.3	0.4
2.3	0.2	0.3
2.6	0.2	0.3

By the expression "the erosion figure was 1.6" we mean that $\frac{1}{2}$ square inch of lead exposed to 10 c.c. of the water for one day gave dissolved and undissolved lead equal to 1.6 parts per 100,000 of water.

Irregularities in the action of water on lead.

The erosion test was always done in duplicate, and whilst as a whole the duplicates agreed well, there were occasional striking disagreements. The colorimetric comparison which is used for the estimation of the lead is liable to give some difference. The possible error on a single determination is about 10%; that is, if there is present 1.0 part per 100,000, one may find 1.1 parts or 0.9 part per 100,000, but the differences obtained were far in excess of this. Thus one water, with an alkalinity 2.0, gave 16.0, 3.2, 2.4, and 10.0. It is noteworthy that these disagreements are so rare as to be negligible with all waters except those with an alkalinity between 2.5 and 1.5. A number of experiments were performed with a view to preventing these occasional disagreements.

(1) The 10 c.c. of water was transferred to the test-tube by means of a cylinder instead of a pipette (to prevent possible contamination of the water with saliva or the breath) without advantage.

(2) As it is sometimes impossible to shake off minute air bubbles from the lead, brushing the lead free from bubbles was tried, but poor duplicates were still obtained. Also, large bubbles were allowed to remain on the lead; they did not appreciably affect the amount of erosion.

(3) In some experiments on the influence of contact with glass of test-tubes ordinary tubes were compared with those which had been coated internally with a thin layer of paraffin wax. The mean alkalinity of the water was 2.3. The results showed that the paraffin-coated tubes gave no better duplicates than ordinary tubes, and that the erosion figures in paraffin-coated tubes were higher (mean of 13 tests: paraffin-coated 4.7, ordinary 3.8).

(4) That falling dust particles or bacteria are not the cause of the disagreement of duplicates was shown by plugging the tubes with cotton-wool. It is interesting to note that, as with open tubes, if the erosion was of the crust-forming type, it still occurred with plugged tubes on the upper surface of the lead.

(5) Experiments showed that the careful selection of tubes of the same internal diameter did not prevent disagreements of duplicates if the water had an alkalinity in what may be called the critical range (1.5–2.5).

(6) The method of removing eroded lead from the lead strip by a water jet is not ideal, and an attempt was made to remove the eroded lead completely by stroking with a glass rod covered with rubber tubing. More lead was obtained, but there was no improvement in duplicates.

(7) The following points were demonstrated by the late Mr. J. A. Brown. (a) It is essential in the erosion test that the lead be dropped into the water and not put in a moist tube and the water then run in. The preliminary exposure of lead to moist air greatly increases the erosion figure. (b) Filtration of the water made no difference to the erosion figure, the presence of the ordinary organic deposit having little or no effect.

(8) *Conclusions concerning irregular action of water on lead.*—We have now shown that none of the following is the main cause of the occasional disagreement of duplicates in the erosion test: (1) air bubbles, (2) glass or diameter of test-tube, (3) dust particles or bacteria, (4) method of removing eroded lead, and (5) presence of suspended matter in the water. The subject is of interest, and might be of practical importance, because in test-tubes put on side by side under apparently identical conditions we have considerable difference in the velocity of reaction. Is it possible that these differences are due to the presence and absence of minute quantities of special electrolytes or of enzymes?—or to galvanic currents set up in the lead by metallic impurity which occurs in one inch but not in another? In this connection it will be remembered that commercial lead is a remarkably pure substance, containing on an average about 0.01% of impurity; this consists mainly of metals such as bismuth and antimony, and contains very small amounts indeed of non-metallic substances which on oxidation would produce acids. With regard to the disagreement of duplicates it should be noted that for any particular water they only occurred with those waters within a narrow range of alkalinity, and for all the waters examined within a range of alkalinity 1.5–2.5. Now it will be observed that this is the identical range which generally gives that type of action which we have called corrosion, i.e., the formation of irregular patches of a loosely-held white crust. (We find a close parallel to this in that unequal distribution of rust noticed by Heyn and Bauer (Mitt. K. Materialprüf., 1908, 26, 1–104) which occurs when iron is exposed to water containing a certain amount (0.1%) of potassium carbonate. With waters containing less than this amount there is an even rusting all over, and with waters containing more than this amount there is no rusting). Hence the simplest explanation of the disagreement of duplicates is that waters of alkalinity 1.5–2.5 are just able to coat the lead with a protective layer, and hence the smallest amount of irregular coating leaves a clean surface exposed which the water attacks vigorously. (For further support of this, see the effect of dissolved solids on erosion.)

Determining factors in erosion.

In some cases there is difficulty in drawing general conclusions, because the action appears to follow different laws according as it is erosion proper, corrosion, or formation of protective coating.

(1) *Time.*—The velocity of the reaction falls as time proceeds. Waters of low alkalinity (up to 1.5) show a greater amount, and waters of alkalinity 2.5–3.0 show slightly great amounts of total erosion, with increase of time (see Table 1). The waters of intermediate alkalinity (1.5–2.5) are irregular in action, and may reach their maximum in one day.

2. *Light.*—The action of light is to increase the amount of the action of water on lead. Ten samples of untreated water gave an average of 16.8 of lead when the tubes were exposed to daylight, and an average of 11.9 when the tubes were kept in a dark cupboard during the day of the experiment.

(3) *Water volume.*—In these experiments pieces

of lead 1 inch by $\frac{1}{2}$ inch were used in all cases. Different sized vessels were used and different amounts of water, but the height of the water-surface above the top of the lead was carefully arranged in all cases to be 1 inch.

Table 2.—Relation of erosion to water volume.

Alkalinity of water 2.0		Time of test 1 day.	
Area of water exposed to air sq. cm.	Volume of water c.c.	Lead per 100,000 of water	Total mgrms. of lead (1 sq. in. of lead exposed).
1.5	7	12.1	0.9
2.0	10	10.3	1.0
2.5	12	7.3	0.9
4.5	22	4.5	1.0
1.2	30	2.3	0.9
13.8	63	1.7	1.1
3.1	78	1.5	1.2

With waters that erode freely variation in the volume of water does not affect to any extent the total amount of lead eroded. A large increase in the amount of water appears to cause a slight increase in the total lead eroded.

(4) *Area of lead exposed.*—Dr. Houston (Vol. II., 40), performing the ordinary erosion test with distilled water and strips of lead 1 inch long and $\frac{1}{2}$ or $\frac{1}{4}$ inch wide, found that such variations did not suffice to bring out to any appreciable extent a difference in the amount of lead eroded. On repeating this experiment under similar conditions with untreated water (alkalinity 0.5) we came to a similar conclusion. However, a more carefully devised experiment gave a more decided result.

In this experiment the strips of lead were parts of a cylinder of 1 inch in diameter and rested at right angles to the bases of small glass dishes. Untreated water (alkalinity 0.4) was used, and the amount of lead eroded estimated after 4 hours.

Table 3.—Relation of erosion to area of lead.

Length of lead strips in inches.	Total area of lead strip in sq. in.	Lead per 100,000 parts of water.	Mg. of lead eroded from 1 sq. in. of lead.
With 40 c.c. of water.			
$\frac{1}{2}$	0.62	2.5	1.6
1	1.19	5.3	1.8
2	2.30	10.6	1.8
3	3.44	13.1	1.5
With 80 c.c. of water.			
1	1.19	2.4	1.6

These results show that with water of low alkalinity the amount of lead eroded varies directly with the area of lead exposed, and (as we also showed above) is independent of the amount of water used. It should be observed that in this experiment the water surface exposed to the air was large, and that the results might have been different had the supply of oxygen been less readily available (as it is in closed lead service pipes).

(5) *Area of water-surface exposed to the air.*—We found when working with small strips of lead that, within a considerable range, the area of the water-surface exposed to the air did not produce any appreciable effect on the erosion; this is shown in Table 2 on the relation of erosion to water volume. Doubtless if the water-surface was greatly reduced then the lead eroded would be smaller (see figures for erosions in closed tubes).

(6) In all our tests the lead was completely immersed, and we noted that the less the distance of the lead from the surface of the water the greater the erosion. Thus, in one experiment, the distance

was 21 mm., lead 0·8; 16 mm., lead 1·2; 14 mm., lead 5·0.

(7) *The use of sterile tubes and sterile lead.*—In this experiment untreated water (alkalinity 0·4) was used, and a number of cotton-wool plugged tubes each containing the usual 10 c.c. of water and bright lead strips were made sterile by heating for one hour in a Koch's steam steriliser. From one set the water was poured out (the amount of lead found in this water was 2·8 parts per 100,000) and 10 c.c. of fresh water put in. Another set was left untouched; both sets were examined at the end of one week.

Table 4.—Erosion with sterile materials.

Tubes and lead.	Water.	Erosion test, figure, lead per 100,000.	Mean figure, lead per 100,000.
Untreated	Fresh	53·0	
Sterile	Fresh	58·0	
Sterile	Sterile	18·0	

The above figures show (a) that the sterility of lead and tubes is not a dominant factor in determining the amount of erosion, and (b) that erosion occurs in the absence of bacteria. The probable reasons why the erosion figure with sterile water is so much less than with fresh water will be discussed later under the effect of dissolved gases.

It might be thought that with erosive waters the above test under sterile conditions was unnecessary, that the water in the erosion test must always immediately become sterile, since it is in contact with bright lead. On the contrary, we found that exposure of the water to bright lead for 6 hours did not make the water sterile, 497 micro-organisms being grown in jelly in four days from 1 c.c. of water. A similar quantity which had not been exposed to lead was liquid in that period, so lead has a considerable inhibitory power on liquefying organisms. Other experiments showed that 1 mgrm. of lead in solution present in about 12 c.c. of jelly has little effect on the number of organisms that will grow in jelly, but that 10 mgrms. of lead produces sterility.

(8) *The effect of exposure to glass.*—It was in general found that exposure to glass, whether of bottle or test-tube, lowered the erosive ability of the water. Thus a water of alkalinity 1·9 gave, after standing in the bottle 1 day, 9·5; 4 days, 0·5; 8 days, 8·3; 11 days, 0·4; 15 days, 0·6; 18 days, 0·4; 22 days, 0·3; 29 days, 0·2 (parts of lead per 100,000). It would appear from this that the passage of water through filters of broken glass would reduce its erosive ability.

Some test-tubes were found (by the late Mr. J. A. Brown) to give an alkaline reaction to phenolphthalein after standing with water for some time. These gave lower erosion results than ordinary tubes. It should always be remembered that the "erosion test" is not simply an estimation of the action of water on lead, but of water on lead in the presence of glass, although in normal tubes, as far as our results go, they indicate that for one-day experiments the order of the erosive abilities of various waters is not affected by the presence of the glass.

(9) *Gases dissolved in the water:*—

(a) *Oxygen.*—Traube-Mengarini and Scala (this J., 1910, 700) have shown that distilled water acts appreciably on lead in the absence of oxygen, the lead passing into the water in a colloidal form. With our water the presence of oxygen appears to be a necessity, at any rate if the erosion is to be considerable. We first investigated as to whether the notable difference in the erosive ability of the untreated water and the treated water was due to difference in the amount of oxygen they contained.

Table 5.—Relation of erosion to dissolved oxygen.

Water.	Alkalinity as parts CaCO ₃ per 100,000.	c.c. oxygen per litre.	7 days' erosion, Lead per 100,000.
Untreated water	0·3	7·8	58·0
Treated water (from reservoir)	2·9	7·8	0·4

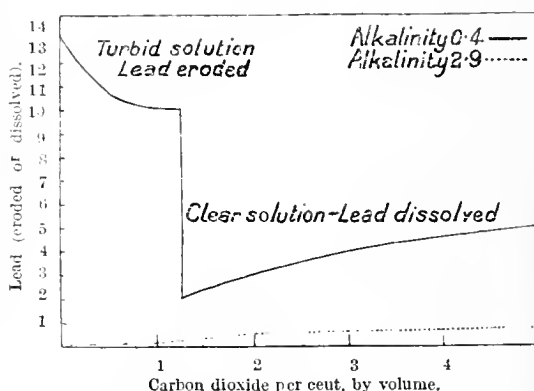
Evidently the great difference in the erosions is not due to differences in the amount of oxygen present. (Oxygen is not so soluble in water containing dissolved salts as in pure water, but the quantity of dissolved salts present here would not produce any appreciable difference in its solubility.) However, the following experiment shows that the volume of oxygen dissolved in the water limits the amount of lead eroded if no more oxygen be admitted during the reaction. (This contradicts the statement of Reichardt, Arch. Pharm., 25, 858.) In this experiment glass tubes with well-fitting stoppers were used, completely filled with untreated water (alkalinity 0·3).

Table 6.—Open and closed tubes.

7 days' erosion. Lead per 100,000.			
Volume of water in tube.	c.c. oxygen per litre.	Open tube.	Closed tube, full of water.
c.c.			
13·5		16·5	12·5
14·5		17·5	12·0
13·5	7·8	24·0	15·0
14·0	7·8	24·0	12·0

These results have an important bearing on the erosion of lead service pipes, for they show that in closed tubes the amount of lead eroded only corresponds to the maximum amount of plumbous oxide or salt that could be produced by the oxygen present (thus 7·8 c.c. per litre of oxygen gives 14 parts of plumbous lead per 100,000). They also indicate that in the ordinary open tube erosion test oxygen is absorbed from the air by the water during the process. This affords an explanation of the effect of the distance of the lead from the water surface.

Influence of Carbon Dioxide on Action of Water on Lead.

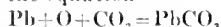


(b) *Carbon dioxide.*—The carbon dioxide for these experiments was prepared by dropping pure dilute sulphuric acid into pure sodium bicarbonate. The gas was washed by bubbling through distilled water. The curve below summarises the results obtained. All the erosions were determined under the usual standard conditions of 24 hours. The waters were various samples of untreated water with an

alkalinity of 0.4 into which carbon dioxide had been passed. (It is worthy of note that on passing the carbon dioxide through the water a decrease in colour was observed. It has been suggested that the passing of air through the water decreases the colour by oxidation. The decrease in the above experiment cannot be due to oxidation, and it is possible that the colouring matter is of a colloidal nature and is precipitated by the carbon dioxide.)

The above curve shows that for waters of low alkalinity (0.4) the increase of carbon dioxide produces little effect until as much as 1% is present, so that in all probability with our ordinary treated and untreated waters carbon dioxide is not an important factor in erosion. From 1 to 2% of carbon dioxide causes a sudden change, for this quantity is sufficient to prevent "erosion" and to cause "plumbo-solvency," i.e., the lead precipitate ceases to appear, lead bicarbonate being soluble. It is noteworthy that with this change there is a sudden fall in the total amount of lead found in the water (compare Müller, J. Chem. Soc., 1888, 54, 225, who appears to have thought there was no action since there was no visible erosion.)

According to the equation—



two volumes of carbon dioxide are required to one of oxygen, and when this ratio is exceeded some bicarbonate will be formed. In the water experimented upon we have about 0.8% of oxygen present, so that we might reasonably expect the type of action to change as soon as the carbon dioxide exceeded 1.6%. As the percentage of carbon dioxide present increases above 2% the amount of lead dissolved increases, but even with 20% present does not become as great as the original figure for lead eroded.

Table 7.—Influence of carbon dioxide on erosion with water of alkalinity 1.9 to 4.0.

Alkalinity as CaCO ₃ per 100,000	Carbon dioxide % by vol.	Erosion, Lead per 100,000 in 1 day.	Description.
1.9	trace	1.1	Erosion
2.1	11.4	4.0	Plumbo-solvency
2.9	trace	0.1	Erosion
2.9	1.0	0.2	Plumbo-solvency
2.9	2.0	0.6	Plumbo-solvency
2.9	10.0	1.6	" "
2.9	20.0	3.2	" "
4.0	trace	0	" "
4.0	11.4	2.3	Plumbo-solvency

With the exception of the first the waters given above, even when they contained only a trace of carbon dioxide, were of sufficient alkalinity not to "erode" lead. As with the waters of alkalinity 0.4, the addition of carbon dioxide produced plumbo-solvency, but with these waters of higher alkalinity more free carbon dioxide was needed to dissolve the same quantity of lead. The figures are sufficiently high to show the danger of allowing soda water to remain in contact with lead pipes.

Table 8.—Influence of carbon dioxide in presence and absence of air.

	Stoppered tube.	Open to air.
	Lead in one day.	
Original water	—	11.0
Boiled one hour	0.6	0.7
Boiled water with 32 v. v. CO ₂	1.6	3.5
	Lead in two days.	
Boiled one hour	0.8	0.8
Boiled water with 32 v. v. CO ₂	4.0	10.0

The alkalinity of the water was 0.5. We see from the above:—

1. That if the water is boiled, the lead put in it gives an erosion of only 0.7 (Houston found a similar result). It is evident from this that it is not a fixed non-volatile acid which causes erosion. It was found that if water were boiled for 1½ hours and then allowed to cool for ½ hour it contained 0.6—0.7 c.c. of oxygen per litre, which is quite sufficient to account for the 0.8 of lead obtained with the closed tube. That boiled water of alkalinity 0.5 should give so low an erosion as 0.7, although freely exposed to the air, is a little remarkable. The boiled water appears to have rendered the lead "passive." The appearance of the lead was quite peculiar; it was still bright, but had a slight bronze tarnish. This may have been a thin film of red lead, and would seem to support the conclusions of Dunstan and Hill concerning passivity (Chem. Soc. Trans., 1911, 99, 1853—1866).
2. The figures above also show that water containing carbon dioxide does not dissolve lead so rapidly if a supply of oxygen is not available. This is to be expected, since the hydroxide of lead is more readily soluble than the metal in carbonic acid.

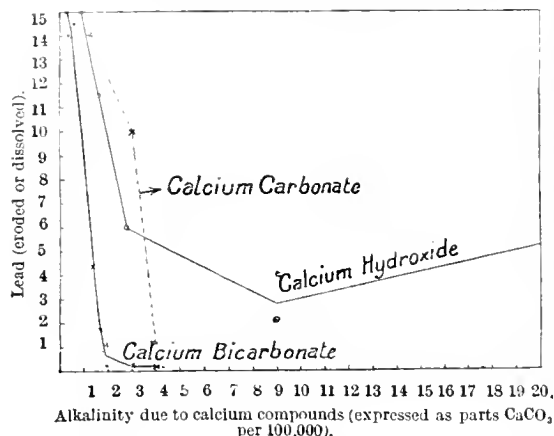
The question may be asked if the presence of carbon dioxide is necessary for erosion to occur. All the evidence we have goes to show that it is not necessary. It will be shown later that erosion takes place readily in the absence of free carbon dioxide; for example, when 1 to 200 parts of calcium hydroxide per 100,000 are present (see curve below). And further, erosion takes place less readily in the presence of bicarbonate and carbon dioxide than with carbonate and hydroxide alone. Under normal conditions for ordinary waters carbon dioxide is an unimportant factor. We have seen that variations in the amount of carbon dioxide from 0.02 to 0.20% do not affect the erosion figure to any extent.

(10) *Liquids dissolved in the water.*—In a number of erosive waters that Allen examined he found free sulphuric acid. This could not be present in our water, for when our water showed acidity to phenolphthalein this acidity was always lost on boiling. This acidity is due to carbon dioxide, and was generally equivalent to about 0.02% of gas. Although the general principles underlying erosion were stated by Müller as early as 1888, in recent years it appears to have become a commonly accepted idea that natural waters erode lead by means of the peat acids which they contain. Our water was always alkaline to lacmoid; that is, it always contained free alkali at least as strong as bicarbonate. Further, a large number of samples were alkaline to phenolphthalein, and many of these eroded lead, so that in these cases at any rate the erosion was not due to free organic acids. The assumption of organic acids as the cause of erosion is quite unnecessary, since water, oxygen, and carbon dioxide will readily account for the observed phenomena.

(11) *Solids dissolved in the water.*—We were principally interested in these with a view to the most effective treatment of the water to prevent erosion. For example, nitrates are said to accelerate erosion. As our water never contained more than a minute trace we neglected them.

A number of experiments were performed with calcium hydroxide and carbonate and bicarbonate. Pure lime was dissolved in the untreated water, and this was mixed in various proportions with untreated water through which carbon dioxide had been passed. With these mixtures the standard erosion test for one day was made with the following results:—

Influence of Lime and Calcium Carbonate and Bicarbonate on Erosion.



The results given above show that:—

1. As the amount of *calcium hydroxide* present in the water increases, the amount of lead eroded falls to a minimum when there is present about 7 to 10 parts of calcium hydroxide per 100,000. After this point increase in the calcium hydroxide causes increase in the erosion, which gradually attains about its original amount.
2. As the amount of *calcium carbonate* and *bicarbonate* present in the water increases, the amount of lead eroded continually falls.
3. *Calcium bicarbonate* is by far the most effective preventer of erosion. Only about half the quantity of bicarbonate is required to form a protective coating as compared with carbonate. This would receive a theoretical explanation if, as seems probable, the coating is a carbonate of lead. As little as two parts per 100,000 of calcium bicarbonate is generally sufficient to form a plumbo-protective coating on the lead. Assuming that the actual coating is lead carbonate, it would form a layer only 0.000016 cm. thick. As suggested above, the irregularities that are observed in the erosion with water about this alkalinity may be due to the fact that the deposit does not quite cover the lead surface and leaves a little bright lead open to the ordinary erosive action.

All these results refer to calcium compounds; but, considering that the solutions are dilute, it is probably safe to infer that the results would be similar with all metals of the alkalis and alkaline-earths, and for similar concentrations of CO₂ and OH ions.

The untreated water taken from the Caban reservoir in Wales has an alkalinity of about 0.2, the erosion for one day is about 14. From December 6, 1905, to January 31, 1906, the alkalinity of the treated water at the Birmingham reservoir at Frankley was never below 2.7, while the erosion in seven days only exceeded 0.5 on three occasions, and that slightly. On the other hand, from May 12 to June 17, 1906, the alkalinity of the water at Frankley was uniformly low, never rising above 1.8. During that period the erosion of the water was high, being only as low as 0.5 on four occasions, while it frequently reached 2.0, and rose as high as 18.0. These results show that as a rule water of high alkalinity has a low erosion and *vice-versa*.

As potassium permanganate is sometimes put into water we investigated its effect on the erosion of

lead. We found that as little as 5 parts per 100,000 of water were sufficient to form a protective coating and prevent erosion.

Conclusions concerning erosion.

1. The waters collected in the Caban reservoir, Elan Valley, Wales, have a slight alkalinity due to bicarbonates etc. equal to about 0.3 part CaCO₃ per 100,000. Their action on lead is mainly "erosive."
2. "Erosion" is due to the action of oxygen in the presence of water.
3. Such variations as occurred naturally in the percentage of oxygen present in the water produced no appreciable effect on the erosion.
4. One day is the most suitable period for the erosion test.
5. Exposure to glass lowers the erosive ability of the water.
6. The greater the depth at which the lead is immersed in the water, the slower the erosive action.
7. The velocity of erosion falls as time proceeds.
8. Variation in the volume of water does not appreciably affect erosion.
9. For the untreated water the amount of lead eroded varies directly with the area of lead exposed.
10. Small changes in the area of the water surface produce no appreciable effect on the erosion.
11. Erosion readily occurs in waters which contain no carbon dioxide.
12. Such variations as occurred naturally in the percentage of carbon dioxide present in the water produced no appreciable effect on the erosion.
13. The presence of from 1 to 2% of carbon dioxide causes a sudden change from "erosion" to "plumbo-solvency."
14. Carbon dioxide dissolves lead more rapidly in the presence of oxygen.
15. Given oxygen the alkalinity of the water is the principal factor determining the amount of "erosion."
16. Not only the amount but the kind of erosion depends chiefly on the alkalinity of the water.
17. The addition of small quantities of calcium hydroxide, carbonate and bicarbonate, or of potassium permanganate, decreases erosion.
18. Calcium bicarbonate is the most effective preventive of "erosion," as little as two parts per 100,000 being generally sufficient to prevent erosion.
19. Erosion occurs in the absence of bacteria.
20. The bacteria in the water are not all destroyed by exposure to lead for 6 hours.

Discussion.

The CHAIRMAN asked if the authors had used chemically pure lead in their experiments as well as the ordinary commercial lead. There might be considerable difference found in the amount dissolved by comparing the effects on two leads. Chemically-pure lead almost entirely resisted both dilute and strong sulphuric acid. Commercial lead gave way much more readily. He also enquired what the effect of varying temperatures was on the erosion. The variations of effect of water of different alkalinity reminded one also of the irregular behaviour of acids of varying strengths in the dissolution of metals; with iron there was a much greater effect with dilute than strong acid. In the case of aluminium small amounts of impurity caused a much greater dissolution of the metal to take place; for instance, a small amount of nitrogen getting in through faulty treatment during casting greatly increased the amount of solvent action.

Mr. H. SILVESTER said that having frequently to test the action of water on lead he had found it satisfactory to adopt the Birmingham water as a standard for comparison. His experience showed that after 24 hours of exposure of new bright lead only one part per million, or less, of lead was taken up by the Birmingham supply, which he considered satisfactory, as with such a result under practical conditions none was found in the water as distributed. This was in agreement with the results given in the paper. As to the use of various chemicals to prevent lead attack, he found that where a quick result apart from cost was required, small quantities of sodium carbonate were effective, an alkalinity of 2 or 3 grains per gallon destroying the lead-solvency powers of the water.

Mr. J. RACE said that our knowledge of corrosion and erosion of lead had been more or less chaotic, and several conflicting hypotheses had been suggested. Heap (this J., 1913, 771, 811, 847), after a large number of experiments, concluded that oxygen was the predominating factor; while at a meeting of the American Water Works Association, which he had attended some years ago, the general opinion was that carbonic acid was the causative agent. The practical importance of the subject could not be over-stated. Whether iron pipes or lead pipes were used, there was the ever-increasing danger of corrosion due to electrolysis, consequent upon the larger use of electricity. He asked what was the effect of the seasonal flora in the reservoir. During certain seasons there should be a definite turnover of the water which caused variations in the oxygen and carbonic acid content and might produce a variable effect on lead. He suggested that the effect of water on lead should be studied in conjunction with hydrogen-ion concentration, as it was possible that this method would give better correlation with the amount of lead eroded and dissolved. It had often been assumed that, because a water was alkaline to the usual indicators, it contained no free carbonic acid, but recent work on dissociation has shown that this view is incorrect. If carbonic acid was a contributory factor to plumbo-solvency, a definite hydroxyl-ion concentration might be found that would arrest the action. Another point of importance was the nature of the pipe used for carrying the water. Waters that attacked lead pipes also attacked iron ones, producing what was known as "red water" trouble; they also dissolved the zinc coating of galvanised pipes, and so produced zinc poisoning. The Local Government Board had some years ago refused to sanction the use of galvanised pipes for drinking services in a large town on this account. In America these difficulties had led to the use of copper and brass pipe. It was found that the cost of installation was not prohibitive, because the absence of incrustation led to smaller friction losses and enabled pipes of smaller dimensions to be used. In the industrial area in North-East Lancashire he had found that the acid in the atmosphere gave a marked acid character to the rain water and was sufficient to produce plumbo-solvency.

Dr. E. B. MAXTED enquired as to the influence of agitation on the rate of solution of lead; agitation might loosen any protective deposit that might be formed and accelerate solution.

Mr. F. R. O'SHAUGHNESSY said that although the authors had shown that lead was acted upon by sterile water, it seemed possible that biological action might exert some influence. Had the authors investigated the question in relation to chlorinated waters? There was no object in preventing entirely lead from going into the water if the expense was so great and if small traces did not in any way prejudice public health.

Mr. J. O'SULLIVAN asked if the water was alkaline or acid at its source. In the North of England waters from a peaty source were treated with calcium carbonate to prevent the solution of lead.

Mr. ASHMORE suggested that if copper and zinc were kept entirely immersed they became passive; in other circumstances that was not so.

Mr. F. H. ALCOCK remarked that in one of the results it was shown that about one-fifth of the solid components of the water was not determinable. What was the amount of sulphate present?

Mr. KNAPP, in reply to the discussion, said that the tests were made with ordinary commercial lead, which was extremely pure—the amount of impurity probably did not exceed 0.01%. He had not made tests as to the effect of varying temperature or of agitation. As to why they did not regard erosion so serious as plumbo-solvency, he pointed out that in the severe tests which were made the water which would erode lead would not act upon lead pipes. They had not used sodium carbonate on account of cost. He agreed as to the effect of electricity in causing erosion in lead pipes. In one case a service pipe gave high figures which were entirely due to close proximity to electric wire. So far no seasonal variation in erosive ability had been found. No doubt the erosion would be much greater if the lead was only partly immersed, particularly along the line at which air, water, and lead came together. The matter which in the water analysis was classed as "undetermined" was organic.

Mr. LIVERSEEGE also replied, pointing out that they did not now rub the lead with a duster to polish it; experiments of the late Mr. J. A. Brown showed that such rubbing could not be standardised. There would be differences in the erosive action. They preferred, therefore, to scrape the lead and lightly to dust off any lead particles. Another improvement was the use of methyl red in place of lacmoid as indicator. The addition of chalk to Welsh water was useful as, in addition to preventing any risk of lead poisoning, it also reduced the corrosive action of the water on iron. There was a difference as to the importance to health, of erosion and plumbo-solvency, and he thought this was shown by the fact that in some Yorkshire towns there had been a number of lead-poisoning cases which, as far as he knew, had been due to plumbo-solvency, and not to erosion. In some cases untreated water was used which was decidedly erosive, and the medical officer stated that he had had no trouble from lead poisoning. He thought there was considerable difference in the importance of the two actions of lead, and that the Birmingham water was free from any risk of plumbo-solvent action. The Walker-Kay test was not useful in relation to the Birmingham water. There was very little peat in the Elan Valley, and the result was a water that was neutral or slightly alkaline.

Communications.

VAPOUR PRESSURE OF ARSENIOS OXIDE IN SULPHURIC ACID SOLUTIONS.

BY F. SCHWERS.

An important item in the manufacture of sulphuric acid by the contact process is the purification and drying of sulphur dioxide, which includes bubbling through sulphuric acid. In the factories erected by the Ministry of Munitions an acid containing 0.001% As_2O_3 was at first deemed sufficient

for this purpose. But it was soon found (1916) that the activity of the catalyst decreased rapidly, and suspicion arose as to whether part of these minute traces of white arsenic had not found their way to the catalyst. Mr. K. B. Quinan, who had charge of these plants, asked Professor F. G. Donnan to investigate the matter. I undertook the work at the latter's suggestion, and wish to thank him for his advice.

At first sight it appeared rather unlikely that under the working conditions any measurable quantity of arsenious oxide could ever be volatilised during the passage of the gas, all the more as Adie states that arsenious oxide forms a compound $\text{As}_2\text{O}_3 \cdot \text{SO}_3$ in presence of sulphuric acid. Since we have no figures for the tension of As_2O_3 below 150°C . and none in sulphuric acid solution, this point could only be decided by experiment. From existing vapour pressure data for As_2O_3 from 150°C . upwards no extrapolation could be made down to 60°C . with any chance of accuracy.

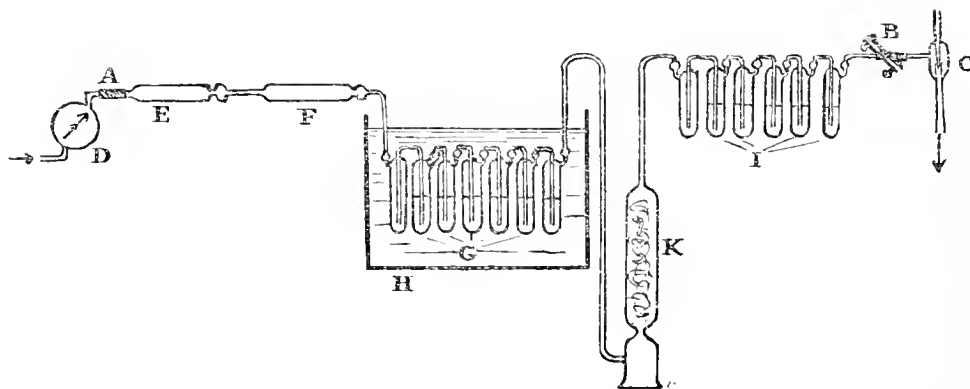
A dynamical method was resorted to as the only one appropriate to the estimation of such low vapour pressures as were expected. A glass apparatus was fitted up through which air was slowly aspirated by a water suction pump, C, carefully regulated by a pinchcock, B. The volume of air was measured by a gas-meter, D, it was then dried over calcium chloride, E, freed from carbon dioxide by caustic soda, F, after which it passed through six bubblers, G, partly filled with sulphuric acid containing minute quantities of arsenious oxide. These bubblers were immersed in a water-thermostat, H, kept at about 60°C ., which is the temperature to which the washing acid rises in a large-scale plant when the hot gases are bubbling through. Then came another set of absorption tubes, I, half filled with a 10% caustic soda solution, to retain any arsenious oxide volatilised. The shape of the apparatus was

16.7°C .) The average temperature of the thermostat was 59.4°C .; since the passage was very slow, we may admit that the air was soon in temperature equilibrium with the acid, and that 2.08 cubic metres of air at 59.4°C . actually bubbled through the acid. No record of the barometric pressure was taken, but one may take it that the average was close to 760 mm.

The estimation of arsenic was made by Thorpe's electrolytic method.* It was first ascertained in this way that the caustic soda used was absolutely free from arsenic; in fact, no trace of it could be detected. But at the end of the experiment the alkali was found to contain 0.00040 gm. As_2O_3 , which had been carried over by the air. From this it is calculated that the partial pressure of arsenious oxide over a 0.015% solution in sulphuric acid is slightly above 0.00001 mm. of mercury at the temperature of 60°C .

Though very small in the absolute sense, the quantity of As_2O_3 carried away seems large under the circumstances. In order to check this first result a second experiment was started by filling the bubblers with a sample of the very acid used in the factory, without any further addition of arsenic beyond the 0.001% it already contained. In the course of 1390 hours there passed 3.36 cubic metres measured at 15.0°C . (=3.93 cubic metres at 61.0° , average temperature of the thermostat). This time 0.000050 gm. As_2O_3 was found in the caustic soda solution, i.e., 0.000013 gm. per cubic metre at 61°C .

By comparing with the first experiment, where the arsenic concentration was 15 times greater, it is seen that the amount of arsenic carried away as vapour is almost exactly 15 times larger. Such remarkable agreement was hardly to be expected, and is partly due to chance; in fact, the probable



so devised as to prevent any actual spraying of acid into the absorbing part (see fig.). Moreover, a vertical column of glass-wool, K, was interposed between the acid and the alkali bubblers. The first absorption tube of each set was put the other way round, so as to act as a safety valve in case of back pressure. The whole glass apparatus between A and B was sealed together; there were only two unavoidable rubber connections, one to the gas-meter, the other to the water-pump.

Two experiments were carried out.

In the first experiment a little white arsenic was added to sulphuric acid (sp. gr. 1.84), and the solution containing 0.015% As_2O_3 (as found by analysis) was put into the bubblers. Air was passed during 660 hours at the rate of 1 to $1\frac{1}{2}$ bubble per second. The volume of air recorded by the gas-meter was 1.80 cubic metres (average temperature

error in the analysis of such minute quantities of arsenic may be put at about 10%).

It is now interesting to form an idea of what this means in actual practice. Let us assume, for sake of argument, that the gas to be washed through sulphuric acid is the exact mixture ($2\text{SO}_2 + \text{O}_2$) necessary to make SO_3 . If we disregard water vapour and other impurities and assume the law for perfect gases, it is calculated that 8000 cubic metres of the gaseous mixture† passing through the drying acid with its 0.001% As_2O_3 will carry away as much as 0.1 gm. of arsenious oxide. Only a small part of this is necessary to "poison" the catalyst.

University College, London.

* By courtesy of Sir James Dobbie, F.R.S., the arsenic determinations were made by the Government Laboratory, London.

† Corresponding to 19.3 tons of 100% H_2SO_4 .

THE DETONATION OF HOLLOW CHARGES.

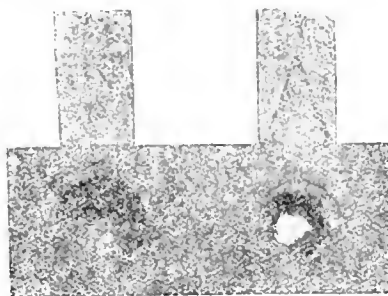
BY A. MARSHALL.

In my book on Explosives (1st Ed., 1915, p. 321; 2nd Ed., p. 554) I have mentioned the increased effect that is produced when an unconfined cylinder of a high explosive is hollowed out. This discovery I ascribed to Egon Neumann, as he definitely claims it in the *Zeitschrift für angewandte Chemie* of November 24, 1911, for himself and his co-workers. He says: "We have found in the last few months that if a hollow be made in an explosive cartridge on the side towards the object to be blasted the effect is increased four- or five-fold." The *Westfälisch-Anhaltische Sprengstoff-A.-G.* took out patents for the discovery (Ger. Pat. Anm. W. 36,269 of 14.12.1910; Eng. Pat. 28,030 of 13.12.1911), and in the *Zeitschrift für das gesamte Schiess- und Sprengstoffwesen* of May 15, 1914, Neumann describes it as "einen ganz neuen Weg." I have, however, learned from Professor Charles E. Munroe, of Washington, that he made this discovery many years ago and gave it a wide publicity in America (Scribner's Magazine, 1888, 3, 563-576; Amer. J. Sci., 1888, 36; Popular Science Monthly, 1900, 56, 300, 441). I therefore now wish to give the credit where it is due. Professor Munroe carried out a number of interesting experiments with these charges and found that the more he hollowed out the face resting on the iron plate the greater was the depth of the depression formed, until, when the charge was completely perforated, it perforated completely the iron plate. When he interposed such articles as a piece of lace or the leaf of a tree or a coin between the hollow charge and the plate he obtained a detailed impression of the article on the iron, the raised portions of the objects forming raised impressions. The effect is much the same when the cartridge is removed some distance from the surface of the iron or steel plate, and under water the results are similar to those in air. By tying a number of dynamite cartridges round an empty tin and firing them with a priming charge, also of dynamite, Professor Munroe was able to make a considerable hole in a stout safe, whereas a similar charge made up in the ordinary way merely produced a concavity, but no perforation (Executive Document No. 20, 53rd Congress, 1st Session, Washington, D.C., 1894).

No practical use has apparently been made of this discovery, but it is of interest as throwing a light on the nature of the detonation wave. It is evident that, although the products of detonation are gases and have a very high temperature, they must possess in the wave-front a density even greater than that of the solid explosive. The wave, consisting of such gas constantly renewed, advances through the explosive with a velocity of several thousand metres a second. Where the wave is in contact with the boundary of the explosive the gas flies off at right angles to the boundary and a fresh wave is formed of compressed air, or whatever other material the surrounding medium consists. This secondary wave is inclined to the original one, but advances much in the same way as the bow-wave of a ship. In the axis of the hollow of one of these bored-out charges the waves of highly compressed air come together with enormous violence, and necessarily produce a blast in the same direction as the original wave of detonation. This is not only much more intense than the original wave, because it is more concentrated, but it also lasts longer, with the result that the metal plate is carried right away. As one would expect, a charge with a cylindrical hollow bored in it produces quite as deep a hole, if not deeper, than one with a conical hollow. Although a

hollow charge produces a much greater local effect, it does not exert nearly so great a total shock as a whole charge, as is shown by the fact that when detonated on a lead cylinder it does not compress it to the same extent.

Section of complete cylinder 310 g. TNT. Section of hollow cylinder 247 g. TNT.



Wrought iron plate 25 mm. thick on which both charges were detonated in upright position.

Effect of hollowing-out a charge.

Although a detonation wave resembles a sound wave in some respects, it also differs in important properties. One of these is that the gases permanently change their position and do not simply swing forwards and backwards as in a sound wave. It constitutes in fact what French investigators have called a "discontinuity," because the rise in pressure is quite sudden, but of course it is more intense than a discontinuity produced by other means. When two waves of detonation meet the two opposing layers of compressed gases are obliged to spread out sideways, making a distinct line on the supporting plate. This forms the basis of Dautriche's ingenious method of measuring the velocity of detonation by means of a piece of detonating fuse.

RAPID METHOD OF ESTIMATING LEAD IN CASSIA OIL.

BY O. F. LUBATTI.

It is a well-known fact that cassia oil exported from China is contaminated with lead because the oil is sold on this market enclosed in leaden cans. It is usual to report the lead contained in such oils as "traces," the test employed being the addition of a few drops of ammonium sulphide to a solution of cassia oil in 70% alcohol. The reagent gives under these conditions a decided coloration, showing that the oil contains more than mere traces of the metal. The author, therefore, has tried to improve upon this qualitative test so as to render it of a more informative nature.

The principle is very simple and consists in the usual method of estimating lead colorimetrically by means of a solution of ammonium sulphide, which is usually attributed to Pelouze.* The fact that the reaction occurs in an organic liquid and that no other metallic impurities are present is instrumental in giving an extremely subdivided precipitate which seems to be very adapted for this kind of determination.

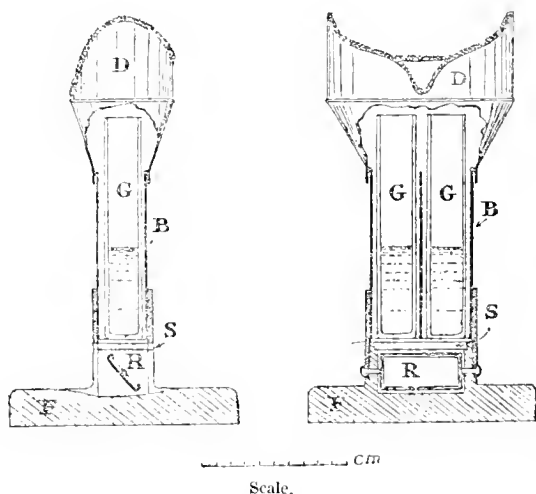
Procedure.—5 c.c. of the cassia oil to be tested is diluted to 20 c.c. with 90% alcohol; 5 or 2.5 c.c. of this solution is taken for the estimation according as the lead content is more or less than 0.025%, which will be determined by a preliminary test. This amount is pipetted into a 50 c.c. Nessler glass of small bore (2.5 cm.) and diluted to about 20 c.c.

* I. J. Pelouze, Ann. Chim. Phys., 1841, (3) 79, 103.

with 90% alcohol. A blank from lead-free cassia oil, obtained by distilling the commercial oil, is prepared in similar way. 1 c.c. of ammonium sulphide solution is added to both glasses, and standard lead solution (in 90% alcohol; 1 c.c. = 0.0001 grm. Pb.) added to the blank until the brown coloration produced matches the sample under examination, the liquid being stirred three times after each addition of standard lead solution by means of a piston-shaped glass rod.

The results are calculated by means of the formula $x = n \times 0.04 \div m$, where x is the percentage of lead sought, n the number of c.c. of standard lead solution required, and m the amount of original solution taken.

The tint of the solutions can be compared by looking through the Nessler glasses resting on a white tile. But better results are obtained by using a Dubosq colorimeter or the apparatus described below.



The colorimeter (see fig.) consists of a wooden foot, F, in which a white tile reflector, R, turning on a pivot is situated, above this is the body, B, of the instrument, which consists of a tube of CD section in which are introduced the Nessler glasses containing the liquid under observation. On top of the glasses is a device, D, to fit the face of the observer, similar to a stereoscope without lenses. When the face of the observer is applied to D the light can pass only through the bottom of the glasses, which are supported by a glass base; all the joints are made light-tight by means of felt washers. The upper part fits loosely on the body, and can be easily and quickly removed to permit the addition of the standard solution. As sometimes the standard and the liquid under observation show before the test slight difference in tinge, a slot, S, is situated under the glass which supports the tubes, in which glasses of suitably pale amber tinge can be introduced to match the colour of the liquids.

The accuracy of the method was tested on samples of cassia oil containing known amounts of lead. The results are tabulated below.

The values for lead found by this method are thus slightly high, the average excess found being 0.00018 grm. Therefore more accurate results would have been obtained by subtracting 0.00018 grm. of lead from the amount found by experi-

ment. As the test is only approximate the errors may be said to be well within the allowed limit and the method accurate enough for technical purposes.

A comparison of the results on five commercial oils of cassia by the method described in this

Grade of cassia oil.	Amount of lead added to 10 c.c. of oil.	Amount of lead found
0%	grms.	grms.
0.005	0.0005	0.00064
0.010	0.0010	0.00128
0.015	0.0015	0.00157
0.020	0.0020	0.00216
0.025	0.0025	0.00272
0.030	0.0030	0.00328
0.035	0.0035	0.00360
0.040	0.0040	0.00416
0.045	0.0045	0.00468
0.050	0.0050	0.00520

paper and those obtained by a method suggested by Fryer and Weston,† in which the lead is extracted by warm dilute nitric acid and estimated by precipitation as lead sulphate, gives the following results:—

Oil.	% found by gravimetric method.	% found by colorimetric method.	Difference
1	0.0566	0.0536	+ 0.0030
2	0.0524	0.0520	+ 0.0004
3	0.0494	0.0512	— 0.0018
4	0.0546	0.0520	+ 0.0026
5	0.0611	0.0592	+ 0.0019

The results agree fairly well, but the gravimetric method shows, on the average, results higher than the colorimetric method, and perhaps no nearer to the actual amount present. The cassia oil after the nitric acid extraction was tested for lead according to the method described in this paper, and found to give but a very faint discoloration, showing that the extraction had been carried out satisfactorily.

It seems evident that as very similar results are obtained both by the use of the lengthy gravimetric method and the colorimetric method, the latter, which can be performed in a very short time, is to be preferred when results are needed urgently.

Using the method described above, experiments were made on the lead-dissolving power of cassia oil. 10 c.c. of the lead-free cassia oil was introduced into each of twenty-two test-tubes together with a strip of bright lead about 1 cm. broad and 6 cm. long. The tubes were then closed with clean corks, shaken at intervals, and tested at intervals.

The amount of lead absorbed increased with the time and reached a maximum of 0.074% after one month, and then seemed to remain constant. It is noteworthy that after that period the lead content exceeds the percentage found in commercial samples, which is, as a rule, from 0.04 to 0.06%.

ERRATUM.

THE SOFTENING OF PLASTIC MATERIALS. By Alan Speedy (this J., Jan. 31, 1920, 18—20 T).

Page 19 T, col. 2, softening graph, curve c should be "Balata (high grade)," not "low grade."

† Fryer and Weston, "Technical Handbook on Oils, Fats and Waxes, Vol. 2, page 20.

Birmingham Section.

Meeting held on March 29, 1917.

MR. A. E. TUCKER IN THE CHAIR.

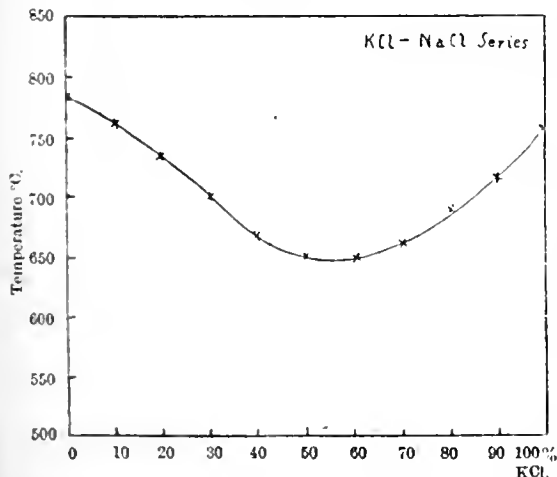
SOLIDIFICATION POINTS OF MIXTURES OF METALLIC CHLORIDES.

PART I.—MIXTURES OF SODIUM, POTASSIUM, AND CALCIUM CHLORIDES.

BY FRED. C. A. H. LANTSBERRY, M.Sc., F.I.C., AND R. A. PAGE.

MODERN methods of heating steel in the various operations of hardening and tempering frequently demand the use of molten baths. For the high temperatures used in hardening high-speed steel, barium chloride with or without the addition of potassium chloride is used, while at lower temperatures sodium and potassium nitrates and molten lead find frequent application. These latter materials possess fairly obvious disadvantages; hence some years ago the author endeavoured to find a mixture of chlorides which could be used for tempering operations at temperatures in the region of 600° C., which entails the use of a mixture which melts somewhere about 500° C. Reference to literature brought to light some very complicated formulæ for low-melting salt mixtures, a typical recipe being NaCl 1, KCl 1, CaCl₂ 2, BaCl₂ 1, and SrCl₂ 3 parts. This mixture was supposed to have a melting point of 500° F. (260° C.), a value which could only have been realised by leaving a considerable proportion of the water of crystallisation in the mixture. It seemed possible therefore that research would reveal much simpler mixtures, and an investigation was commenced which attained the desired object and led to results of sufficient theoretical interest to be placed on record. Samples of potassium, sodium, and calcium chlorides were obtained and their solidification points determined by the cooling curve method, using a platinum, 10% rhodio-platinum thermo-couple, which was frequently calibrated during the research at the temperatures of boiling water and of freezing tin (232° C.), lead (328°), zinc (419°), aluminium (657°), and silver (961°). The values obtained were: Sodium chloride, 785° C.; potassium chloride, 760° C.; calcium chloride, 745° C.

Fig. 1.



The solidification points of the binary systems were next investigated by the methods of thermal analysis well known to metallographists. Weights of salt corresponding to the desired composition were introduced into a salamander crucible and fused. After thorough admixture a thermo-couple was introduced into the melt and observations taken of the times required for the temperature to fall through successive equal intervals—in this case about 8° C. By plotting these time intervals as functions of the temperature, the so-called inverse rate curves in which arrest points are clearly marked, are obtained, typical examples being shown in Fig. 4.

NaCl—KCl series.

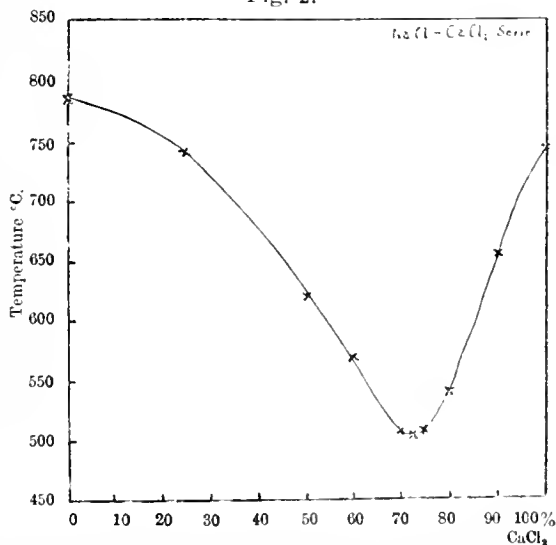
The results of the thermal analysis of this series are summarised in Table I.

TABLE I.

Test.	% components.		Freezing point, °C.
	NaCl.	KCl.	
1	100	0	785
2	90	10	760
3	80	20	735
4	70	30	709
5	60	40	697
6	50	50	652
7	40	60	652
8	30	70	662
9	20	80	690
10	10	90	715
11	0	100	760

By plotting the freezing points as functions of composition the so-called liquidus curve is obtained, and this is shown in Fig. 1.

Fig. 2.



It would appear that these two components form a continuous series of solid solutions, since none of the cooling curves showed any arrest point after solidification was complete, indicating the absence of a eutectic. The lowest melting mixture contains 55% KCl and 45% NaCl, and solidifies at 650° C.

These results confirm those obtained by Smits and de Bruyn (Proc. K. Akad. Wetensch., 1913, 557—561) and Brand (Jahrb. Min. Beih., 1911, 627—700).

CaCl₂—NaCl series.

This system was next examined and gave the results collected in Table II.

TABLE II.

Test.	% components.		Freezing point. ° C.
	NaCl.	CaCl ₂ .	
1	0	100	745
2	10	90	657
3	20	80	540
3a	25	75	508
3b	27.5	72.5	505
4	30	70	508
5	40	60	570
6	50	50	620
7	75	25	740
8	100	0	785

Fig. 3

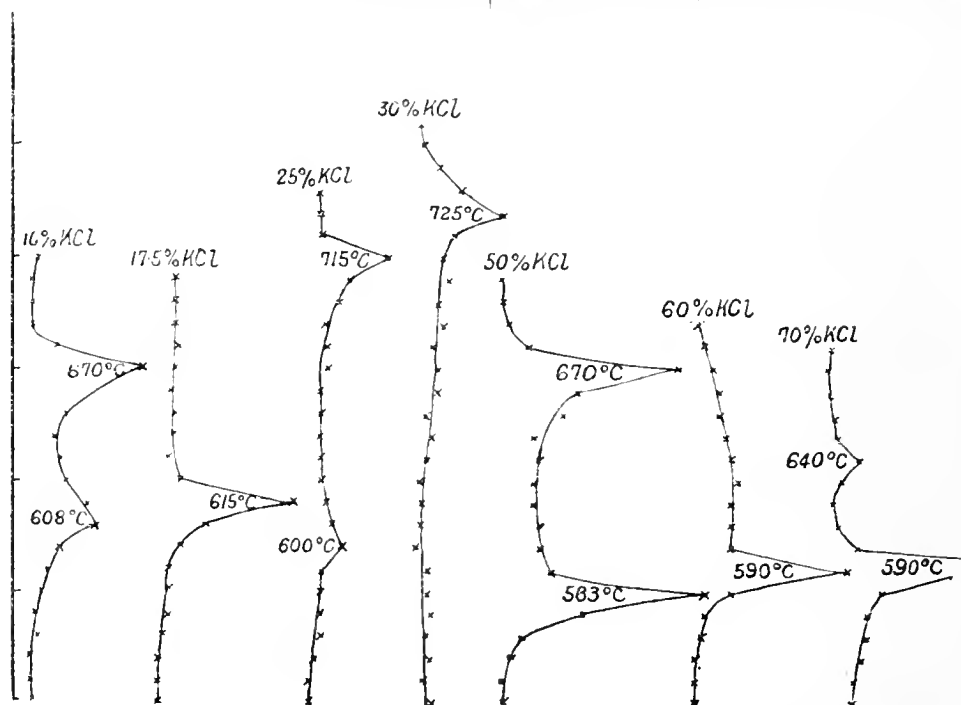
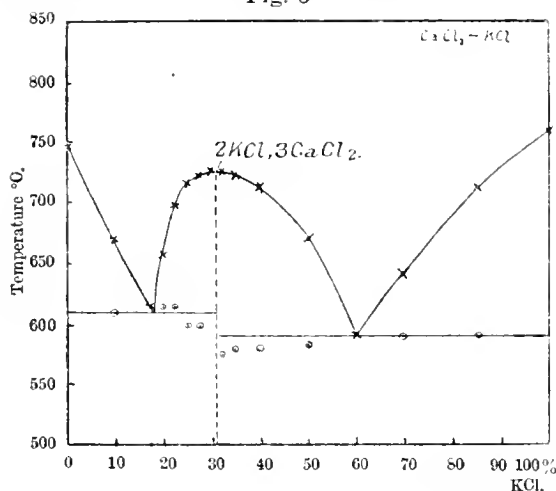


Fig. 4.

Check analyses were made on melts and gave the figure 72.3% CaCl₂ in test 3b. This proves that with proper care the ultimate compositions can be

made to agree closely with the quantities of material weighed out. The liquidus curve plotted from these figures is reproduced in Fig. 2.

Here, again, the two components apparently form a continuous series of solid solutions, for no eutectic point was observed in any melt.

The addition of sodium chloride to calcium chloride caused a considerable lowering of the freezing point; the lowest melting mixture contains 72.5% CaCl₂ and 27.5% NaCl, and freezes at 505° C.

CaCl₂-KCl series.

The third binary series was finally examined, and the results obtained are shown in Table III.

TABLE III.

Test.	% components.		Freezing point. ° C.	Eutectic point. ° C.
	CaCl ₂ .	KCl.		
1	100	0	745	—
2	90	10	670	608
2a	82.5	17.5	615	—
3	80	20	655	615
4	77.5	22.5	695	615
5	75	25	715	600
6	72.5	27.5	720	600
7	70	30	725	—
8	67.5	32.5	725	575
9	65	35	723	580
10	60	40	713	580
11	50	50	670	583
12	40	60	590	—
13	30	70	640	590
14	15	85	710	590
15	0	100	760	—

This series presented some rather unexpected and interesting results, in so far as it is quite different from the two previous systems. The new features are that the eutectic points (of which there are two) are most marked, while the liquidus reaches a

maximum at 725° C. when the mixture contains about 70% CaCl₂. This maximum corresponds to the formation of a double compound of formula

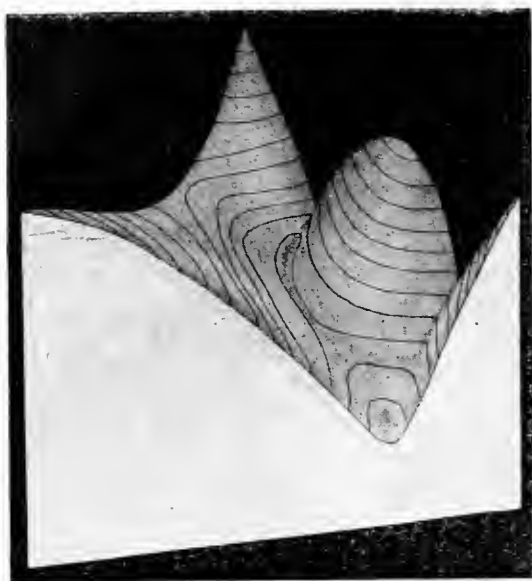


Fig. 6.

able amount of light on this point, which will be dealt with in its proper place.

Ternary series KCl—NaCl—CaCl₂.

The usual method of graphic representation of the results obtained during the investigation of a ternary series is to plot compositions in the plane of an equilateral triangle and to plot the property (in this case temperatures of thermal disturbances) at right angles to this plane, according to the method described in detail in text-books of metallography.

Mixtures of the three salts were made up according to the compositions indicated by the small crosses in Fig. 5, in which the numerals represent the temperatures at which freezing commenced. Cooling curves were taken, the results of which are summarised in Table 4.

The graphical representation of the results takes the form of a solid model, a perspective view of which is shown in Fig. 6. But the more general method of representation of the results of the investigation of a ternary series is to draw the isothermals on the liquidus surface and project them on to the base of the model. This is shown in Fig. 7.

In this system two ternary eutectics are formed, one situated at (a) in Fig. 7, containing 70% CaCl₂, 25% NaCl, and 5% KCl, and the other consisting of

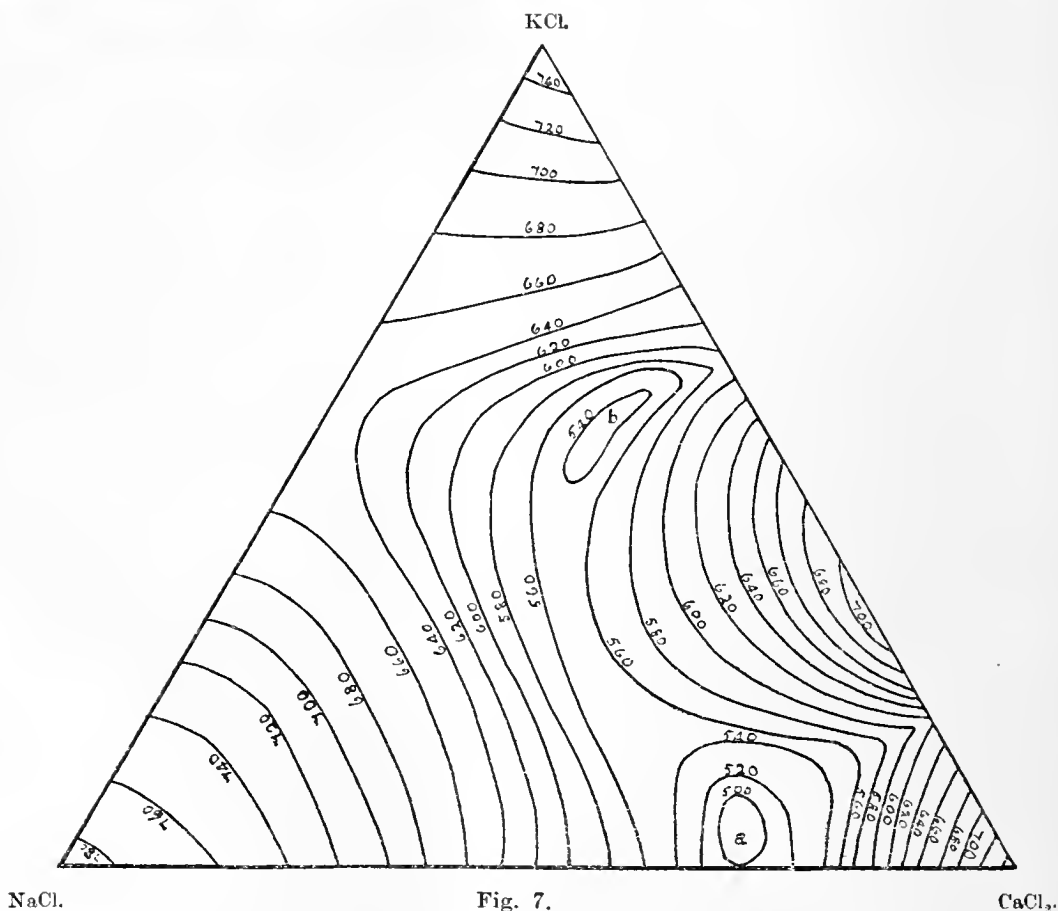


Fig. 7.

28.8% NaCl and solidified at 495° C., and this corresponds very closely with the minimum in the liquidus described in the present paper. Further, the work on the ternary series throws a consider-

30% CaCl₂, 20% NaCl, and 50% KCl situated at (b). Mixture (b) begins to freeze at 530° C., while mixture (a), the lowest freezing mixture of the whole series, begins to freeze at 495° C.

The results of the investigation of the ternary series are somewhat disappointing, because it was hoped that the addition of potassium chloride to the CaCl_2 — NaCl eutectic would effect a considerable lowering of the freezing point, which would have increased the scope of utility of the materials in tempering operations. Nevertheless, it is of considerable interest from the technical point of view to know that the low melting mixtures are prepared from the cheapest materials, because no real advantage is gained by adding 5% of the comparatively expensive potassium chloride in order to obtain a lowering in the freezing point of only 5°C .

TABLE IV.

Test.	% components.			Freezing point, $^\circ \text{C}$.	Eutectic point, $^\circ \text{C}$.
	CaCl_2 .	KCl .	NaCl .		
1	60	20	20	590	530
2	40	40	20	590	—
3	40	20	40	578	550
4	20	60	20	608	540
5	20	40	40	608	530
6	20	20	60	700	540
7	80	10	10	580	510
8	10	80	10	690	540
9	10	10	80	748	550
10	65	10	25	—	515
11	57.5	20	22.5	600	540
12	50	30	20	608	540
13	30	60	10	560	—
14	70	20	10	617	550
15	60	30	10	657	—
16	50	40	10	645	530
17	40	50	10	608	540
18	70	10	20	512	—
19	30	60	20	530	—
20	10	70	20	663	525
21	60	10	30	540	—
22	50	20	30	548	—
23	40	30	30	560	530
24	30	40	30	558	520
25	20	50	30	585	530
26	50	10	40	605	540
27	10	60	30	640	540
28	30	30	40	592	540
29	10	50	40	630	540
30	40	10	50	655	530
31	30	20	50	650	530
32	20	30	50	655	530
33	10	40	50	655	530
34	30	10	60	690	530
35	10	30	60	690	540
36	20	10	70	725	530
37	10	20	70	725	540
38	75	15	10	590	550
39	35	55	10	558	—
40	77.5	12.5	10	565	—
41	32.5	57.5	10	542	—

Allusion has already been made to the disagreement between the results of the present investigation and those of Menge with regard to the existence of the compound $4\text{NaCl}.\text{CaCl}_2$. The formation of such a compound would result in an inflexion in the liquidus not only in the CaCl_2 — NaCl series, but also in the series containing constant but relatively small percentages of potassium chloride, i.e., those series represented by vertical planes cutting the ternary model parallel to the CaCl_2 — NaCl side of the triangle. Such sections show no signs of inflexions, so that for the present the authors are compelled to reject the idea of the formation of any compounds between sodium and calcium chlorides because the whole of the data obtained from the ternary series strongly favour the conclusion that these two salts form a continuous series of solid solutions.

It is hoped to extend these observations to other ternary salt mixtures at some not very distant date.

In conclusion, the authors would take this opportunity of thanking the Birmingham Small Arms Co., Ltd., in whose laboratories this work has been done, for granting the necessary facilities to carry out these investigations.

Meeting held at Birmingham University, on Thursday, November 13, 1919.

MR. L. P. WILSON IN THE CHAIR.

THE MANUFACTURE OF ARTIFICIAL ABRASIVES IN THE ELECTRIC FURNACE.

BY C. J. BROCKBANK.

Artificial abrasives require for their production large quantities of electrical energy, such as can only be furnished cheaply by the enormous water power developments of Canada, the United States, and similar countries abroad. Due to this natural advantage there have developed in Canada several large plants devoted entirely to the manufacture of artificial grinding materials called by various trade names, but consisting of two groups, one silicon carbide, and the other aluminous abrasive, or artificial corundum. The fact that upwards of 140 million pounds of these materials was produced in Canada during the year 1918 shows that the industry has become one of enormous importance. The refractory by-products are also of great importance, and have contributed very materially to increased efficiency in the operation of many types of furnaces. The value of abrasive products exported from the United States alone in the year 1918 was upwards of one million pounds.

Early History.

There are many references in ancient history to the use of abrasives. Emery was mined at the Island of Emiri by the ancient Egyptians during the time of the Pharaohs, and used by them for grinding and polishing those magnificent monuments in existence to-day. The ancient Indian races used a mixture of crushed emery powder and beeswax for polishing stone, and emery and natural corundum constituted the only abrasives of an efficient character in use from those times until silicon carbide (carborundum), the first artificial abrasive, was discovered by Dr. Acheson, in the year 1890.

Unfortunately, silicon carbide cannot be used for all classes of grinding. It is most suitable, and most highly efficient, in the grinding of brittle materials, such as cast-iron, marble, mother-of-pearl and materials of that kind, and the finishing and sueding of leather and so on. It was the attempt of the Carborundum Company to make it a universal grinding material that so nearly led to financial disaster in their early days. The physical properties of silicon carbide render it unsuitable for producing the fine, smooth finish required in steel grinding. It is intensely hard (the next hardest material to the diamond), and very brittle. During grinding the edges of the individual grains are constantly fracturing, exposing fresh cutting edges, which score deeply into a tough material such as steel. Subsequent to the discovery and commercial introduction of silicon carbide, therefore, emery and natural corundum continued to hold their own in a large section of the abrasive field. Unfortunately, the deposits of natural corundum were rapidly exhausted both in Canada and the Southern United States, and the properties of Naxos emery were so uneven that considerable difficulty was experienced in making wheels from it that would meet the diverse conditions and exacting specifications of the steel industry. The discovery that such aluminous ores as bauxite and emery could be electrically smelted with coke, and commercially pure, crystallised artificial corundum obtained therefrom, completed the requirements of the abrasive field and rapidly forced natural emery into the background except for a few restricted operations, such as polishing, particularly the

polishing of glass, for which none of the artificial abrasives is suitable to-day.

The Manufacture of Silicon Carbide.

Silicon carbide is formed by the reaction at high temperatures of silica and carbon. In practice it is found necessary to add varying amounts of sawdust and sodium chloride. Sawdust is used to maintain porosity in the mixture, so that the large volumes of carbon monoxide may escape without causing blowholes at local spots in the furnace; the salt assists in the elimination of the impurities present in the coke and sand as chlorides. A typical furnace mixture when all new materials are used is: Petroleum coke 1500 lb., white sand 2520 lb., sawdust 300 lb. (18 cub. ft.), salt 40 lb. A considerable proportion of the mixture used in a



First stage in formation of silicon carbide. Second stage in formation of silicon carbide. (Crystals forming.)



Surface of silicon carbide crystal. $\times 20$.

furnace is not converted into silicon carbide, acting merely as a heat-insulating blanket, and this material will ordinarily be returned to the mixing department and added in definite proportions to the new materials. Inasmuch as this old mix has lost part of its carbon by oxidation, the proportions are somewhat altered, and a typical operating mixture for continuous use becomes as follows: Petroleum coke 1500 lb., sand 2455 lb., sawdust 300 lb., old mixture, 400 lb., salt, 40 lb. Petroleum coke is the carbon residue left in the stills when distilling crude oils and is the form of carbon that can be used most satisfactorily for producing the highest grade of silicon carbide. This does not appear to be due altogether to its purity, since good silicon carbide cannot be made from lampblack, but is in

some way due to the readiness with which it is converted into graphite by heat. Charcoal is readily changed to graphite, and similarly will make excellent silicon carbide.

Silicon carbide furnaces are built in sizes varying from 1000 h.-p. capacity to 3000 h.-p. capacity. A furnace of less than 1000 h.-p. size cannot be operated economically. A 3000 h.-p. furnace will produce approximately 7% more silicon carbide per unit of power input than a 1000 h.-p. furnace, although the advantage is somewhat offset by the fact that the quality from the largest size is not so good. A 1000 h.-p. furnace is 20 ft. long between the heads carrying the electrodes and 5 or 6 ft. wide inside. The heads carrying the electrodes are 40 in. thick, and two electrodes are used for each terminal, made from graphite 10 in. diam. and 48 in. long. The method of loading the furnace is as follows. The furnace is charged with mixture slightly above the level of the electrodes, and a trench formed in this mixture 36 in. wide and 10 in. deep, which is filled with about 1200 lb. of graphite powder. This forms the resistor core and conducts



Surface of silicon carbide crystal. $\times 20$ diam.



Devitrified layer of quartz in silicon carbide crystal. $\times 20$.

the current from one terminal to the other. The charge is then piled on this core and the furnace is ready to operate.

Power for the plant of the Exolon Co., at Thorold, Ontario, is furnished from the hydro-electric station of the Toronto Power Co. at Niagara Falls. It is three-phase twenty-five-cycle current, and is transmitted to a sub-station adjacent to the abrasive plant at a potential of 60,000 volts. In this sub-station it is transformed down to 12,000 volts, at which voltage it is delivered to the plant.

The furnace transformers used are of the oil-insulated water-cooled type, single-phase, and by means of taps taken off the high-tension side it is

possible to obtain any voltage between 160 and 85. When a furnace is first cut in the highest voltage is used. The furnace should reach full load in less than three hours at this voltage, after which the voltage must be periodically reduced, or the furnace would run overload, due to the negative temperature coefficient of the graphite resistor in the furnace; with a core of the size given, stable conditions should be attained between 85 and 100 volts. Very soon the carbon monoxide evolved will ignite on the outside of the furnace. It is usual to introduce a definite amount of electrical energy and to take off the furnace when that is done. For the size described 22,500 k.w. hrs. is used each furnace run, and the duration of the run will vary from 32 to 34 hours. There is a considerable shrinkage of the mixture during the run, the level gradually falling below the side walls of the furnace. The side walls must not be taken down till at least 24 hours after a furnace has been disconnected; otherwise the product is liable to be injured by oxidation. Subsidiary reactions involving to some degree recrystallisation of the product take place after the furnace is disconnected.

A silicon carbide furnace, efficiently operated, should produce 0.16 lb. of SiC per h.-p. hr., or should require approximately 5 k.w. hrs. for each pound of crystalline silicon carbide. In addition to the crystalline material, one ton of firesand or impure amorphous silicon carbide will be produced per furnace run. This is a very valuable refractory for the lining of brass-melting furnaces, outlasting many firebrick linings, and the demand greatly exceeds the supply, since it can only be produced indirectly as a by-product.

The costs of production have increased greatly since the year 1915, due to the increase in the prices of all raw materials and labour. With power costing \$14 per horse-power year these costs are as follows for the years 1915 and 1918:—

Manufacturing Costs per ton of 2000 lb.

	1915.	1918.
Raw materials	\$19.00	\$40.61
Power	21.82	23.94
Labour	10.51	34.96
Repairs and supplies ...	2.70	5.49
Total manufacturing cost		
(exclusive of overhead)	\$54.06	\$105.00

Finishing of Silicon Carbide.

The crystalline silicon carbide after removal from the furnace is crushed in a pan mill and treated successively with sulphuric acid and caustic soda. This removes the surface impurities from the grain, consisting of graphite, silicon, silicides of iron (mainly SiFe₂), and aluminium silicide. It is then washed, dried in a rotary dryer, and passed over screens which separate the grains into various sizes from $\frac{1}{2}$ in. mesh to $\frac{1}{220}$ in.; finer sizes than this are graded by hydrostatic methods. This material forms the pure product used by the wheel manufacturer for making abrasive wheels. It is mixed with a porcelain bond, and fired in pottery kilns. The process of manufacturing wheels is beyond the scope of this paper.

Aluminous Abrasives or Artificial Corundums.

These are manufactured by smelting a mixture of bauxite and coke in an arc furnace; the impurities, mainly silica and iron oxide, are reduced and settle to the bottom of the furnace, and the fused alumina is recovered in the form of a large pig or ingot weighing 4 to 5 tons. The bauxite used will usually have approximately the composition: SiO₂ 8.40, Al₂O₃ 50.68, Fe₂O₃ 13.22, TiO₂ 2.65,

loss on ignition 24.93. A typical furnace mixture is calcined bauxite 1750, coke 100, iron borings 350 lb. The furnace used is mounted on a water-cooled track and consists merely of a four-sided iron frame with an inner lining of fire-brick. The dimensions will be approximately 5 ft. x 7 ft., and the height of the side walls 5 ft. The hearth is made of pitch and carbon. The furnace is started by placing a layer of mixture about 1 ft. deep in the furnace and laying a train of graphite powder from one electrode to the other on the top of this. As soon as a small path of molten ore is formed this carries the current, and more mixture is charged into the furnace as required.

These furnaces operate at about 100 volts and require 550 k.w. An ingot weighing 4 or 5 tons should be produced with a power input of 18,000 k.w. hrs., but this will vary widely according to the quality of ore being used. When the furnace is full it is removed bodily and a new one inserted in its place.

Reduction must not be carried too far; otherwise the abrasive will be too pure and brittle. The presence of 1–2% of iron oxide, silica, and titanium dioxide increases the toughness of the product. A typical analysis of high-grade abrasive would be Al₂O₃ 97.40, SiO₂ 0.90, Fe₂O₃ 0.32, TiO₂ 1.38%. The metallic residue recovered will contain 10–15% of silicon. Sufficient iron borings are added to the mixture to keep the silicon content of the alloy below 15%; otherwise the material would be too weakly magnetic to be separated from the crushed corundum.

Manufacturing Costs.

During the year 1918 the costs of manufacture were as follows per ton of 2000 lb.: Raw material, \$45.65; electrodes, \$10.82; power, \$7.79; labour, \$19.73; and repairs and supplies, \$6.57; total, \$90.56. It will be noticed that power is a comparatively small proportion of the total manufacturing cost of artificial corundum, and it would therefore seem to be quite practicable that aluminous abrasives should be manufactured in England at a cost that would compete with the foreign product. Important developments in this field are to be expected therefore in the near future.

DISCUSSION.

THE CHAIRMAN asked if the presence of graphite were detrimental to the product as an abrasive; and whether silicon was available commercially for acid-resisting castings, also if its properties were such that it could be worked. He took it from the way in which it was treated that carborundum was resistant to chemical action—both alkaline and acid.

MR. A. E. TUCKER said that if a carborundum wheel were run much below the speed specified it would soon be destroyed, and it was well known that a disc of copper or even of paper had great abrasive action if run at sufficiently high speed. He would like more information about the crystals because they did not appear always to present cutting angles to the work operated on.

MR. F. C. A. H. LANTSBERY asked for further information with regard to the melting of alumina, and also as to the cost involved. What amount of impurities was permissible? Melted pure alumina would probably not be quite so hard as alumina containing a certain amount of impurities. Generally speaking, the addition of impurities, in small quantities, to pure material had first the effect of increasing hardness, more particularly if the impurities went into solution.

Mr. R. A. PAGE enquired whether the electrodes in the furnace melting alumina were vertical or horizontal, as in the carborundum furnace; and also, if three-phase current were used, were two or three electrodes employed?

Mr. J. C. MANN enquired if it were essential that the carbon used should be free from ash or volatile matter; and what was the author's experience of pitch coke?

Dr. H. W. BROWNSDON, referring to the author's suggestion to use "fire sand" mixed with sodium silicate or clay for lining brass furnaces, asked if that was impure carborundum, and what was the price? If the life of such material was ten times greater than that of the ordinary firebrick it could be used fairly freely.

Mr. A. E. TUCKER stated that about three years ago he had obtained for a steel furnace a brick which he had shaped down so as to allow round it about an inch thickness of carborundum. When the other bricks had become worn out the one so treated had remained in first-rate condition.

Mr. BROCKBANK, in reply, said he believed the presence of graphite was decidedly detrimental, particularly for some classes of work. Carborundum, not free from graphite, did not seem to behave well; and to get the best results graphite should be removed from such articles as refractory bricks. The pure silicon casting had not become what he would regard as a commercial success. The cost of such castings was about 5s. per lb.; the few that were provided lasted well. Carborundum was insoluble in acids and alkalis. Filter plates had been made from carborundum which could be used to filter the strongest acids. These were made with 85 lb. of "grain" and 15 lb. of petroleum coke, held together with dextrin or glue and exposed to the vapour of silicon. It was necessary that the crystals should be solid grains of kernel shape; the rougher the better so far as surface texture was concerned. He did not think the precise conditions of successful grinding were understood. The carborundum was harder than the alumina abrasive, but the difference in the properties did not appear to be due to variations in hardness. Alumina abrasives would operate for 20–28 hours, but much depended on the quality of the ore. The difference in properties appeared to be due to the fact that the aluminous products became polished, but particles of the carborundum were continually being broken, exposing fresh cutting edges. The power absorbed varied considerably; there was much waste energy in an alumina furnace, because at times it must simmer. There was the danger of getting a spongy or overburnt material if the energy were forced into the furnace. He found that the toughest alumina abrasive would contain 2% of iron oxide and 2% of titanium dioxide; whether one or the other was responsible for the toughness he could not say. He had never manufactured a material from absolutely pure material; he thought both oxides increased the toughness of melted alumina. The electrodes were vertical; the furnaces were not lined, the mixture forming the lining, and there was a layer about 2 inches thick left when the operation was finished. The outside of the furnace was not water-cooled. Petroleum coke was peculiarly suited for producing carborundum, but pitch coke, if it did not contain too much volatile matter, was quite suitable. "Fire sand" was amorphous carborundum; its price was about £12 per ton.

THE CHAIRMAN, referring to a question as to what was an abrasive, said that a wet cellulose thread was capable of cutting glass under certain conditions when running at a high speed.

Newcastle Section.

Meeting held at Chemical Industry Club, Newcastle, on January 28, 1920.

PROF. P. P. BEDSON IN THE CHAIR.

NOTES ON TUNGSTEN ORE DEPOSITS IN BURMA.

BY J. COGGIN BROWN
(*Geological Survey of India*).

Owing to the constant association in many parts of the world of the natural tungstates of iron and manganese with cassiterite, the common ore of tin, these minerals must have been known to man from a very remote antiquity. It was not until 1781 that Scheele recognised a new acid in the mineral which was later called after him. In 1783 the brothers D'Eluhardt, pupils of Bergman, proved that wolfram also contained tungsten and isolated the new element. The commercial life of tungsten commenced in 1847, when Oxland patented a method for the preparation of sodium tungstate and metallic tungsten. In 1857 Oxland took out another patent for the preparation of alloys of tungsten with iron, steel, and nickel. This was followed in 1859 by Mushet's patented steel, which contained 6–8% of tungsten, 2% of manganese, and a high percentage of carbon. The credit for the more modern development of high-speed steels by heat treatment is said to be due to Taylor and White, who exhibited their products at the Paris exhibition in 1900. About the year 1903 a great demand arose for wolfram, which before that time was regarded as an impurity in tin concentrates; the price began to rise and production to increase, until during the years of the war unprecedented prices were given in uncontrolled countries, and the very ends of the earth were ransacked for supplies.

Tungsten steels are prepared with suitable admixtures of other metals, such as chromium and vanadium. The material used for making the best tools contains 18–20% of tungsten. Such steels are not only exceedingly hard, but they maintain their hardness at high temperatures. As these qualities may be impaired by the presence of small quantities of tin, sulphur, phosphorus, arsenic, bismuth, or copper, all of which occur in nature with tungsten, the necessity for dressing the ores as clean as possible is emphatic.

The introduction of high-speed steel is said to mark one of the greatest advances ever made in the metallurgy of iron and to have completely revolutionised the machine shop practice of the civilised world. An American writer has remarked that to deprive a nation of tungsten is to cripple its military power and its industrial power in times of peace.

The use of tungsten filaments in incandescence electric lamps has also transformed this particular industry, though without conferring any benefit on the producer of tungsten ores, owing to the small quantities used in making millions of lamps. Tungsten steel is also used for making permanent magnets, for the valves of some makes of internal-combustion engines, as contacts for sparking plugs, tremblers, and voltage regulators, for targets and cathodes of Röntgen ray tubes, and in gramophone needles. The metal is a constituent of "stellite," "partinium," and various other alloys. Some of its salts have limited industrial uses.

There are but few natural compounds of tungsten; they are all definitely oxidic in character, and

form a strong contrast to the behaviour of the sulphidic ores. Most of the latter are connected with intrusive rocks of the basic type, but the tungsten ores are usually, if not invariably, associated with strongly acid rocks. The only ores of commercial importance are the tungstates of iron and manganese and of calcium. The iron and manganese tungstates form a striking example of an isomorphous series. The theoretical end products are ferberite (FeWO_4) and hubnerite (MnWO_4), but they have never been found pure. In the absence of chemical analyses it is better to use the term "wolfram" to cover the whole series.

For centuries there has been a small indigenous tin-mining industry in Lower Burma. Thus Ralph Fitch, an English adventurer who travelled there in 1599, has left it on record that he passed "the Iland of Tavi, from whence cometh great store of tinne; which serueth all India." One of the earliest references to the occurrence of wolfram is in a work published by Dr. Mason, an American missionary, in 1846, who states that the tungstate of iron, or wolfram sand, much resembles tin and is found in most neighbourhoods where that ore occurs. He relates how a certain Government officer erected furnaces and tried to smelt wolfram sand under the erroneous impression that he was dealing with cassiterite; but though he tried hard and raised the heat to the highest point he was capable of, the ore still remained refractory and would not turn into tin.

Wolfram was re-discovered in Tavoy by the Geological Survey of India, in 1909, and was first exported in 1910, when about 400 tons was produced. At that time the world's production was about 6000 tons of 60% WO_3 concentrates, and the chief producing countries were the United States, Portugal, and Queensland, with smaller amounts from the Argentine, Bolivia, and New South Wales. By the next year, 1911, wolfram mining was thoroughly established in Tavoy, and an output of over 1300 tons made Burma the leading tungsten-producing country in the world, a position which she maintained easily until 1916, when the boom in the Americas caused both the production of the United States and of Bolivia to exceed hers. In 1914, out of a world's production of some 8000 tons Burma alone produced about 2300 tons. By this time other countries had entered the list—Japan, China, Siam, the Malay States, and Billiton in Asia, the Northern Territory of Australia, Tasmania, New Zealand, and Peru, while the European countries, including Germany and Austria, contributed their small portions.

Although Germany has no important domestic supplies and possessed none in any of her foreign territories, she has been credited with the control, in 1913, of two-thirds of the world's production. Thus she treated nearly 6000 tons of 60% concentrates. The British steel makers generally obtained their supplies of finished tungsten products from the German manufacturers, and it is no secret that August, 1914, found this country with but a few months' stock in hand. It is not within my province to describe the admirable way in which British metallurgists and chemists got the nation out of its difficulties, but I may indicate the manner in which Burma responded to the imperative demand for the raw material. In 1915 all the Imperial supplies were earmarked for the Home Government at a fixed price of 55s. per unit of WO_3 . This was raised in 1917 to 60s. per unit. These prices may be compared with 9s. and 51s., the minimum and maximum prices for the years 1897 to 1914. In Burma itself the Government assisted the miner in every possible way, by importing labour, making roads, and creating a special administrative and technical advisory staff. The mining firms did their best,

and great advances were made in mining and milling methods. As a result, the output increased from 1688 tons in 1913 to 4480 tons in 1917, and from the beginning of the war-year to the end of 1918 no less than 17,636 tons, of a total value of £2,322,000, was exported.

The American output, under the stimulation of an uncontrolled price, which reached a maximum of \$934 a unit, or six times the price offered in the Empire, in 1916 rose to over 5900 metric tons of 60% concentrates, and there was a corresponding increase in all other producing countries, so that the world's production of about 8000 tons in 1914, rose to 12,000 in 1915, 23,000 in 1916, 28,000 in 1917, and 35,000 in 1918. In 1917, China, which had been an insignificant producer before that time, brought 1500 tons into the market, rising in 1918 to over 10,000 tons, or more than the whole world's production for any one year before 1915.

In 1918 92% of the world's tungsten ores came from the mountains bordering the shores of the Pacific Ocean, 5% from the Iberian Peninsula, and 3% from England, Germany, and other European countries.

Tungsten ores are almost invariably associated with the more siliceous granite rocks, and the most important metallogenic province is the Sino-Malayan one, which includes Burma and the Shan States, the Malay States, the Dutch East Indies, Siam, Tongkin, and South China. The deposits of Japan and Korea may represent an extension of this province.

Wolfram and cassiterite have been found at intervals over a distance of 750 miles in Burma, always in direct association with the great granite ranges which stretch from an unknown termination in the Shan States to the extreme south of the province. The isolated occurrences of the Kyaukse, Yamethin, Karreni, Thaton, Amherst, Tavoy, and Mergui districts are links in a chain which forms the northern extension of the Indo-Malayan mountain system, and which continues south through Lower Siam and the Malay States to Banca and Billiton, forming the core of the Peninsula, and carrying its distinctive metallogenic characteristics throughout its entire length.

The geological history and structure of Tavoy, the most important mining field of Lower Burma, are simple. A series of very old sedimentary rocks in which clay slates predominate, was intruded at an unknown period by a white, very siliceous granite, which is now exposed where denudation has removed its earlier covering. The ores of tungsten and tin are always found in or near the outer margins of the granite intrusions.

Both wolfram and cassiterite occur in Tavoy as segregation deposits in the granite itself, in pegmatite veins, and in quartz veins which penetrate the granite or the sedimentary rocks. Other minerals that may occur in the veins are mica, fluorite, molybdenite, pyrite, chalcopyrite, pyrrhotite, arsenopyrite, galena, zinc blende, native bismuth, bismuthinite, and, in one case, topaz. Both minerals occur in greisens, the bands of altered granite adjoining the veins themselves, and in residual, detrital and talus deposits of the slopes. Cassiterite is transported and found in river gravels and other alluvial deposits. Wolfram is not.

It is stated in text-books that the Tavoyan field is characterised by the universal presence of tourmaline, and that columbite is a common mineral in it. Both these statements are incorrect.

Every granite intrusion in Tavoy has its associated ore-bearing veins in some part or other, though large expanses of granite are found also which do not contain veins. This is either due to the fact that veins were not formed there originally or have been cut away by denudation at a later period.

Every producing mine is situated on or near the granite contact. The veins themselves vary a great deal, yet they possess certain generic features which may be classified as follows:—(a) They are formed by the filling of fissures; (b) they often occur in parallel groups; (c) they often form a series of overlapping lenses; (d) the lenses themselves are irregular, they thin out and thicken again, or split up and reunite; (e) they generally possess clean and well-defined walls; (f) alteration of the granite walls to greisen is universal; (g) the vein quartz is dense, milky white and very compact; (h) mica is invariably present and sulphides very often; (i) secondary movements of the fissures are common.

The veins are of all sizes and lengths, from small stringers to well-developed individuals which can be traced for miles. The undergrowth of the forests and the thick soil cap make it difficult to follow the veins, but several are known over a length of 1200 feet. Both wolfram and cassiterite have a tendency to deposit in little quartz veinlets half an inch or more in thickness, and when these are in close association, and especially if the ground is soft, and can be broken down by water under pressure, valuable sources of the ores are formed. Most of the veins strike in the same direction as their parent granite masses, that is north and south to north-west and south-east. Dips are usually very high. The mica of the veins occurs in small flakes, and muscovite is the commonest variety. Green chlorite is also found, and a lithium-bearing chlorite has been reported. Wolfram and cassiterite have a very erratic distribution in the veins. In extreme cases great slugs of wolfram up to a ton in weight have been exposed, but any profit which comes to the miner therefrom is usually lost in driving through barren ground to find the next one. This irregular distribution, coupled with a pernicious local system of mining, makes the exact valuation of the veins a very difficult matter. Contents varying from 1 to 3% are recorded, and in most cases are probably underestimated. No mining has yet been carried on in Tavoy below about 400 feet from the granite contact, but a study of all the available evidence leads to the conclusion that there is not much likelihood of wolfram giving out as greater depths are attained in most of the larger mines. In my opinion the question of the permanence of wolfram in depth is of secondary importance to that of devising means of mining narrow veins in hard granite, and of recovering the mineral content by modern methods with the poor labour supply available.

The order of deposition of the metallic minerals is a subject which has evoked much discussion, as it follows that if cassiterite was deposited at a higher temperature than wolfram there is a possibility of wolfram giving place to tin ore at depth, and of the field becoming a large tin producer, like the Federated Malay States further south. Theoretical speculations on geochemical processes, which are impossible of reproduction in the laboratory by any means at our disposal to-day, may be put aside. Field evidence is convincing that molybdenite was the first ore mineral to form, that it was followed by wolfram, and it, in its turn, by cassiterite, for hundreds of specimens occur in which cassiterite was clearly secondary to wolfram in cases of mixed growths.

Although it is possible to imagine theoretical conditions under which tungsten ores in the upper part of a vein might be dissolved to form solutions, which percolating downwards should have their tungsten contents precipitated in a lower horizon, the fact remains that in Burma there are no zones of any economic importance in wolfram mines which have been enriched by solution and reprecipitation in any way comparable to those, say, of copper and silver, in other parts of the

world. Tungsten solutions are very easily precipitated by ferric salts, but this precipitate is colloidal and difficult to filter. It is doubtful if it would lodge in a definite deposit. Ferric tungstate and the hydrated oxide, natural tungstic acid, are in any case compounds which it is impossible to recover by ordinary concentrating devices.

The relative proportions of cassiterite and wolfram in the concentrates vary a great deal from mine to mine. The tin content in vein material is higher in granite veins than in those of the sedimentary rocks. Concentrates which are practically tin free only occur in a few cases, and the veins from which they are derived are all in argillites or clay slates. Concentrates from surface deposits are always richer in tin ore than those from veins. The best Tavoy concentrates contain over 70% of WO_3 , and all the separated material contains between 65% and 70%. A calculation based on the probable composition of the concentrates produced from 50 mines in 1918 showed that as produced the total bulk averaged 55% of WO_3 , and 15% of metallic tin. These mines produced over 95% of the district's total in that year.

Mining methods of the early days were of a very primitive character, and the industry still suffers because they have never been entirely eradicated. The first discoveries were made by Burmans and Karens, who collected the ores from the outcrops. They were speedily followed by Chinese immigrants, whose "crude methods exasperate equally with their bland unwillingness to improve on them." A gang of Chinese works under a contractor, who is paid according to the amount of clean, high-grade concentrate he brings in, and usually a footage rate for underground operations as well. In many cases the Chinese miner was allowed to work as he pleased, with the result that vein workings became rabbit warrens and valuable surface deposits were buried under débris. Ore concentration methods were of the crudest kind, the wolfram-bearing quartz being crushed by hammers on a flat stone and the powdered stuff washed up in a cradle or pan. The whole sequence of operations was extremely wasteful. Low-grade ore was left underground or thrown away on scattered dumps, while the losses in crushing and dressing were very high. Of late years there has been a change for the better. Tributing is still widely practised, but it is controlled to a degree hitherto unknown; deep level exploratory work is now carried on by the aid of machine drills driven by compressed air; concentrating mills have been designed and erected; large-scale mine plans and sections are required by the mining laws.

Wolfram is a difficult mineral to recover even by the most modern devices. It possesses a perfect cleavage and breaks down into thin, mica-like plates when it is powdered. By the action of stamps these naturally become slimed, and this leads to heavy loss. It appears to me to be better to adopt the practice in which a minimum amount of fine material is produced, and I am of the opinion that a concentration system which makes full use of hand picking, coarse crushing in rolls, sizing, and jigging, with the removal of wolfram and cassiterite at every stage where they can be obtained as coarse as possible, followed by full use of present practice as regards the inevitable fine-ground material, is preferable to the earlier one in which the unsorted ores were taken direct from the mine to stamps and reduced to the consistency of tooth powder before any attempt was made to separate their valuable contents.

In a climate like the Tavoyan one, which has a monsoon rainfall of 200 inches or more, the quartz veins undergo rapid denudation and shed their contents into the surface soil. Valuable deposits are thus formed containing wolfram and tin ore in

the surface covers of the hill sides, and of tin-stone alone in the true water-sorted alluvium of the valleys. The greater part of the output is still derived from these. In the first stages of a mine's history the beds and banks of the stream are cleaned up. Then water is led by ditches or flumes on to the profitable ground which lies at higher levels. Most of the larger mines are supplied with water brought by elaborate systems, often from considerable distances. If a gravity supply is not available, water is lifted by pumping, provided the area and value of the bearing-ground warrant the cost. Occasionally rich residual deposits are excavated by hand and treated in a pan similar to those used in washing gem gravels at the Burma ruby mines or the Kimberley diamond fields. It is claimed that the pan saves a larger proportion of fines and does the work with about a third of the water required in hand working with an ordinary sluice box. When the rich ground is buried under overburden it is often broken down and washed in one operation; for this purpose monitors or hydraulic giants are used, the ores being recovered in sluice boxes in the usual way. Hydraulic elevators become necessary when the tailings can no longer be disposed of by gravitation, or gravel pumps are used instead of the elevator by reason of their greater efficiency. In the most advanced installations of this kind, when the water can be obtained from a source very much higher than is necessary for hydraulicizing, it may be used to drive a nozzle pump by means of a Pelton wheel, or the whole system may be electrified by its means, as is done at the Kanbank mine, where the same water is used repeatedly for hydraulicizing and the pumps are mounted on a barge which can be floated nearer to the working surface as it recedes.

Previous to 1916 the mixed concentrates were shipped as they came from the mines. In that year a magnetic separating plant was installed by the High-Speed Steel Alloys Mining Co., Ltd., so that a portion of the output is now separated before export. The machines used are of the Ulrich type.

Very erroneous ideas are prevalent in literature regarding the behaviour of the natural tungstates when exposed to the action of air and water. Thus, W. H. Emmons classifies tin and tungsten together with gold (in part), bismuth, chromium, and molybdenum. Again, R. H. Rastall writes:—"The outstanding feature of the tungsten minerals is their great stability and resistance to any kind of chemical or mechanical alteration. . . . They are specially liable to occur in placers and other forms of transported deposits. . . . In this respect both wolfram and scheelite behave like stream tin, gold, and platinum." If the word "placer" means stream-borne and water-sorted alluvium, then there are no wolfram-bearing placers in Burma. Wolfram may occur at the foot of slopes, where the detrital deposits of the hill sides merge into the true placers of the valley bottoms, but long before the clays, sands, and gravel deposits have been sorted out and transported by running water the wolfram has disappeared, though it comes from the same veins as the tin ore which remains. The rapidity with which wolfram decomposes in the surface soil is very remarkable, and is accounted for by its cleavage, resulting in its disintegration on movement with the production of a comminuted form eminently suited for chemical decomposition. Wolfram is readily leached from the outcrops of veins. The ready oxidation of its sulphide associates yields acid solutions which attack the mineral until nothing is left but cindery oxides of iron and manganese or a bloom of tungstic acid in the spaces once occupied by the crystals. R. W. Gannett has carried out laboratory experiments to determine the effect of various solutions such as are found

in ground water on tungsten minerals, and to determine what natural reagents precipitate it from solutions. He concludes that tungsten minerals are somewhat soluble. The field evidence in Burma undoubtedly supports this conclusion. In this connection I would point out that the natural sulphides of molybdenum and bismuth are also leached, dissolved and oxidised away in their passage from a vein through a soil cap towards the valley deposits in tropical countries.

All observers are agreed that the wolfram, cassiterite, scheelite, and associated minerals emanated in some form or other from the original molten granite, but there are differences of opinion as to the manner in which they were collected and then deposited in the veins where they are now found. It is generally admitted that after the crystallisation of the main mass of the granite a watery and highly acid mother liquor was left in which the tungsten and tin compounds were dissolved. Whether this mother liquor derived its water from the original magma, or from some external source is a matter of speculation, as also is the part taken by mineralising gases in the operations. The facts on which we have to build our theories are these: that tourmaline, which would indicate the presence of both fluorine and boron, is absent; that calcium fluoride is a widely-distributed accessory mineral; that sulphides of various elements are common and persistent; that wolfram and cassiterite occur in pegmatites; and that some of the pegmatites can be observed passing laterally into quartz veins of the ordinary type carrying the minerals. The presence of fluoride and the fact that both tin and tungsten form highly volatile compounds with fluorine, coupled with Daubree's synthesis of cassiterite by the action of water on tin fluoride, are used by the advocates of the pneumatolytic hypothesis in support of their views.

Whatever the inner details of the processes may have been, it seems certain that the mineral veins appeared as a direct result of fractional crystallisation, through a series of varying phases, induced in the original magma by decreasing temperature.

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DISCUSSION.

Mr. F. H. SHARPE said that tourmaline was present in all Australian ores he had handled. He asked if the author had any theory as to the origin of bismuth; he had specimens which suggested that a sort of transmutation had taken place. It was interesting to observe the similarity which existed in Australian and Burmese ores.

Dr. H. PEILE said that figures regarding the cost of mining etc. would be very useful. The future depended on a regular supply at a low cost.

Mr. W. JONES said that in Portugal exactly the same characteristics occurred as in Burma. In Portugal, as in Burma, the wolfram invariably existed at the contact of the Silurian schist and granite. In his opinion the geological maps of Portugal were the best in the world so far as exact information was concerned. What was the average percentage of concentrates in Burma? He considered a mine giving one ton of tungsten concentrates from each 100 tons of quartz was a good mine, and it was difficult to get a higher yield than that. Sometimes mines gave 3 or 4%, but they were soon worked out. His experience, not only in Portugal, but in Bolivia and the Argentine, was that mining tungsten was the most difficult of mining operations. In those areas he had never seen decomposition on such a large scale as Mr. Brown had mentioned, but he supposed it was due to the climatic conditions and the acidity of the soils. So far as tungsten was concerned there was no need to fear American competition. The Americans were considering the advisability of imposing a duty of one dollar a pound on our tungsten products.

Dr. J. H. PATERSON referred to the progress made in machine tool practice owing to the adoption of high-speed steel.

Mr. COGGIN BROWN, replying to the discussion, said that he recognised that most of the wolfram of the world was associated with tourmaline, a fact

which made its absence in the Tavoy field all the more remarkable. He could advance no particular theory on the presence of bismuth, but thought it belonged to a later epoch. Costs of production in normal years could not be judged from war-time conditions; besides, it was impossible to lay down a general figure over such a large area as Burma, especially as the system of working varied greatly on different mines. The cheapest wolfram was that obtained by hydraulic surface deposits. There were mines which had made large profits in the past when the value of wolfram was much below 55s. per unit of WO_3 . He had already mentioned the difficulties of valuation, and that contents varying from 1 to 3% of concentrates had been recorded and were probably underestimates. The only way to arrive at a correct valuation in vein deposits with scattered values of this sort was, in his opinion, by large-scale stoping tests, and the field as a whole had not advanced to that stage yet. Some of the mining engineers of the district adopted a minimum of 1.5% recovery in their calculations, and he thought that that was a fairly safe figure.

Communications.

A CONSIDERATION OF SOME FACTORS AFFECTING THE OXYGEN ABSORPTION OF LINSEED OIL.

BY A. DE WAELE, A.I.C.

The literature on the subject of the oxygen absorption of linseed oil is very rich, the earlier experimenters (Livache, Bishop, Hübl, Lippert, etc.), however, interpreting what is more correctly expressed as increases in weight of the oil on exposure as oxygen absorption or oxygen values. I have already dealt with the incorrectness of this view (de Waele, *Chemical World*, 1914, 3, 11, 300) and given a criticism and résumé of the work of the later experimenters (Weger, Genthe, Krumphaar, and Wilson and Heaven), whose results on theoretical grounds may be considered as approximating more closely to the true oxygen absorption.

A tentative view is held in some technical circles as to the differences in mechanism in the process of oxidation resulting from the use of different driers. An extension of this hypothesis is to the effect that lead and manganese, as driers, perform their catalytic functions in different ways according as to whether the oxidation takes place in a dry or a damp atmosphere.

In consequence of indications that atmospheric conditions appeared to be responsible for variations in the properties of the oxidation products of linseed oil in large-scale practice in linoleum manufacture, some experiments were carried out in which the variation in weight of linseed oil on exposure were considered in relation to the humidity of the surrounding atmosphere. The atmospheric conditions are capable of resolution into two factors: the actual proportion of moisture present, which would be given by the pressure of water vapour, and the relative saturation, i.e., the percentage of moisture present on the maximum amount at saturation.

Since linseed oil on exposure to air not only takes up oxygen, but loses some as volatile products, it is reasonable to suppose that equilibrium conditions of the oxidised product with the surrounding atmosphere will be to some extent determined by the actual proportion of decomposition products present in the latter. Thus, at the point

(5) The relative stability of polymerised oil is clearly shown both by the continual upward tendency of the curve and by the closeness of the ordinates shown at the 33rd and 133rd day periods.

(6) For the period considered it is interesting to note that the more rapidly oxidisable unpolymerised oils (lead and lead-manganese) do not ultimately show such a rapid decomposition (as shown by a fall in the end of the curve) as the less rapidly oxidised oils (unsiccated and manganese oils). The polymerising effect of lead drier (R. S. Morrell; Chem. Soc. Trans., 1918, 113) with production of weak ring formation may be the cause of this.

The influence of moisture on the variations in weight may also be considered in the light of the conclusions reached by Gardner (Circular No. 70, Paint Manufacturers' Assoc. of the U.S.; this J., 1919, 832A), in which the first decrease following on the initial increase due to partial saturation by oxygen is assumed to be due to the evolution of volatile decomposition products of the oxidised glyceride, the second increase being due to decomposition of the glyceride into fatty acids and glyceryl radicle, which latter would then absorb water to form glycerol with increase in weight. The second decrease in weight would then be due to decomposition of glycerol to formic acid etc. The present writer is, however, of the opinion that the liberation of free fatty acid on oxidation is improbable, the high acid values observed in oxidised oils being more probably due to groupings which under suitable conditions of hydrolysis, such as in neutralisation, would function as carboxyl or hydroxyl groups. Such hydroxyl groups attached to ring nuclei formed by inner polymerisation subsequent to oxidation would function in the same way as phenolic hydroxyl groups, the existence of which are, strongly indicated by the high acetyl values of oxidised oils.

ESTIMATION OF PARAFFINS IN COMMERCIAL TOLUOL.

BY E. LEWIS.

With reference to the paper by Mr. H. G. Evans under the above title (this J., Dec. 15, 1919, 402 T), attention may be drawn to a method which was in use in a number of T.N.T. factories during war time.

The determination was actually carried out on the mononitrotoluol and the paraffin content calculated on the original toluol. One litre of mononitrotoluol was subjected to fractional distillation using a three-section Young's evaporator column, collecting that portion which distilled below 160° C. The distillate consisted of a small quantity of mononitrotoluene, together with the whole of the un-nitrated toluene and paraffins. Extraction with 90% sulphuric acid removed the nitrotoluene and sulphonation with 20% oleum and subsequent extraction with 90% sulphuric acid removed the toluene, the residue being the paraffins, which were weighed and a solubility correction applied.

This method avoids the sulphonation of a large volume of toluol, a reaction which is difficult to regulate. The un-nitrated toluol may also be determined by weighing before and after sulphonation. It may be of interest to note in this connection

that during an investigation of the composition of toluol supplied by The Gas Light and Coke Co., Beekton, it was found that the paraffin present consisted almost entirely of isomeric octanes, which in spite of surviving the initial nitrations were decomposed during the more vigorous, final nitration of the toluol into trinitrotoluol.

ESTIMATION OF PARAFFINS IN COMMERCIAL TOLUOL.

BY H. G. COLMAN.

The method described above by Mr. Lewis, whilst probably giving a fairly accurate estimation of the percentage of true paraffin hydrocarbons of the C_nH_{2n+2} series in commercial toluol, does not give a correct figure of the total percentage of saturated aliphatic hydrocarbons in the original toluol. These consist not only of true paraffins, but also to a considerable extent of cyclo-paraffins, which last are to a very material extent attacked and removed both in the original nitration of the toluol and in the subsequent treatment with 20% oleum prescribed by Mr. Lewis.

A RAPID METHOD OF ESTIMATING WATER IN CRUDE CAMPHOR.

BY K. W. LANE, B.A., AND O. F. LUBATTI, DR. CHEM.

The following method has been devised for the rapid estimation of water in crude camphor. Five grms. of the crude sample (in the form in which it was received, "coarse, granular fragments") is placed in a centrifuge tube having a barrel of 25 mm. and a narrow tube of 4 mm. internal diameter, the latter being sealed at the lower end and graduated. In shape it resembles the central portion of a pipette. Resting on the coned junction between the narrow and wider tubes is a small 60° funnel-support of nickel wire gauze, on which, before the introduction of the camphor, is placed a piece of cotton-wool sufficiently large to act as a filter. Thirty c.c. of benzene, which has been saturated with water at laboratory temperature and centrifuged in a suitable vessel before separating the water in a separating funnel, is introduced into the tube, which is then placed in a suitably shaped wooden holder and introduced, balanced by a second similarly treated tube, into a centrifugal machine. After running the centrifuge for a couple of minutes the water in the sample is found in the narrow tube, the insoluble impurities being retained by the cotton wool. The narrow tube, which is calibrated by running in small volumes of water from a burette with a fine point and etching with hydrofluoric acid, gives the percentage of water direct. Any particles of water which may have been retained mechanically by the wool or walls of the tube are disturbed by careful stirring with a glass rod, the tube is re-centrifuged, and a second reading taken. When stirring gives no further increase in the volume of the water in the narrow tube the separation is considered to be complete. In the case of unusually pure samples, the gauze filter may be dispensed with. Using this method, it has been found that constant readings are given by the same sample, and that the addition of a known volume of water to the experimental tube produces, after centrifuging, the theoretical increase of the reading in the narrow tube, showing that there is no appreciable retention of water by the filter.

London Section.

Meeting held at Burlington House on February 2nd, 1920.

MIR. JULIAN L. BAKER IN THE CHAIR.

THE THEORY AND PRACTICE OF LUBRICATION: THE "GERM" PROCESS.

BY HENRY M. WELLS AND JAMES E. SOUTHCORBE, M.Sc.

The considerations which we wish to bring forward in this paper are the results of many years' experience of the problems of lubricating oil and lubrication, studied from a physical and physico-chemical standpoint, assisted by an intimate and daily contact with the lubrication of all types of machinery and prime movers. Our primary object was to elucidate the reason for the obviously superior lubricating efficiency of fatty glycerides over "straight" mineral oils in a large number of practical cases which had come to our notice.

As a corollary to this we had to review the physical principles upon which lubrication depends and to seek an explanation of the peculiar property which has been called "oiliness," "body," etc., by authorities in the past.

It may not be out of place here to review the position of our knowledge of lubricants and lubrication.

Professor C. V. Boys, in his Presidential address to the Physical Society in 1908, crystallised the position very happily in these words:—

"It was found that the lubricating property of oil depended on something which at present is unknown—It is not viscosity—animal and vegetable oils lubricate better, i.e., they are more 'slippery' than mineral oils of the same viscosity, and though the oil trade has known how to make good 'slippery' mixtures, no one at present knows what 'oiliness' is, and this is at the present time an important physical quest of the engineer."

Again, Professor J. S. Brame has said that "the property of 'oiliness' was one of the most puzzling of the properties of oils. By some it was regarded as unnecessary to connect it with viscosity directly, since it was possessed in a much greater degree by some fixed oils than by many mineral oils which had practically the same viscosity."

It is necessary to distinguish clearly between two distinct classes of lubricating practice. On the one hand we have the lubrication of fast-moving shafts etc. supplied with a large excess of oil frequently under pressure, and in this case the frictional values are dependent primarily upon the viscosity of the oil; the mathematical and experimental investigation of these cases have been amply treated by Reynolds, Tower, Lasche, and others. On the other hand we have to deal with slow speeds at high bearing-pressures, frequently with a very limited supply of oil, and it is in these cases that the special property of "oiliness" or "body" is requisite to maintain the film, and it is here that viscosity measurements no longer assist us in the choice of the lubricant.

This view received unanimous support at the recent discussion on lubrication at the Physical Society.

What are the possible physical properties of a liquid which influence its character as a lubricant? They are viscosity, density, capillarity or surface tension, compressibility, and tensile strength. It is true that very little work has been done on compressibility and tensile strength, but from the observations of Worthington and others it would appear doubtful whether they would play a *distinguishing* rôle in the differentiation of oils; and, further, as we shall point out, there is what appears to us an adequate explanation of the nature of oiliness without calling upon these properties.

So far as density is concerned there exists a wide range of petroleum mineral oils possessing specific gravities identical with those of the fatty or fixed oils, hence it is clear that density plays no determining part. Viscosity is, of course, of great significance in the cases of high speed etc. just referred to.

Now it remained for Ubbelohde to point out that only a liquid which "wets" or "spreads over" the solid can constitute a true lubricant, because in order for the liquid to force itself into the narrower spaces of higher pressure it is essential on capillary grounds that the said liquid shall "wet" the solid surfaces.

Consider the case of two eccentric glass surfaces which are being forced together with a drop of mercury or oil between them. Now since the mercury (fig. 1) does not "wet" or spread over the glass, the meniscus in this case will be convex to the liquid, while in the case of the oil (fig. 2), which wets the surfaces, the meniscus will be concave.

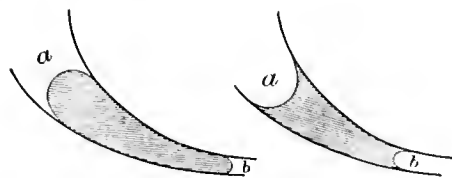


FIG. 1.

FIG. 2.

In the case of fig. 1 (mercury) the tendency on capillary grounds will be for the liquid to gather itself up into a drop and to pull the liquid film in the direction of *a* away from the narrower restricted area of greater pressure at *b*. In the case of oil (fig. 2.) the opposite will be the case. The oil will, owing to its meniscus, tend to be pulled towards *b* or, in other words, will force itself into the narrow spaces.

This is exactly what is required in a lubricant, namely, that it shall penetrate into the narrow spaces between journal and bearing, and from the above considerations one clearly sees that liquids which do not "wet" solid surfaces cannot be described as lubricants.

Only those liquids which "wet" a solid surface possess lubricating power in the generally accepted sense.

Although these considerations appeared to indicate the connection between lubrication and capillarity, it will be seen that they only go so far as to enable us to say that mercury is not a lubricant and that oil is a lubricant. This is a conclusion of considerable theoretical interest, but it is not very

helpful to the oil technologist who desires to differentiate between different classes of oils.

Accordingly we find that these speculations of Ubbelohde led to no practical result.

On the other hand, certain theoretical considerations had led to the conclusion that the permanence of liquid films depended upon capillary relationships, and in particular that *pure liquids would not form stable films*. These principles had not been applied, however, to the problem of lubrication, and, in fact, they were directly contradicted by prevailing practice in which there was a constant tendency towards greater purity, *i.e.*, in the direction of refining the mineral oils which were used, and in the direction of using acid-free oil as a compounding ingredient.

The generally accepted view was therefore that capillarity was not a deciding factor in connection with the phenomenon of "oiliness," and various standard works contain statements to this effect.

It was under these conditions that we began our experiments. We were at once met by the fact that it was not possible to measure the surface tension between oil and the solid metal bearing, and it is probably on this account that Ubbelohde's speculations remained abortive. The usual "surface tension" is that of oil against air, and we confirmed the results of previous investigators, *viz.*, that the results so obtained, for instance, by observing the rise in a capillary tube, shed no light at all on the question under consideration.

At this point we decided to measure the surface tension of the oil against an immiscible liquid in the hope that this might furnish some criterion of "oiliness." On proceeding to measure this interfacial tension, *i.e.*, the surface tension between oil and water, startling results were at once obtained. The method of experiment was by the drop pipette as follows:—The pipette consists of a U-shaped capillary tube provided with a bulb A and a ground glass orifice B. The bulb A is filled with oil to the mark E. By means of the capillary D a very slow flow of oil may be obtained at B by opening the stop-cock C. The orifice is immersed in a beaker of water, and the number of drops formed by the given volume of oil in A is counted. The surface tension oil-water is inversely proportional to the number of drops.*

A series of mineral oils were tested with this instrument, and then a series of animal and vegetable oils and compounded oils.

The following table shows a few results selected from a very large number of trials:—

Table of Interfacial Tension by Drop Numbers of Various Oils Against Water.

OIL.	Mean temperature 70° F.		Tension in arbitrary units.	
	No. of drops at constant orifice and head.			
Paraffinum liquidum	95	100		
0-905 mineral	101	94		Mineral oils.
Solar red mineral	102	95		
Non-viscous neutral	99	93		
Olive	132	72		
Rape	138	68		Fatty oils.
Coconut	161	59		
Lard oil	128	73		

A glance at the table shows the surprising fact that *the interfacial tension against water of the vegetable and animal oils is much lower than in the case of a mineral oil.*

What is more, we were struck by the fact that we had here a test which showed a distinct physical difference between mineral and saponifiable oils independent of viscosity, density, etc., and this difference appeared to be in conformity with the lubricating properties of the oils.

It now remained to enquire what was the reason for the difference in tension. After considerable experimentation we proved that the lowering of the interfacial tension against water in the case of fatty oils was due to their slight content of free fatty acidity.

The following table shows some of the results:—

OIL.	Free fatty acids, calc. as oleic.	Drop no.	Interfacial tension.
0-905 mineral	nil	101	100
98% mineral	1-9	125	80
2% com. fatty acids			
97% mineral	2-8	130	78
3% com. fatty acids			
Olive	2-2	125	80
Olive	4-5	140	72
Rape	2-5	132	76
Coconut	4-1	148	68
Olive (neutral)	0-1	110	92
Rape (neutral)	0-15	108	93

By removing the free fatty acids from the saponifiable oils the tension rises, and by adding free fatty acids to the mineral oil the tension can be lowered.

Now, all commercial animal and vegetable oils contain small quantities of free fatty acids, and even if the utmost care has been taken in refining to remove the acidity, hydrolysis soon sets in and free acids are formed which, in even relatively minute quantity, suffice to lower the surface tension.

The following table shows the percentage of free fatty acids in representative samples of animal, vegetable, and compounded oils on the market:—

	Acidity (as oleic).
Animal oil, pale	4-2 to 25-55
" " brown	12-02 to 30-32
Castor, firsts	0-49 to 1-70
" seconds	2-12 to 7-40
Coconut, Cochio	1-26 to 19-11
Colza, Belgian	1-97 to 3-22
" Stettin	1-41 to 4-48
Lard, pressed	0-28 to 0-71
Olive oil, Algerian	2-52 to 13-72
" Gallipoli	12 to 33-14
Palm oil	24-68 to 56-05
" bleached	14-1 to 27-49
Rape oil, Black Sea, refined	1-82 to 4-34
" East India	1-26 to 4-24
Sperm oil, Arctic no. 1	0-56
" " no. 2	0-42 to 4-34
" Southern	0-7 to 2-86
Tallow	1-55 to 43-71
Standard Brands of Compound Oils on the Market.	
	Acidity (as oleic).
Marine engine oil, D	1-3%
" " " O	5-15%
" " " T	4-5%
Motor oil, X	0-26%
" " Y	0-5%
" " Z	2-5%
Compound steam cylinder	0-4%
Medium gas and oil engine oil	0-25%
	0-38%
Light gas and oil engine oil	1-6%
	1-3%
Heavy gas engine oil	1-28%
	2-5%
Oil engine oil	0-7%

The above table is representative of the general glycerides and commercial compounded lubricating oils in daily use on all types of power plant, and we see that fatty acids are always present to a certain extent. From the foregoing it is demonstrable that:—

1. Capillary effects (hitherto ignored in lubrication) play a fundamental part.

* Donnan, Zeit., f. Physik. Chem. vol. 31, etc.

2. The presence of fatty acids in an oil lowers the surface tension of said oil against water.

3. A neutral glyceride possesses a similar tension to a neutral mineral oil.

4. The addition of a relatively minute amount of a fatty acid to a neutral mineral oil reduces the tension to that of a commercial animal or vegetable oil or compounded lubricating oil.

Now it remains to enquire how far these results influence the theory and practice of lubrication of solid surfaces. It might be argued that the interface between oil and water is a different thing from the interface between oil and metal, and that the conclusions drawn from the one case are not necessarily applicable to the other. As a matter of fact such similarity in effect is not unknown in other instances, and to obtain more conclusive evidence Professor Lewis has measured the interfacial tension between oil and a liquid metal—mercury. (It should not be forgotten that solid-liquid interfacial tension cannot be measured.) His results are as follows:—Pure neutral mineral oil, 100; same mineral oil plus 2% of commercial fatty acid, 89. It is seen that again there is a lowering in the interfacial tension as a result of the addition of the organic acids, and, what is more striking, the relative lowering produced is very much the same as it is in the case of an oil-water interface.

Professor Lewis remarks:—"One may conclude therefore with some confidence that the addition of the organic acids will lower the tension at any metal-oil interface."

Again, as already pointed out, the permanency of films is dependent upon a diminished interfacial tension between the oil and the metal in contact therewith. If such a film is broken the possibility of its uniting again to form an unbroken layer depends entirely upon the interfacial tension being low. Any substance which lowers the interfacial tension causes the liquid to spread over a larger area of the solid. It follows therefore that if a substance be added to an oil which brings about a lowering of interfacial tension, such addition will act favourably as far as lubrication is concerned by preventing a rupture of the liquid film and preventing in turn the metals coming into direct contact.

The capacity for spreading may be considered as partly physical and partly chemical, due presumably to residual valency. The effect is to render the transition layer between the liquid and solid less abrupt. This diminution in abruptness can be brought about by chemical action direct or indirect across the transition layer or by the solubility of some third substance in both phases.

H. S. Allen has recently pointed out that on Langmuir's view oiliness depends on the chemical forces called into play between the active part of the oil molecule and the solid surfaces of the bearing.

Now it is obvious that there is a tendency for chemical activity between the metallic surfaces of bearing and journal and an oil containing free fatty acidity, while such tendencies are less pronounced in the case of a neutral mineral oil. Such a tendency would render the transition between oil and solid surfaces less abrupt, would manifest itself by reduced interfacial tension, and would result in better spreading and consequent increased efficiency in lubrication.

There is little doubt in our minds that the physical rationale of the property of "oiliness" is now explained, and we have confined ourselves so far to a statement of the physico-chemical experiments which we have made and to the development of the physical theory.

Conclusive as it appears to us we have proceeded to test and verify the conclusions by direct friction measurements, and finally by the only real touchstone, namely, the test of experience in a long series of practical trials on all types of machinery and prime movers of the very largest sizes.

We will proceed to consider some of the results in detail.

First we beg to tender our thanks to Mr. L. Archbutt for the frictional test made by him, which he has communicated to the Physical Society at its recent discussion on Lubrication.

Archbutt has made a series of determinations of the frictional coefficient on a Thurston machine under a load of 270 lb. per sq. in. at the very slow speed of 7 ft. per minute.

His results are as follows:—Pure mineral oil, 0.0017; do. plus 1% rape oil fatty acids, 0.0033. He finds that 1% of free fatty acids lowers the coefficient as much as does 60% of pure rape oil (acid-free).

These results have been confirmed by us on an independent machine, as we shall show later.

He has also shown, however, that pure neutral rape oil also possesses a lower coefficient than mineral, and concludes that "these results would suggest that the oiliness or lubricating efficiency of the unsaturated molecules of rape oil was really as great as that of the free fatty acid molecules, but that the acid molecules were much more active in their influence on the hydrocarbon molecules of the mineral oil."

These results, which show that 1% of free fatty acids of rape oil added to a mineral oil are as effective in reducing the value of the frictional coefficient as is the addition to the mineral oil of 60% of neutral rape, are striking confirmation of the above described principles, and coming from a totally independent and unbiassed experimenter afford great support to our contention that it is not the glyceride but free fatty acids in a compounded oil which improves its lubricating value.

Through the courtesy of one of the largest engineering firms in the country we have been enabled to make a series of measurements in a large friction testing machine, the results of which we here reproduce:—The test journal of the machine—which is of the Thurston type—is 3.8 in. diameter, giving approximately one foot peripheral per revolution. The length of the journal is 6½ in., and in all our experiments the load was 200 lb. per sq. in. The machine is provided with a revolution counter and a drum upon which the reading of the arc is automatically traced. The driving motor was operated by a variable speed controller, and all care was taken to ensure steady and constant speeds. The journal and bearing were thoroughly cleaned before each test by washing with toluol, and finally rotated against velvet pads to remove all superficial dust and moisture. The experimental temperature was kept between 60° and 64° F.

The values of the frictional coefficients calculated from the curves obtained directly from the machine are given in the table on p. 51 T.

By way of illustrating our point, four oils were chosen of identical viscosity and density but differing in composition thus:—

These results afford great support to the views expressed above, and, coupled with our experience in practical lubrication, about to be mentioned, confirm our explanation of the property of oiliness and open out a new and invaluable field in the manufacture of lubricating oil.

We should here mention that this principle of making lubricating oils by adding to mineral oils small quantities of fatty acids or substances which

lower the interfacial tension has been accepted by the Patent Offices in all civilised countries.

We should add a word here with regard to the possibilities of corrosion.

tendency, while one containing the lower members will possess a de-emulsifying tendency. This is a feature which we have tested by shaking oils and water at various temperatures for long periods of

Oil.	Viscosity at 60° F. Seconds Redwood.	Sp. gr., at 60° F.	Total acidity, calc. as oleic acid.	Mean temp. of expts.	Peripheral speed in feet per minute.	Quantity of oil used for test.	Arc.	Coefficient of friction.
A. Pure mineral	973	0.909	nil	62	11	5 c.c.	40	0.0084
B. 97% Pure mineral 1% Pale cylinder * 2% Com. fatty acids	973	0.900	1.9%	62	11	5 c.c.	25	0.0052
C. 80% Mineral 20% Olive	980	0.908	0.3%	63	11	5 c.c.	40	0.0084
D. 40% Mineral 60% Olive	970	0.907	0.9%	64	11	5 c.c.	35	0.0073

* Added to keep the viscosity constant.

As will be observed from the table above, all compounded oils which have been and are in daily use for years contain notable amounts of free fatty acidity, yet one rarely hears of any active corrosion.

In our case we add only very minute amounts of fatty acids, and the quantity is strictly limited and controllable. But where fatty glycerides are employed the amount of acidity which can form is potentially very large because hydrolysis is constantly going on, giving rise to the production of free acids.

During recent years a great deal of attention has been devoted to the study of the colloidal characters of the fatty acids, and it has been shown that while the lower members of the fatty acid group possess relatively little colloidal character, the higher members are highly colloidal in character. Donnan and Potts have shown that there is a gradation in these properties as one ascends the scale, lauric acid occupying a sort of intermediate position. Also the lower members of the series possess strong acid characteristics, while the higher members are very weakly acid. Now the fatty acids which occur in commercial oils are never pure chemical individuals, but are mixtures in varying proportions of a considerable number of fatty acids. Coconut oil, for example, is characterised by containing appreciable percentages of the lower members of the series, while rape oil rarely contains anything but the higher members.

It is only to be expected, therefore, that the behaviour of these oils will differ in accordance with the fatty acid groups which predominate in them, and it is possible to reproduce the capillary properties of any particular animal or vegetable oil by adding suitably chosen fatty acids to mineral oil.

Consider the case of a steam engine using saturated steam, where there is a tendency for condensation of water to occur in the cylinder and valves. It follows in such a case that the presence of a substance in the oil which lowers the surface tension against water will in such circumstances assist in the formation of oil films by enabling the oil to spread more readily or by overcoming the tendency of the water to wash the oil film off.

There is a certain type of lubricating phenomena to be considered where the oil is brought into contact with water and it is desirable that the oil shall either separate itself rapidly from the water (de-emulsification) or, conversely, that it shall mix or emulsify with the water.

Now the phenomenon of emulsification is dependent upon the colloidal properties of the oil, while de-emulsification is brought about by a greater concentration of hydrogen ions. Consequently one would expect the oil containing the higher members of the fatty acid group to possess an emulsifying

time, and to a great extent we have been able to substantiate this view as the result of experiments.

After the soundness of our theory was clearly demonstrated in the laboratory it remained for us to put it to the touch of actual practice to prove its real value in the world of engineering.

Not a single failure has occurred in practice of an oil or an oil "essence" made on the "germ process" as a reliable lubricating oil when used for the purpose for which it was supplied. The meaning of "oil essence" is explained later.

We use the expression "germ process" to describe the oil made by using one or more fatty or other acids with one or more mineral oils, because the world has been taught for generations to look upon acid as the deadliest enemy to good and safe lubrication. As the *germ* is the first principle of organic life, by analogy we call a suitable acid when dissolved in mineral oil the "germ," which gives to mineral oil its life and activity as a more perfect "instrument of lubrication."

We do not claim that a "germ process" oil is better for all conditions of lubrication, but for many purposes where lubrication depends upon the oil alone and not upon any mechanical means, such as pressure, to maintain a continuous film.

We have proved to our entire satisfaction that the addition of fatty oils to mineral oils for many purposes of lubrication is unnecessary and a waste of valuable material, and that for such purposes oils combining small percentages of suitable free fatty acid or acids with suitable mineral oils are at least equal to oils compounded on the old formulæ for those purposes. Therefore many of the old formulæ and specifications for compound oils are obsolete. Where the expression "compounded oils" is used, it indicates such old formulæ oils. Also, "mineral oil" indicates in many cases one or more than one mineral lubricating oil. "Fatty oil" includes tallow and other "fats" used for lubrication.

Gas engines.—It has always been considered that the addition of coconut oil to a mineral oil gave the best results. Refined rape oil or other fatty oil was also used in conjunction with coconut oil to the extent of about 5% of each; that is, about 10% of fatty oil, 90% mineral oil. Especially on large horizontal units such a compounded oil was deemed essential. For smaller units smaller proportions down to 5% total fatty oil to 95% mineral oil; though some small units apparently required heavily compounded oils.

So far as our business is concerned, "germ process" oils have entirely displaced such compounded oils for horizontal and vertical engines up to the largest units of both types.

Some gas engines run on "straight" mineral oil with good results, but roughly it can be claimed that ten units run on "germ process" oil to one on straight mineral.

Oil engines.—Compounded oils, and for some types "straight" fatty oils such as olive oil, have been considered essential for oil engines. For several types it was thought necessary to use a compound of one-third refined rape oil to two-thirds mineral oil. That oil has been entirely superseded by an oil with a slightly higher percentage of fatty acid than the average "germ process" oil for oil engines, but not over the maximum considered safe in practice. For other oil engines, land and marine, for which compounded oils were thought necessary, their place has been taken by oils made from one or more fatty acids with mineral oils of suitable quality.

The quality of the mineral oil with which the acid is blended is a very important factor in lubrication. The germ process gives to a mineral oil of fair merit that property lacking for some purposes; while it increases the lubricating value of a "good" oil, making it still better for many purposes. In both cases they become more economical.

Steam cylinder lubrication.—It is almost universally considered that for "perfect" lubrication of steam cylinders with certain types of valve gear (as one example, "Corliss"), and for engines working under certain conditions—say with much condensation in the cylinder—a compounded oil is essential.

"Germ process" oil incorporated in very small proportion with the correct mineral cylinder oil gives equally good results on engines with Corliss valves up to over 3000 h.p. working at 160–170 lb. per square inch pressure, superheated 480–500° F.; on horizontal engines with Corliss valves up to 750 h.p. up to 160 lb. pressure without superheat. Various mineral cylinder oil bases to correct "germs" in different but small proportions give thoroughly good lubrication on vertical and horizontal engines of many types, sizes, and pressures.

Oils for steam turbines, crank cases of vertical steam, gas, and oil engines.—The property most essential in oil for steam turbines, namely non-emulsification, is the one which largely governs successful crank-case lubrication in vertical engines. In such cases and in turbines water from condensed steam or from the cooling system falls into the crank case and gets "churned" as it is pumped round with the lubricating oil. For these conditions one has to know something about impurities in the water, as the water is the deciding factor. The "germ" must be carefully adapted to the water as well as to the oil, and the oil to the mechanism. Where one oil is used for cylinder and crank-case lubrication (say, of gas and oil engines) the "germs" can be balanced to give what for convenience can be described in a general way as a positive effect in the cylinder where extra capillarity is required, and a negative effect in the crank case where emulsifying is undesirable, and where the engine is steam, of the splash lubrication type, to give just enough emulsion and no more for its intended work in the cylinder, also in the crank case, where it must emulsify. The germs, for they are many, can be so adapted when one thoroughly apprehends their properties, to give perfect lubrication in all such cases.

Marine steam engine bearings—open type.—For a good heavy marine engine oil it has always been considered necessary, and is so to-day, to use from 10 to 25% thickened or blown oil—as a rule, thickened rape oil. This gives great viscosity, also very good "lathering" properties to the oil.

The standard specification for marine bearing oil for one of our semi-Government departments is a compound of about 20% "of fatty oil"; but the total fatty acid content must not exceed 1%. This has now been successfully replaced by "germ-process" marine-engine oil.

On February 5, 1918, we filed our patent* for oils made on this new process, and immediately its "publication or communication" was prohibited by the Admiralty, who carried out trials over many months on about fifteen ships of the mercantile marine, including a fair proportion of liners. The result was quite satisfactory.

At this stage we decided to supply the fatty acids in a form, which we term "essence," mixed with mineral oil and which can be conveniently used on board ship; about 2½% is added to the mineral oil as required. The result justified all claims. The consumption of mineral oil was considerably reduced, hot bearings were cooled, and thrust blocks were kept cool by the addition to mineral oil of suitable "germs." The trials were a complete success.

We think it desirable to say that it may not be advisable for oil users in general to buy fatty acids, to mix them in their oil and imagine all advantages mentioned will accrue. Suitable acids must be chosen for the purpose.

Conclusions.—We have shown (1) that fatty compounded oils are unnecessary for many purposes of lubrication; (2) that fatty oils are not essential for such work; (3) that fatty acids can entirely and completely displace fatty oils for those purposes.

Our thanks are here recorded to the following for invaluable assistance in our experiments and trials:—Prof. W. C. McC. Lewis; Messrs. Armstrong, Whitworth and Co., Ltd.; Corporation of Manchester Electricity Dept.; Mr. M. Shenton.

DISCUSSION.

Mr. L. ARCHBUTT said that he had made some friction tests with the process in the laboratory. It was obvious that under the conditions which obtained in an ordinary journal bearing running at a good speed, in which a film of liquid oil completely separated the journal from the bearing, fatty acids could be of no advantage, and that the only way in which they could act beneficially would be by improving the oiliness of the lubricant. The Thurston machine, as generally used, would not have measured anything but the fluid or viscous friction, but by running this machine at a very low speed and with a good load on the bearings a certain amount of contact friction had been obtained, and in all the tests he was about to describe the speed was 7 ft. per minute and the load about 270 lb. per square inch. A few preliminary tests were made with "Tonicol," containing about 25% of free fatty acid, and these showed that 2½% of "Tonicol" mixed with mineral oil lowered the coefficient of friction to the same extent as about 10% of ordinary commercial rape oil. Further tests had been made with a paraffin-base oil of practically the same viscosity as rape oil at 60°–65° F., the temperature in all the tests. In each series the machine had been run with the mineral oil alone until the friction had become steady; then, without stopping the machine the oil had been changed to a mixture containing fatty acids prepared by himself from rape oil, and then a final check test was made with mineral oil alone. The result of the first series was as follows:—

	Mineral oil.	Rape oil fatty acids.	Friction coefficient
1.	100.0	oil.	0.0066
2.	99.5	0.5	0.0049
3.	99.0	1.0	0.0045
4.	98.0	2.0	0.0042
5.	100.0	nil.	0.0066

This was conclusive. In further tests nearly 80% of perfectly neutral rape oil, specially prepared, had been required to reduce the friction as low as 2% of rape oil fatty acids; 100% of the rape oil had actually given a rather higher average friction, but this small difference was within the limits of the error of the method.

* Eng. Pat. 130,677; see this J., 1919, 674A.

The results were as follows:—

	Mineral oil.	Neutral rape oil.	Friction coefficient.
1.	100	nil	0.0066
2.	90	10	0.0065
3.	80	20	0.0062
4.	60	40	0.0053
5.	40	60	0.0047
6.	20	80	0.0041
7.	nil	100	0.0043
8.	100	nil	0.0062

Finally, another series of four tests had been made, comparing the mineral oil, commercial rape oil, and neutral rape oil, and the results were as follows:—

	Oil	Friction coefficient.
1.	Mineral oil	0.0078
2.	Neutral rape oil	0.0050
3.	Acid rape oil (2.4% fatty acids)	0.0045
4.	Mineral oil	0.0078

These results appeared to him to prove the authors' contention as regards the friction-reducing value of free fatty acid to be perfectly sound, but he could not agree that the lowering of the interfacial tension between oil and water, or even between oil and mercury, though very interesting and important, was a sufficient explanation of the nature of oiliness. The authors' own experiments showed that the interfacial tension between nearly neutral rape oil and water was almost as high as that of mineral oil, and much higher than that of acid rape oil. Yet his (Mr. Archbutt's) experiments showed that neutral rape oil when mixed with mineral oil lowered the friction, and there was very little difference between the coefficients given by neutral rape oil and acid rape oil. Mr. Deeley, in his recent communication to the Physical Society, had expressed the opinion that oiliness was a chemical property, and that the unsaturated molecules of the lubricant united with the solid metal surface and formed a new surface which opposed less resistance to shear than the unlubricated metallic surface. The remarkable papers by Langmuir and Harkins strongly supported this view. Langmuir said: "Since energy must be expended in breaking apart a solid, the surfaces of solids must contain more potential energy than do the corresponding number of atoms in the interior. Since the potential energy is probably electro-magnetic energy in the field between the atoms, the interatomic forces are more intense on the surface than in the interior. This intense field (unsaturated chemical affinity) is one of the causes of the phenomena of condensation and adsorption" (J. Amer. Chem. Soc., 1916, 38, 2293), and in a later paper, after describing a number of experiments, Langmuir said: "These results afford the strongest kind of confirmation of the theory that the spreading of films on surfaces is determined by the shapes of molecules and the relative activities of the different portions of the molecules" (*ibid.*, 1917, 39, 1881). "Consider first the adsorption of a liquid containing active groups. The molecules will become orientated and will pack into the surface layer in much the same manner as in the case of oil films spread on the surface of water" (*ibid.*, 39, 1901). W. B. and F. K. Hardy (Phil. Mag., 1919, 38, 32) had come to a similar conclusion. They said that "the function of a lubricant is to reduce the energy of the surface, and thereby to reduce the capacity for cohesion and the resistance to slip when two composite surfaces are applied the one to the other." All recent work pointed to the fact that it was the chemically reactive and unsaturated constituents of lubricants which promoted oiliness, and that they did so by forming new composite films on the surfaces lubricated, with lower surface energy and opposing less resistance to shear, and the great activity of free

fatty acids was quite in accordance with this theory. Referring to the statement in the paper as to the occurrence of hydrolysis and formation of free acids in oils, he did not think much hydrolysis took place in an ordinary bearing, but it did in a steam cylinder. He agreed that so long as the quantity of free fatty acid in a lubricating oil did not exceed 1 or 2%, which was as much as was necessary, as his own tests had shown; $\frac{1}{2}$ % did most of the work, and an addition up to 2% did not make a very great difference; this small quantity of fatty acid could not do any possible harm and was far more useful than a larger quantity of fatty oil, besides the saving it effected. A little misconception might arise from the statement attributed to him that 1% of free fatty acid lowered the coefficient as much as did 60% of pure rape oil. That was not commercially pure rape, but a special neutral rape oil which he had made in the laboratory, and which was not an article of commerce. He was struck by the fact that 80% of mineral oil and 20% of olive oil made no difference to the friction coefficient, and that 60% mineral oil and 40% olive oil only lowered it to 0.0073. He wondered what that olive oil was. The sentences quoted in the paper from the communication which he himself had made to the Physical Society needed the context, because what he meant was that, comparing acid rape oil with neutral rape oil, there was not very much difference, so that the effect of the free fatty acid in the rape oil was not nearly so great as when it was added to the mineral oil.

Mr. ARNOLD PHILIP agreed with Mr. Archbutt that the authors had not succeeded in showing what oiliness was. Oiliness could not be explained as being merely due to the wetting of the rubbing surfaces by the lubricant, because glycerin, which had a very similar viscosity and density to that of some oils, would certainly wet ordinary bearing surfaces, but as a lubricant it was, of course, valueless. The property of "oiliness" possessed by lubricants, excluding the views to which Mr. Archbutt had referred, had up to the present only been measurable by the results obtained when using the lubricant on machinery under ordinary running conditions or by special tests made with mechanical oil testers, such as that of Thurston. The authors' results, however interesting, were not referred to these standards, and as an explanation of oiliness were, he considered, meaningless. General commercial practice had shown for many years past that the admixture of small amounts of fatty oils with mineral oils did distinctly improve the mineral oil for many purposes. He believed that this had been stated to be due to the formation of metal soaps on the bearing surfaces. In any case he had this explanation very strongly indicated in his own practice by the fact that some fourteen years ago difficulty had been experienced with machinery running at low temperatures, due to solidification of the sperm oil which was used as a lubricant. To remedy this a pure non-freezing mineral oil had been substituted for the sperm oil. The result had been that the mineral oil had come away from the bearings full of turbidity, caused by flakes of solid material, which consisted of oleates and stearates of iron and copper stripped from the bearing surfaces. These had been formed by the use of the sperm oil in the first place, and this solid coating of the metal bearing surfaces had afterwards become displaced and washed out by the mineral oil subsequently used. As this mineral oil was a perfectly clear, water-white liquid the presence of the flakes of oleates and stearates in it after use had been very evident. After the stripping of these heavy metal soaps from the bearing surfaces by the mineral oil it had been found that the friction had increased, but whether this was actually due to the removal of the stearates and oleates was not quite clear. Some eight or ten years ago he had

had the important function of the free fatty acid in such lubricants still further impressed upon him by the manufacturer of a fatty oil, for making blended or compounded fatty and mineral oils for lubricants, who had informed him that he always made an oil containing a large percentage of fatty acids. In fact, he spoke of the use of this acid fatty oil as being a special secret, and stated that it gave particularly satisfactory results for making blended lubricating oils. Consequently, when about two years ago the proposal of the authors for using mixtures of fatty acids and mineral oils had been referred to him by the Admiralty, he had approved it and reported that it appeared, on the face of it, to be good not only on account of the saving in fatty oils which it effected, but also because the use of compound mineral oils and fatty acids appeared technically correct for certain lubricating purposes. What the authors had done which was novel was to use a fatty acid as distinct from a fatty oil for blending with mineral oils. He deprecated the use of the term "germ" as applied to the process and also the use of the word "essence," both of which were misleading.

In a written communication PROF. F. G. DONNAN pointed out that a great deal of the matter brought forward apparently as novel by the authors had been already discussed and elucidated by himself. For example, he had shown experimentally so long ago as 1899 (*Z. Physik. Chem.*) that the interfacial tension between a fatty or hydrocarbon oil and an aqueous solution was dependent mainly on (a) the presence of alkali in the water, (b) the presence of free fatty acid in the oil. In the paper referred to the quantitative dependence of the interfacial tension on these two main variables had been experimentally measured, and the cause of these phenomena fully elucidated. Many other important interfacial tension and concentration effects had been investigated in later papers by his pupils (work of Lewis, Potts, Barker, Ellis, Powis) working under his direction.

MR. T. THORNTON asked if the authors had used aqueous emulsions as lubricating agents. The function of a lubricant was to keep metal surfaces separate with as little expenditure of energy as possible, and if an aqueous emulsion were used an effect was obtained similar to that of a ball bearing. It gave the superiority of a ball bearing over an ordinary friction bearing. Instead of having a reduction of about 60% by introducing fatty acids, an aqueous emulsion containing about 25% mineral oil would reduce the expenditure of energy by at least 85%; e.g., when using 100 amperes at a known voltage with a full mineral oil and then turning over to an aqueous emulsion containing 25% of some mineral oil, the current expenditure had fallen to 15 amperes. Such an emulsion could be prepared by homogenising a mineral oil and soap solution containing 2½% of ordinary soft soap. These emulsions had been used in certain special cases with a good deal of success for something like twelve months on fairly heavily loaded bearings at medium speeds.

DR. W. R. ORMANDY said that it seemed impossible to draw the conclusion that interfacial tension was the basis of what was called "oiliness," because certain experiments with an oil containing free fatty acids as against water gave a certain result which was more or less directly comparable with the results obtained in practice on a metallic surface, and it would have been wise to have tried the experiment of oils in other liquids, with which they were immiscible in order to see in the first place if the interfacial tension between the other liquids bore anything like the same ratio to the results on a metallic surface. He thought that Mr. Archbutt had adequately explained the bearing of Langmuir's theory of surface energy in its

relation to the question of lubrication. Langmuir would contend that on the surface of the metal there were certain free fields, and these free fields or residual valencies could attract a certain number of fatty acid oil molecules until they were saturated. Langmuir had proved by many experiments that once they had satisfied all the free valencies, as it were, on the surface and had got it covered with a layer one molecule thick, the attraction afterwards did not count, and any addition to the oil produced a very small effect. Similar results had been obtained by Hardy, who had shown that a layer one molecule thick was sufficient to prevent adhesion of one glass surface to another. Therefore if there were sufficient of the fatty acid, or whatever material it was, adsorbed on the metal surface to satisfy the valencies of that surface, any further addition would produce a comparatively slight effect. That was borne out by Mr. Archbutt's first series of experiments.

PROF. J. S. S. BRAME, in referring to the work which had already been done on this subject, particularly by Archbutt and Deeley, and described in their papers before the Physical Society, said that Messrs. Hardy had carried out experiments on the static friction with different fatty acids ranging from those of low molecular weight to high molecular weight, and it was shown, not only in the case of these fatty acids, but in the case of alcohols and amines, that increase of molecular weight was associated with a progressive decrease in static friction. That seemed to have a very important bearing on the question under discussion, and he asked whether the authors found that there was any relationship between the molecular weight of fatty acids and the friction of the hydrocarbon oil mixture. Did the authors find that a saturated fatty acid of approximately the same molecular weight as an unsaturated fatty acid had the same effect? In one of the tables the interfacial tension with water between olive oil and coconut oil containing approximately the same amount of fatty acid was practically the same, viz., 72 to 68. The Hübl iodine absorption number for the fatty acid from olive oil was about 90, and for the fatty acid from coconut oil it was 9, which suggested that perhaps there was not much difference between the action of a saturated and an unsaturated fatty acid. Was there any connection between the action of a given fatty acid with different types of hydrocarbon oils where there was considerable variation in the amount of the unsaturated hydrocarbon present—for example, the average shale lubricating oil, which was rich in unsaturated hydrocarbon molecules, and a "straight" petroleum lubricating oil, where the proportion of unsaturated molecules was very much less? He agreed with Mr. Archbutt and Mr. Arnold Philip, however, in thinking that the authors carried the matter very much further towards arriving at a solution of what "oiliness" really meant.

MR. A. A. DRUMMOND said that some years ago a letter had appeared in "The Engineer" dealing with the corrosion of steel balls through the action of lubricating oils, including mineral oil and fatty oils, like olive oil and lard oil. In no case, after six months, had there been any sign of corrosion except with castor oil, and this had shown quite appreciable corrosion visually, particularly under the microscope. Not only had castor oil shown this and differentiated itself from the others, but the same difference had been noticed in the case of the fatty acid of castor oil as compared with the corresponding acids of the other fatty oils. Perhaps the author could offer some explanation of the difference exhibited.

MR. SOUTHCORBE, in reply, said that they were not anxious to make out a theory. They had

observed certain differences in interfacial tension of commercial lubricating oils against water, and then stated the further fact that the presence of fatty acidity lowered the frictional coefficient, and they supported that by a further fact that the presence of this fatty acidity also lowered the tension against mercury surfaces. Even if residual valency were the explanation it would manifest itself in the same way, *i.e.*, it would cause a lack of abruptness of the transitional layer and that diminution in the abruptness of the transitional layer would manifest itself as a lowering of the interfacial tension, and therefore it was only a question of which side one looked at the problem from. With regard to residual valency, unsaturation was the essence of Langmuir's theory, unsaturation in the sense of the presence of matter of which the valencies were fully saturated so far as ordinary chemical reactivity went, but which had probably some unsaturated linkages not satisfied. The hydrocarbons which constituted mineral lubricating oil were more highly unsaturated than were any of the compounds of the fatty glycerides, taking saturation in the chemical sense. From the experiments of Mabery and others it was seen that the mineral oils which had high viscosity and were very much used as lubricants were characterised by very high unsaturation. In the two oils dealt with in the paper, and from Mr. Archbutt's experiments, he suggested that the one which Mr. Archbutt called mineral oil, 100, was more highly unsaturated than was the pure rape oil, so that if the view point of the authors had not been proved, he did not think they were very much further advanced from a practical point of view by the Langmuir idea. Mr. Arnold Philip had suggested that the effect of the fatty acid was to form metallic soaps. If this were the case in any short period of time then it must be a definite proof of the adsorption idea. The case which Mr. Philip had brought forward was not parallel. He had spoken of a bearing running on sperm oil and changing that for a mineral oil. The sperm oil might have been in the bearing for a very long period and would necessarily have corroded the bearing to a certain extent. On the other hand, the fact that the coefficient of friction was lowered instantaneously by the addition of fatty acids, as shown by the authors' figures and confirmed by Mr. Archbutt, was proof that that had nothing to do with wholesale soap formation. There must be adsorption which would secure an almost instantaneous lowering. He could not agree that the example given of glycerin disproved the "wetting" view. Glycerin was quite a good lubricant, but its hygroscopic properties made it commercially useless because in practice it rapidly becomes an aqueous solution. It was true that Prof. Donnan had shown how to determine the adsorption on surfaces of fatty acids in oils in alkali solution, but the object of his paper was to take a series of fatty acids, from acetic to stearic, and determine the interfacial tension against alkali solution by the drop pipette, and he (Prof. Donnan) had succeeded in showing that as the series was ascended the surface tension became very much lower, but he was not concerned with a diminution in interfacial tension against water. Prof. Donnan's results were of a totally different order of magnitude. His drop number went up from, say, 100 to 200, 300, and 400, against dilute alkaline solutions, which was what he was measuring. Donnan's work did not refer to the problems of lubrication. It was true that the drop pipette method had been applied by Prof. Donnan and his collaborators, but what the authors were claiming to show was that such an instrument could be used in the examination of an oil. They were glad to take this opportunity of stating that Prof. Donnan's work on the drop pipette had been inspiring and of value. Mr. Thornley had spoken of aqueous emul-

sions of lubricating agents. He (the author) did not know the conditions under which these emulsions had been employed, although their use was not new. The only point there which would interest him was the question of the "wetting." Such emulsions were generally pumped under forced feed, and it did not concern the present paper. In reply to Prof. Brame, they had not done anything to show the variation of the frictional coefficient with the molecular weight of the acid. They had, however, carried out a series of experiments with regard to emulsification and de-emulsification which would be published shortly. He did not think that a research as to the effect of degrees of saturation would be very fertile, because, as he had already pointed out, there was equal unsaturation in a great number of the mineral oils themselves possessing high frictional coefficients as compared with the unsaturation of the fatty glyceride. In reply to the other point raised by Prof. Brame, there was a very distinct influence on the properties of the final lubricants by adding different fatty acids to any given mineral oil, and there was also a different result with a compounded oil obtained with one given series of fatty acids with different hydrocarbon oils. With regard to Mr. Drummond's query on the question of corrosion of steel balls and the effect of castor oil, he did not think that came within the scope of the paper. The only thing he could suggest was that with castor oil acids were formed by oxidation which would be very dangerous from the corrosion point of view. These, however, were not the acids used in compounding lubricating oils.

Professor C. V. BOYS, in a letter to the Scientific and Industrial Research Department, writes as follows:—

"I notice that the conclusions arrived at by Messrs. Wells and Southcombe depend on the determination of oil-water surface tension, or rather of comparative values, by the method of counting of drops. As a general proposition I very much doubt the conclusion that the number of drops in a given volume is inversely as the surface tension, although it may be for some particular pair of liquids or with a particular difference of density. The mere fact that the difference of density of the two liquids, which is one of the operative factors in determining the drop size, is not included in the statement and appears to be ignored is alone sufficient to raise very serious doubts; but quite apart from this the very complicated conditions which determine the moment of instability or the breaking off of a drop themselves appear to me to be incompatible with any such convenient conclusion. I say convenient conclusion because if the method were correct in principle it could not be surpassed in ease of application.

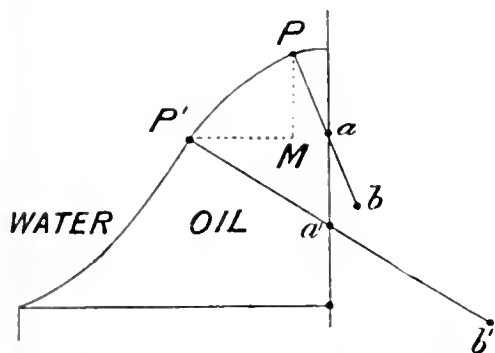
"My object is not destructive criticism, but a desire to propose an absolute method of determining the actual surface tension of the water-oil surface in CGS or other definite units. It seems to me of the first importance as the conclusions of the authors are based on comparative measures of surface tensions that the method of making the measures should be above suspicion; further, mere comparative measures, even if correctly comparable, are less desirable than absolute measures.

"Now, in all methods of finding drop size by allowing liquid to enter the drop, however slowly, until it breaks away, the hydrostatic pressure of the liquid at any level within the drop is unknown, but the following relations hold. If P is any point on the surface of the drop (which is a figure of revolution) and $P a b$ a normal to the surface, then if the hydrostatic pressure in the oil at the level P exceeds that in the water at the same level by the amount p dynes per sq. cm., t the surface

tension in dynes per cm. and p are related thus:—

$$p = t \left(\frac{1}{P_a} + \frac{1}{P_b} \right)$$

a is the point where the normal cuts the axis of revolution, and b is the centre of curvature of the profile of the drop at the point P . At a lower point P' the same is true, but here the difference of pressure is less by the amount (density of water—density of oil) $\times PM \times g$. All distances in cm. and g in this

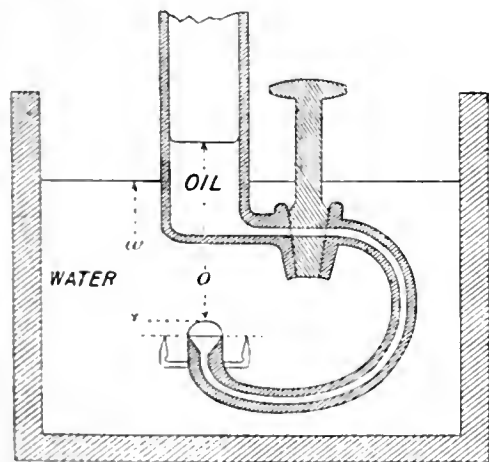


latitude 981. As the drop grows there comes a time when the differences of hydrostatic pressure and the lengths Pa Pb in different parts of the drop are becoming incompatible, and instability results. The breaking depends on differences of hydrostatic pressure within the drop as compared with the surrounding water which determine the rupture. The actual hydrostatic pressure is not known, and its determination by elimination among the measurements of Pa Pb at a number of points P would be very tedious and inaccurate. In a paper* 27 years ago I showed how these forms may be drawn by a device there illustrated with far greater speed and accuracy than had hitherto been possible, but all methods of determining surface tension in slowly growing drops which are allowed to grow until they break away have the disadvantage that there is nothing easily tangible or measurable except the number. In the first edition of my book on soap bubbles I showed a number of photographs, 23 in a cycle, of a growing drop in a water surround, but the coming of instability led to movements of such rapidity even in these large and slow moving drops as to indicate how hopeless measurements at the critical phase would be.

"What I want now to suggest is that the surface tension should be determined absolutely by direct measures of the hydrostatic pressure within and without at the highest point of the drop. This point has the advantage that the contact here of the circle of curvature is a contact of the third order, and the radius of curvature r can at this point be determined with an accuracy far greater than the Pa and Pb referred to above. The Pa and Pb become the same at this point, and thus the right-hand side of the equation becomes $2t/r$. The method of finding r and p and thence deducing t will appear after the construction of the proposed apparatus is clear.

"This consists of a tube of the form shown, which may be held in a micrometer vertical slide (not shown) so that it may be moved gently through known very small amounts vertically. The tube contains some of the oil to be tested, and the stop-cock is used to stop all movement of oil when once it has reached the drop-forming surface and during the time that it is being immersed in the water and secured to the micrometer slide. The water is contained in a cell with plate glass sides through which all that is happening may be observed. The shape and radius of curvature of the drop will ultimately be determined by means of an enlarged photograph taken with a lens of, say, 4 in. focus, and in order that the scale of magnification may be known two points a definite distance apart attached to the drop end of the tube are photographed at the same time. The straight line joining them is exactly central. It would be advisable also to make the points at the same level as the drop end of the glass tube as will appear. All photographs of drops and other measurements made with a cathetometer are done when the stop-cock is open and equilibrium is attained. Then $w \times$ density of water $\times g$ is the hydrostatic pressure of the water at the level of the top of the drop; $o \times$ the density of the oil $\times g$ is the hydrostatic pressure of the oil in the drop at the same level, and this product is the greater of the two when the drop shows any convexity. They are equal when the oil surface is plane. Calling this difference p , and r the radius of curvature obtained from the enlarged photograph $p = 2t/r$.

"Now in order to make an experiment the tube is placed in position with the stop-cock closed, and is moved vertically until w and o are as nearly as possible inversely as the respective densities. Then, all being quiet, the stop-cock is opened. The surface of the oil at the drop orifice should be very slightly if at all curved. The slide is moved until as far as the eye can tell the surface is plane. If the surface reflects light this can be done with very great accuracy. w and o measured at once give the density, and a thermometer in the water the temperature. Now the tube is raised a small known amount by the micrometer screw of the slide, and the oil surface becomes slightly convex, and the curvature is determined by an enlarged photograph. w and o may again be measured with the cathetometer, but the micrometer screw will give a more accurate measure, for it gives a direct measure of the lifting of the apparatus. This gives the increased effective hydrostatic pressure of the oil subject to two small corrections. (1) The water level falls by an $\frac{1}{m}$ part of the elevation, m being known from the area of the free water surface and the area of the tube and stop-cock stem intersected by the water surface. The oil depth is reduced by the height of the drop, which can be got from the photograph of the drop and two points, and also by the fall of oil in the tube due to the small content of the drop. These may be calculated with great accuracy, or with a good cathetometer w and o may be directly measured each time.



* "The drawing of curves by their curvature." Phil. Mag., 1893, July, p. 81.

"A series of photographs and measurements of drops successively more developed give a number of values of surface tension in absolute measure, and the consistency of these will give a measure of the accuracy with which the determination is made.

"I am not sure what is the best diameter of the drop end of the tube. The authors have probably found this already by experiment. The diameter has to do with the possible development of the drop shape, and extremes either way give less convenient forms for the purpose of measurement.

"The above observations are directed solely to the experimental operation of finding the oil-water surface tension. I am not discussing the propriety or utility of this determination in relation to journal friction.

"If the water and oil were absolutely immiscible and uncontaminated either by the other it would be a matter of indifference whether a water drop were formed in a surround of oil hanging downwards or an oil drop were formed in a surround of water hanging upwards: the surface tension would be the same, and the sizes and forms of the drops for the same p would be identical. If, however, as is probably the case, there is some degree of miscibility and contamination, the conditions might well not be the same, if so little water and much oil are different materials from little oil and much water. If there is a difference, I do not know which measure would more truly give the quantity that the authors seek to find. Further, I am not satisfied that there is justification for using the water-oil surface tension any more than the oil-air surface tension. However, the real object of this letter is not to go into this question of justification, but to give the best help I can in a class of experiment in which I have a good deal of experience."

Manchester Section.

*Meeting held at the Grand Hotel on Friday,
December 5, 1919.*

MR. JOHN ALLAN IN THE CHAIR.

THE ACTIVATED SLUDGE PROCESS OF SEWAGE PURIFICATION—LARGE SCALE OPERATION.

BY EDWARD ARDERN, D.S.C., F.I.C.

In view of the satisfactory results obtained from the experimental investigation of the activated sludge process as applied to the treatment of the sewage received at the Withington Sewage Works (Chorlton-cum-Hardy) of the Manchester Corporation, which were described in 1916,* a large-scale continuous-flow demonstration plant, designed to treat 250,000 gallons of sewage per day, was installed at these works in October, 1917, with the view of studying the process under working conditions.

In the present communication it is proposed to give a description of this installation, together with a general account of the results obtained during the two years it has been in operation.

The earlier investigations were practically confined to the operation of the process on the "fill and

draw" system, in which both the aeration of the sewage and settlement of the sludge were effected in the same vessel. There are several objections to this intermittent method of operation, chief of which are the fall required for the discharge of the clarified effluent and the constant attention involved.

In translating the process to the working scale, therefore, the plant was so designed as to allow of its continuous operation, which not only obviates the above-mentioned disadvantages, but at the same time renders the process more adaptable to the treatment of the fluctuating flows of sewage received at the disposal works.

The plant, which was designed by Messrs. Jones and Attwood, Ltd. (Stourbridge), in consultation with the author, occupies one-third the area of one of the existing sedimentation tanks, and may be described as follows:—

Aeration Chamber.

The aeration chamber, 100 ft. long by 22 ft. wide (overall) and 6 ft. deep (water level to diffuser), is divided by four longitudinal walls so as to form a channel 4 ft. wide and 500 ft. in length. Its capacity is 55,000 galls. The floor of this channel is constructed on the ridge and furrow system, the ridge having a slope of 35° with the horizontal. Aeration is effected through rows of diffusers placed in the furrows transversely to the flow of sewage. The diffusers are each 1 ft. square over all, so that their total area is approximately one-seventh of the aeration tank area. The ratio of the net diffusion area to total tank area is 1:10. An overhead system of air pipes is provided, and the air is admitted to the diffusers through a series of down pipes, arranged so that one pipe serves two diffusers. Valves are attached to the various distributing pipes, so that the air supply may be regulated and distributed uniformly.

Settlement Tank.

Separation of the activated sludge from the purified effluent is effected in a deep pyramidal tank, 23 ft. 6 in. by 26 ft. 6 in. in plan, and 23 ft. 6 in. deep from water level to apex of tank. The sides of the tanks have a slope of 60° to the horizontal.

The mixture of effluent and sludge is admitted to the centre of the tank through a 15 in. pipe within a guard chamber 6 ft. square, and discharges into a deep wrought-iron bucket suspended from the top of the pipe. This arrangement was designed by Mr. W. Clifford with the view of dissipating the energy of the incoming flow by the creation of eddies within the guard chamber, and thus to produce, as far as possible, a quiescent condition at the bottom of the guard chamber, and so allow of efficient settlement of the sludge. The final effluent leaves the tank over four corner peripheral cells. The total capacity of the settlement tank is 27,000 galls.

Sludge Circulation.

The sludge is discharged by hydrostatic head from the apex of the tank through a 6 in. pipe to a separate chamber fitted with diffusers, from which it is returned by air lift to the aeration chamber.

Surplus sludge is either pumped from the return sludge chamber on to drainage beds, or is removed by siphon to the existing sludge well and discharged mixed with the sludge from the sedimentation tanks on to the land.

Air Supply.—For this purpose a belt-driven Reavell compressor, designed to deliver 500 cub. ft. per minute at anything up to 15 lb. pressure is employed. The machine is driven by a disused tandem high-pressure air compressor, which has been converted into a steam engine by removal of the air piston and the provision of a new driving pulley.

* This J., 1917, 65—68.

Flow measurement.—A Lea flow recorder has been installed at the exit end of the plant, and the purified effluent is measured as it passes over one or more V notches. Four such weirs are provided so as to obtain an accurate record of considerable variations in actual flow.

Air consumption.—This is measured by calculations from piston displacement and engine speed.

The sewage prior to treatment in this plant is screened and flows through two detritus tanks, the combined capacity of which allows of 40 minutes' detention in normal dry weather.

When the plant was first brought into commission a flow of sewage was passed through at the rate of 100,000 galls. per day for fifteen hours out of the twenty-four (6 a.m. to 9 p.m.). During the rest of the day the flow was shut down and the contents of the installation circulated by means of the air-lift placed in the sludge pit connected with the settlement tank. In this way satisfactorily purified effluents were obtained during the period required for the production of an active sludge.

As the amount of sludge increased the volume of sewage treated was gradually raised without interfering with the degree of purification effected.

When the sludge present in the aeration chamber had increased to 20% by volume, measured after one hour's settlement, the flow of sewage was stopped and the contents of the plant circulated as previously described until the liquor was free from ammonia. At this stage the activity of the sludge was such that the installation was capable of treating 100,000 galls. of sewage per day with practically complete nitrification. During the next few days the flow of sewage was gradually increased from 100,000 galls. to 250,000 galls. per day, with the maintenance of a high degree of nitrification. In this manner an adequate supply of activated sludge was produced in rather less than a month, to enable the plant to be brought into full and regular commission.

Following are a series of average results over various periods, which demonstrate the capacity of the plant under varying conditions:—

110,000 galls. to 250,000 galls. per day (average 150,000).

It will be seen that the effective capacity of the plant is well above the 250,000 galls. per day for which it was designed.

The figures given for the results obtained when treating the sewage at the rate of 500,000 galls. per day cover a period of eight weeks continuous operation at this rate. Towards the end of this period the sludge showed signs of diminished activity, which necessitated a return to a lower rate of flow. It is, however, evident that the plant is capable of operation at this high rate for limited periods when the sewage is diluted by appreciable rainfall without interfering with its efficiency.

The chief characteristics of the effluents obtained are their remarkable clarity, the ease with which they pass the Royal Commission suggested standard of 20 parts dissolved oxygen absorbed per 100,000 after incubation with aerated water for five days at 65°, and their low bacterial content, which averages about 20,000 organisms per c.c. (G.P.B.), which represents a reduction of 98% on that of the sewage treated.

Special attention has been paid to the amount of sludge produced in the process. Two typical determinations are given below:—

Sludge Production.

	1919.	
	Sept. 18th to 30th	Oct. 1st to 31st
Volume of sewage treated (gallons)	3,360,000	6,310,000
Volume of surplus sludge (galls.) per million gallons sewage treated	23,200	35,300
Water content of sludge	99.4%	99.6%
Dry solids (lb. per million gallons sewage flow)	1,342	1,348

The above volumes of sludge are the quantities actually removed from the plant (during the respective periods) by siphon from the sludge chamber, connected with the settlement tanks, during the operation of the plant.

Sludge of increased density and consequently reduced volume could be obtained if the operation of the plant was interrupted during sludge removal. Where actual volume of sludge is a deciding factor

Results in parts per 100,000.

	Dry weather sewage.				Wet weather sewage.			
	Maximum air supply*		Low air supply†		Low air supply.*			
	Sewage†	Effluent	Sewage†	Effluent	Sewage†	Effluent	Sewage†	Effluent
Four hours' oxygen absorption	3.53	0.66	3.90	0.73	2.53	0.67	2.93	0.60
Free and saline ammonia	3.00	1.53	2.66	2.31	1.94	1.63	2.11	1.54
Albuminoid ammonia	0.59	0.10	0.66	0.115	0.46	0.13	0.53	0.11
Nitrate and nitrite (as NH ₃)	—	0.64	—	0.14	—	0.37	—	0.34
Dissolved oxygen absorption	—	0.93	—	0.93	—	1.00	—	1.34
(Royal Commission test)								
Suspended solids	18.0	3.0	—	—	13.0	3.4	—	—
Vol. of sewage treated (galls. per day)	250,000		250,000		340,000		500,000	
Air consumption (cub. ft. free air per gallon of sewage)	1.7		1.0		0.8		0.0	

* Uniform rate of flow.

† Flow roughly proportional to rate sewage is received.

† Screened and detritus-free sewage.

The volume of sludge employed in the aeration chamber has varied from 15 to 30% measured by volume after 1 hour's settlement.

The figures for air consumption include the volume required for working the air lift, returning the sludge from the settlement tank to the aeration chamber, which amounted on the average to about 10% of the total volume employed. The volume of thin sludge circulated by the air lifts varied from

in respect of its disposal, ancillary settlement tanks should be provided whereby a reduction in volume can readily be effected. These two periods are given as illustrating the great variation of the density of the sludge in circulation, and this question of sludge volume will be referred to later.

In connection with this important question of sludge production the following information is of interest in regard to the respective suspended solid

content of the screened and detritus-freed sewage treated and of the effluent yielded.

Suspended solids parts per 100,000 (excluding colloids)	Average Total	Sept. 18th to Oct. 3rd 1919.	
		Mineral matter	Organic & volatile matter
Sewage	12.7	3.5	9.2
Effluent	1.1	0.4	0.7
Difference	11.6	3.1	8.5
Equivalent to lb. per million gallons	1,160	310	850
Dry matter calculated from sludge production	1,346	306	1,040

Judging from these results, the sludge produced (dry matter) is greater than is accounted for by the difference between the suspended solids contained in the sewage treated and in that passing away in the effluent.

It would thus appear that any resolution of the sludge by carbonaceous fermentation is considerably more than counterbalanced by the solid matter resulting from the flocculation of colloidal solids and the growths of numerous higher organisms.

Attention should be called, however, to the difficulty of obtaining over a long period a true sample of such a heterogeneous mixture as sewage, and it is possible that some of the grosser suspended matters escape sampling.

The sludge as it is withdrawn from the plant is dark greyish brown in colour and possesses a not unpleasant earthy odour, something like that of exposed seaweed. It is quite flocculent and has the appearance of an iron or alumina precipitate. Its density varies very considerably, but it may be reduced to a fairly constant water content of 98.5% by settlement alone. When the percentage of water is reduced to 90% by treatment on drainage beds, centrifugal action, or pressure, the sludge is no longer fluid, but is of the consistency of a fairly stiff jelly, due to its gelatinous nature.

Mention has been made on a previous occasion of the high bacterial content of this sludge, which varies from 10 to 30 million organisms (G.P.B.) per c.c. A special study has been made of the great variety of protozoa present in the sludge, but up to the present without revealing their rôle in the purification process. A study of the fauna of the sludge does, however, afford valuable information with respect to its presumptive activity.

The following is a typical analysis of the dried sludge: Loss on ignition, 75.2%; mineral matter, 24.8%; total nitrogen as N, 6.4%; phosphate (P_2O_5), 3.8%.

With the view of testing its value as a fertiliser certain field trials have been carried out under the advice of Dr. E. J. Russell, F.R.S., Director of the Rothamsted Experimental Station. A plot of land at the Withington Works, upwards of an acre in area, was divided into 45 plots, the area of each of which was 108 sq. yds. Three crops, viz., spring wheat, mangolds, and cabbage were grown in triplicate, so that each crop was cultivated on 15 plots divided as follows: Three control plots without manure; three plots treated with sulphate of ammonia equivalent to 40 lb. of nitrogen per acre; three plots similarly with sulphate of ammonia equivalent to 80 lb. of nitrogen per acre; while the remaining six plots were manured with activated sludge such that the nitrogen added was equal to that contained in the sulphate of ammonia.

The results obtained with the spring wheat crop are recorded in the accompanying table.

It will be seen that on the average the addition of the sludge has doubled the weight of grain produced, and further that the fertiliser value is equal to that of the nitrogen in the form of sulphate of ammonia, or, in other words, the nitrogen in the sludge is readily available for plant nutrition. This

Crop—Spring Wheat.

Treatment	Corn.					Straw	
	Plots					Total cwt.	Per acre cwt.
	No. 1 lb.	No. 2 lb.	No. 3 lb.	Total lb.	Bushels per acre		
Control unmanured ..	17	16	13	46	11.5	2½	34
Ammonium sulphate 40 lb. N per acre	35	24	37	96	24.0	2½	41
Activated sludge 40 lb. N per acre ..	36	31	22	89	22.2	2½	38
Ammonium sulphate 80 lb. N per acre	35	35	26	96	24.0	3	45
Activated sludge 80 lb. N per acre ..	37	37	34	108	27.0	2½	41

is in accordance with the results obtained by other investigators.

Unfortunately, both the other crops proved failures, the mangolds mainly by reason of too late sowing, and the cabbage as the result of prolonged drought immediately after planting and of the appearance of the cabbage maggot.

While no legitimate conclusions may be drawn with respect to these crops, it may be said that the plots manured with activated sludge showed 100% increase over the control in the case of the mangolds, and from the manured cabbage plots about three times the weight of marketable (shop) plants were obtained as compared with the control plots.

A general idea of the character of the land used for these trials may be obtained from the following results of examination of the soil, carried out by Mr. Ernest G. Gaul, M.Sc., of the Frankland Laboratory, Manchester University, to whom the author is indebted for valuable assistance in connection with this work.

The soil was "air-dried" in the boiler-house. Samples were taken (1) from the S.W. end of the field, (2) from the N.E. end of the field.

Mechanical analysis.

	Soil No. 1 1-3%	Soil No. 2 0-6%
Stones
Soluble matter	5.22%	5.68%
Gravel	0.68	nil.
Coarse sand	4.08	2.92
Fine sand	28.22	24.69
Coarse silt	20.12	22.68
Fine silt	26.40	28.64
Clay	2.29	3.63
Moisture	1.78	1.70
Organic matter	10.42	9.72
	99.21	99.66

Chemical analysis.

Moisture	1.78	1.70
*Organic matter	10.42	9.72
Ferric oxide	6.1	6.05
Aluminium oxide	5.89	5.44
Calcium oxide	0.76	0.76
Potash	0.54	0.51
Phosphoric acid (P_2O_5)	0.43	0.40
*Containing nitrogen	0.195	0.218
Available potash	0.04	0.03
Available phosphate (P_2O_5)	0.113	0.074

In conclusion, it may be stated without hesitation that the two years' experience gained with this installation has demonstrated that the activated sludge process is a practicable process as applied to the purification of the sewage received at the Withington Works.

It is also satisfactory to record that fears with respect to the impracticable amount of scientific control required for the successful operation of the process, with regard to the uniform distribution of air, and in connection with the use of diffusers

have all been found to be without foundation. As a matter of fact, one of the outstanding features of the experience gained with this plant is the lack of trouble with diffusers. As stated previously, the plant has now been in operation for two years, and it has not been necessary to renew any of the 300 diffusers employed. Only eight have been removed from the tank (by undoing the union joint), and these have been replaced in good condition after brushing and blowing. The air pressure (3.1 lb.) is not increasing, so that there are no indications that the diffusers are becoming clogged. On the other hand, further information is required, particularly with respect to the conditions controlling the character and activity of the sludge with specific reference to the role of the higher organisms. This question is at present receiving attention on account of certain occasional difficulties that have arisen, due to the sudden and abnormal "bulking" of the sludge in circulation.

The important problem of dewatering the resultant sludge preparatory to drying has been studied with special reference to the use of centrifugal force both with and without preliminary treatment of the sludge. Comparative trials with filter pressing are about to be undertaken, and it is hoped that it will be possible to publish in due course the results of the research, for which the writer is only partly responsible.

The author has to thank Mr. H. Edwards, manager of the Withington Works, for the capable control of the operation of the plant described, and Mr. P. Gaunt, research chemist, for valuable assistance with respect to the investigations carried out during the past nine months. He is also indebted to the Rivers Committee of the Manchester Corporation for permission to publish the results obtained.

DISCUSSION.

MR. W. THOMSON asked whether a method of separating the solid matter had been tried similar to that adopted in the case of peat, *i.e.*, grinding, the use of high pressure steam, or the electrolytic method. He had noticed that in the elbows of the River Irwell where sewage was precipitated it was filled with a mass of red worms. Had these any purifying effect on the sewage?

MR. J. DRUMMOND PATON mentioned that during an investigation of the humidification of cotton a grey slime had been produced as a result of the water used, and a brown spot formed which came out as a distinct streak on the cloth. The slime when kept at 76° developed brown bodies about $\frac{1}{4}$ in. long. Did these bacilli, whether of fungoid or animal growth, absorb oxygen from the air, which was evidently necessary to produce this coloration, and retain in their own structure an excess of nitrogen, and was there an excess of nitrogen in activated sludge by reason of this same process?

MR. SPEIGHT mentioned that in the author's earlier experiments it had been found that although a satisfactory effluent could be obtained with aeration, yet in order to maintain the activity of the sludge the process had to be carried through to complete nitrification. Frost also produced an inhibitory effect. Had the plant itself been sufficient to maintain the necessary activity?

MR. H. N. MORRIS was of opinion that the success of the activated sludge process depended very largely upon the adoption of an economical method of dehydrating the sludge sufficiently to employ it as a fertiliser. What result could be obtained by the use of a centrifugal?

MISS ROSA ROBINSON asked over what period of time the experiments on the use of fertilisers had been carried out, as she had been informed that some years ago sludge had been tried as a fertiliser near London, and though for a time it proved a

successful fertiliser, yet ultimately it had tended to clog the soil, turning it into a clay-like mass.

DR. ARDERN, in reply, said that the sludge used for the field trials was simply dried on a hot plate, so that the product contained about 15% moisture. The nitrogen content was about 5%, and consequently about four times the weight of sludge was applied as compared with the actual amount of ammonium sulphate used. Mr. Ross had raised a very important point, *viz.*, the relative importance of the time of reaction as compared with the volume of air employed, a question which he was not fully prepared to answer. While it was advantageous to the process to have as long a contact with the sludge as possible, this involved the building of a plant of large capacity. At the designed rate of 250,000 gallons per day there was an average period of retention in the aeration chamber of about four hours. By doubling the rate of flow the average rate of retention was roughly halved, and if practically the same air supply were maintained there was a corresponding reduction in the air consumption. He was not prepared to express an opinion as to the economic limit between volume of air supplied and period of aeration allowed for. He thought the most desirable course was to aim at the lowest possible capacity consistent with the maintenance of satisfactory effluents, for the reason that the more air that could be supplied to the diffusers the less likelihood of trouble ensuing; and, of course, the less tank capacity used the lower capital and sinking fund charges there were on the plant. The main purification change was effected very rapidly, the colloidal matter being removed almost immediately by surface action on exposing the sewage to an intimate contact with the sludge, and the main function of aeration appeared to be to maintain the sludge in a state of reasonable activity, the actual absorption of air being very small indeed. The admission of air into the sewage stream had the effect of creating circulatory action, and thus exposing to the atmosphere a varying surface of the mixture of sludge and sewage. The process was being carried out very successfully by purely mechanical means, without the use of compressed air. While it was true that the volume of sewage dealt with at the Withington works was small as compared with the total flow for the city of Manchester, which was something like 55 million gallons per day, the capacity of the plant under observation was as large a unit as would be provided for a works dealing with the flow received at the Withington works, and was consequently a working scale plant. In connection with the dewatering of the sludge, a continuously operated centrifuge had been used for some months, though the results had not as yet been particularly encouraging, the water content being only reduced to 85%. The effect of electrification had been tried, and it was proposed to continue the experiments, especially in connection with the use of sludge presses. The presence of worms of any type in the activated sludge was accidental and not normal. Dr. Russell had under consideration the question of nitrogen content of the sludge, and was endeavouring to prepare an accurate balance sheet of the nitrogen as between the sewage treated and the effluent leaving the tank. A number of determinations had already been made, but the sewage was such a heterogeneous mixture, and there was so many small errors in sampling, that the results were rendered uncertain. The effect on the cloth, mentioned by Mr. Paton, was probably due to iron organisms which were sometimes found in settlement ponds, though, as a rule, they were not present in the sludge. There was no smell noticeable from the air escaping from the sewage, and very little likelihood of it containing any amines

or allied nitrogenous compounds. The inhibitory effect of frost had been practically negligible, the only result being a rather reduced amount of nitrification. The agricultural trials referred to in the paper had been undertaken during the previous season only. There was, however, no reason to anticipate any deleterious cumulative effect, which could only arise from an undue grease content; activated sludge contained very little greasy matter. The trials referred to by Miss Robinson probably related to the use of ordinary sewage sludge, which had not only a very much lower nitrogen content than activated sludge, but was liable to contain a much higher percentage of grease.

Communications.

THE “HEAT-RISE” METHOD OF TESTING SULPHURIC ACID AND OLEUM.

BY R. CURTIS AND F. D. MILES.

In the manufacture of sulphuric acid and oleum by the contact process it is essential for satisfactory control to be able to determine rapidly and frequently the strengths of the acids in the various reservoirs of a unit. In 1916 the authors were led to consider the various methods available, in order to find one suitable for use on the oleum plants of H.M. factories.

The method of titration with alkali requires care, skill, and time, particularly when used for oleum samples. As the most accurate means of estimating total acidity it is necessarily used for the standardisation of other methods. It has, however, the disadvantage of being less sensitive to small variations in free trioxide content of oleum than a method which estimates this free trioxide directly and is not immediately concerned with the total acid contents. Direct determination of the percentage of free SO_3 is achieved by the method of titration with water (Parker, this J., 1917, 692); but here also more skill is required than can be relied on if the estimations are to be carried out by operatives on the plant.

A method of estimation depending on the rise in temperature consequent on mixing oleum and sulphuric acid of about 93% was worked out by Howard (this J., 1910, 3). Amounts of 100 grms. of each of the acids were mixed in a small silvered vacuum flask. It was found by us that for plant purposes the use of much larger amounts of acid—about 400 grms. of each—was a marked improvement. The mixture cools slowly and the resultant temperature can be read with fair accuracy. Only the simplest apparatus is required—a straight-

sided earthenware mug holding about a pint, two measuring cylinders, and a thermometer. The thermometer should have the scale marked inside the stem and should respond rapidly to changes of temperature. It may conveniently be encased in a thin steel tube filed thin at one end and pierced with a number of holes to allow the acid to have free access to the bulb.

The method of carrying out the test is given below. The standardisation was carried out with samples of oleum and acid the composition of which had been very carefully determined by alkali-titration. The results were plotted as graphs, and from the smooth curves the values given in the table were read off. They have received substantial confirmation from other observers.

In every case the samples should be cooled to 25°C . at least.

(A) *Test for oleum* (of not more than 25%). 200 c.c. of oleum (temperature $a^\circ\text{C}$.) is poured quickly, with vigorous stirring, into 200 c.c. of sulphuric acid at $b^\circ\text{C}$. in the mug. The rise of temperature $= c - \frac{1}{2}(a+b)$, where c is the maximum temperature of the mixture. The percentage of free SO_3 is found from Table I. After mixing the liquid should not fume.

Variation in the strength of the sulphuric acid between the limits of 92.5 and 94.5% was found to have no influence on the result. In five exactly similar experiments the rises of temperature were 23° , 22.8° , 22.5° , 22.5° , 23.2° , showing a maximum variation of 0.4° from the mean (or 0.2 – 0.3% free trioxide). The results are reliable within 0.5% free trioxide, or 0.1% H_2SO_4 .

(b) *Test for sulphuric acid* (96–100%).—The procedure is as above, but the acid is poured into the oleum (20–25%). Rise $= c - \frac{1}{2}(a+b)$, as before. Table II. is used for this case. After mixing the liquid should fume. If it does not fume, test again, as in c. Variation in the strength of the oleum from 18 to 25% was found to be without effect if free SO_3 were in excess at the end of the test. The results are reliable within 0.3% H_2SO_4 .

(c) *Test for sulphuric acid* (88–96%).—100 c.c. of the acid sample (temperature b°) is mixed with 300 c.c. of oleum (20–25%, temperature a°). Rise $= c - (3a+b)/4$. Table III. is used here. After mixing the liquid should fume. As before, small variations in the strength of the oleum have no effect. The error should be less than 0.5% H_2SO_4 .

The procedure described is in practice extremely simple and takes only a few minutes. It was carried out by the men and girls engaged in operating the plant, and was found to yield excellent results at Queen's Ferry, Gretna, Avonmouth, and other of H.M. factories. In one instance it was used to carry out half-hourly tests of each reservoir of an oleum unit for many weeks together. The test may

TABLE I.

TABLE II.

TABLE III.

Rise.	% free SO_3	Rise	% free SO_3	Rise.	% H_2SO_4	Rise.	% H_2SO_4	Rise.	% H_2SO_4
1	0.5	21	12.1			6	93.8	26	92.6
2	1.1	22	12.8			7	98.5	27	92.3
3	1.6	23	13.4	2	99.9	8	98.1	28	92.1
4	2.2	24	14.1	4	99.5	9	97.7	29	91.8
5	2.7	25	14.7	6	99.3	10	97.4	30	91.6
6	3.3	26	15.4	8	99.0	11	97.1	31	91.3
7	3.8	27	16.0	10	98.7	12	96.7	32	91.0
8	4.4	28	16.7	12	98.4	13	96.4	33	90.8
9	4.9	29	17.4	14	98.1	14	96.1	34	90.6
10	5.5	30	18.0	16	97.8	15	95.7	35	90.3
11	6.1	31	18.7	18	97.5	16	95.5	36	90.1
12	6.7	32	19.4	20	97.3	17	95.1	37	89.8
13	7.3	33	20.1	22	96.9	18	94.9	38	89.6
14	7.9	34	20.8	24	96.7	19	94.6	39	89.4
15	8.5	35	21.5	26	96.4	20	94.3	40	89.1
16	9.1	36	22.2	28	96.1	21	94.0	41	88.9
17	9.7	37	22.9	30	95.8	22	93.7	42	88.7
18	10.3	38	23.6			23	93.4	43	88.5
19	10.9	39	24.3			24	93.1	44	88.2
20	11.5	40	25.0			25	92.9	45	87.9

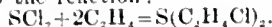
also be recommended for many other plant operations where the strength of sulphuric acid is required, and it has already been used in connection with sulphuric acid concentration, acid mixing, nitric acid manufacture, and sulphonation.

The data on which the tables are founded were obtained from acid and oleum samples which were almost pure, with the exception that they contained sufficient suspended matter to render them cloudy. In the case of acids containing much impurity some slight deviation may be expected. Thus, at H.M. factory, Pembrey, it was observed that oleum made from sulphuric acid recovered from T.N.T. waste acid always gave a greater rise of temperature than is given in the tables, i.e., the figures for SO_3 content had to be reduced about 1%. With oleum made from sulphuric acid recovered from nitrocellulose or nitroglycerin waste acid no such deviation appears to occur.

A SYNTHESIS OF $\beta\beta$ -DICHLORDIETHYL SULPHIDE (MUSTARD GAS).

BY J. E. MYERS AND H. STEPHEN.

On November 20, 1917, we were invited to undertake research on the synthesis of mustard gas. We were informed at the time of the existing synthesis of Victor Meyer (Berichte, 1886, 19, 3260), and owing to the unsuitability of this method we made the suggestion that the compound might be formed according to the reaction:—



We were informed that this reaction did not give the desired compound, and we were given to understand at a much later period that prior to our work attempts had been made to prepare mustard gas by this method, but these had failed to give more than a 7% yield.

After preliminary experiments and a consideration of the work of Guthrie (Quart. Journ. Chem. Soc., 1860, 12, 116; 1861, 13, 134) and Niemann (Liebig's Annalen, 1860, 113, 288) we decided to test the suggestion which we had made at the outset.

Ethylene was prepared by passing the vapour of ethyl alcohol over pumice saturated with orthophosphoric acid heated to 220°C ., and purified to remove vapours of alcohol and water. The ethylene was passed into sulphur dichloride contained in a wash bottle which was cooled. A vigorous reaction took place, and when no further absorption of ethylene was observed the liquid was of a bright yellow colour. During the reaction a considerable amount of hydrogen chloride was evolved.

The crude product was tested for toxicity by application to the skin. A slight smear on the forearm caused acute blistering from the wrist to the elbow and general swelling of the whole arm.

The liquid was found to give on distillation some 20 to 25% of sulphur monochloride, and independent analysis gave some 65% of mustard gas. This preparation was carried out on December 31, 1917, and was repeated frequently during the following week.

Being satisfied of the great toxicity of the product and of the probability that the method could be conveniently used for the production of mustard gas, information of the method and a sample of the product were sent on January 6 and 8, 1918, to the authority inviting us to help in the work.

A consideration of these early experiments showed: (1) That the sulphur dichloride contained sulphur monochloride, and (2) that there was a secondary reaction in which sulphur dichloride chlorinated the mustard gas. The latter effect was no doubt due to the decomposition $2\text{SCl}_2 = \text{S}_2\text{Cl}_2 + \text{Cl}_2$, which was hastened by the heat of the first reaction.

We also examined the sulphur dichloride, and worked out a method for the preparation of the pure substance. In our later experiments we started from this pure dichloride, diluting it with monochloride as desirable.

We then turned our attention to the problem of avoiding the secondary reaction of chlorination. The question of the action of sulphur monochloride on ethylene was being examined elsewhere.

Our next experiments therefore consisted of allowing the dichloride to drop on to a cooled surface in presence of an excess of ethylene. In this way we were able to prepare the product continuously, and when the cooling surface was at about 8°C . we frequently obtained deposits of crystalline "mustard gas." Although this result indicated a high degree of purity in the product, it had the great disadvantage that the solid was very easily chlorinated by succeeding drops of dichloride. We were able, however, to prepare rapidly and conveniently a product which contained 70% of mustard gas with a melting point varying in different samples from $+1^\circ$ to -6°C . This series of experiments was commenced on January 30, 1918, and was demonstrated a day or two later to representatives of an important works concerned in the matter, who set up a semi-large-scale apparatus which by the end of February produced a material very similar to the laboratory product. This we believe to be the first works preparation of mustard gas in this country.

We still sought to reduce the chlorination of the mustard gas, and it was suggested by Sir Richard Threlfall and Prof. A. Lapworth that the chlorination might be overcome by spraying the dichloride into an atmosphere of ethylene, so that the former might be so quickly combined that it would have no opportunity of spoiling the product. In collaboration with Prof. Lapworth and Mr. F. Royle we tested this method, using an atomiser of the scent-spray type whereby the dichloride was sprayed by means of the ethylene. Our first experiments were carried out in a four-litre flask, but eventually we used a 200-litre vessel.

The reaction proceeded in a very satisfactory manner, as will be seen from the following table of typical results. The reaction was almost instantaneous, and consequently we were able to prepare the substance rapidly and continuously:—

Starting material.	M.pt. of crude product. $^\circ\text{C}$.	% Mustard gas by distillation.	M.pt. of mustard gas fraction. $^\circ\text{C}$.
68% SCl_2	—	70	12
80 " "	—	80	10
68 " "	0	88	9
75 " "	2	93	8
80 " "	4	87	5

The variations in the results are due to modifications in the apparatus used. It was found that the best starting material was a mixture of 75% of dichloride and 25% of monochloride. During the reaction some of the latter is used up.

This method we found to be capable of producing a very suitable toxic material, and the method of working could be readily modified to give a product which had a suitable freezing point and which deposited no sulphur.

During the work we investigated the possibility of quickly removing mustard gas from the exterior of shells, and after experimenting with a number of oxidising agents we found that a solution of sodium hypochlorite containing sodium bicarbonate was most efficient in this respect, the hands or pieces of apparatus being almost instantaneously freed from the toxic substance.

In concluding this very brief summary of our work we wish to thank Sir Richard Threlfall and

Prof. Dixon and Lapworth for the kind help which they gave us in many ways.

Chemical Department,
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Received Jan. 14, 1920.

SUGAR IN OAT STRAW AND CATTLE FOODS.

BY S. HOARE COLLINS AND ARNOLD SPILLER.

Most practical farmers attribute much virtue to sugar in cattle foods, but few data have been published on the proportion of sugar in many of the commoner foods. Bodmer, however, has recently referred to the sugar in desiccated coconut (*Analyst*, 1920, 45, 18). The matter is of practical importance, if only because good results may be erroneously ascribed solely to a high priced condimentary food rich in sugar, when the cattle are being fed also with common foods equally rich in sugar. Kellner appears to have ignored the value of the sugar in fodder and meals. The superiority of early cut over late cut oat straw is probably due to the greater content of sugar, which renders the early cut more palatable and nutritious. The fermentation of moistened straw converts the sugar into alcohol, lactic acid, and butyric acid, all of the hope that others will be induced to make further if much advantage is gained thereby.

The following preliminary results are given in the hope that others will be induced to make further investigations:—

Description of foodstuff.	Total sugar. %	Invert sugar. %
Earth nut cake, new, now in use at Cockle Park	8.46	nil.
Palm kernel cake, new, now in use at Cockle Park	3.05	trace.
Oat straw, cut before quite ripe (north country practice)	5.61	not determined.
Oat straw, yellow part hand-picked from same sample	6.32	5.19
Oat straw, green part hand-picked from same sample	6.47	5.5
Oat straw, cut green 18 months ago; sugar perhaps diminished by fermentation	2.89	Not determined.
Oat straw, cut fully ripe 18 months ago; sugar perhaps diminished by fermentation.	1.5	Not determined.
Oat straw, from Midlands, cut fully ripe in 1918, sugar perhaps diminished by keeping	Nil	
Oat straw, from Midlands, 1919 crop, cut greenish	5.00	4.18
Castor cake*, old laboratory sample	4.63	Not determined.

* Poisonous unless seed has been deprived of its ricinin.

From the above results it will be seen (1) that there is ample evidence that when cut green, oat straw may contain upwards of 6% of sugar, mostly invert; (2) that when cut fully ripe the sugar is mostly lost.

The amounts of sugar were determined by Fehling's volumetric method. Some difficulty was found in obtaining clear solutions. The cuprous oxide, instead of separating in bright red clots, leaving a clear liquid on standing, formed a yellow muddy liquor, with the reduced copper persistently remaining in suspension. In many cases the colloidal nature of the solution, after clearing with lead acetate, was due to the presence of fatty substances or essential oils, which we find can be removed by a previous extraction with ether. The use of animal charcoal was found to be of little value.

To determine the sugar, we prepare an aqueous solution, using 5 grms. of food, extract with ether, add 5 grms. of lead acetate and 250 c.c. of water. This mixture is allowed to stand overnight. After filtration 50 c.c. of the solution is acidified with 5 c.c. strong hydrochloric acid and warmed gently

so that it reaches 60° C. after twenty minutes. Any lead chloride is filtered off, anhydrous sodium carbonate added in excess, and the filtered liquid titrated with Fehling's solution.

The invert sugar content of the food is determined in the same way, omitting the heating with hydrochloric acid.

THE CHANGE OF REFRACTIVE INDICES OF FIXED OILS WITH TEMPERATURE.

BY A. F. JOSEPH, D.SC., F.I.C.

In a paper on the above subject (this J., 1919, 392 T), by C. Harold Wright, a means of calculating the refractive index of a fixed oil at a particular temperature from the known result for another temperature is suggested. His formula—

$$n = (n_1 - 1)(1 - 0.0076t_1)/(1 - 0.0076t) + 1$$

(where n and n_1 are the refractive indices at temperatures t and t_1 respectively)—is developed from Gladstone and Dale's expression for the refractivity constant $(n-1)/d$ (independent of temperature), Mendeleef's observation that most liquids expand according to the linear law $V = V_0(1 + kt)$, and his own observations that the coefficient of thermal expansion is the same for all fixed oils.

In a footnote he points out that the much simpler expression $(n - n_1)/(t_1 - t)$ (i.e., the change of refractive index per degree, or dn/dt) is not a constant; in fact, however, it compared quite favourably with his own for constancy. Thus, for the 20 oils quoted from Procter's work, the simple constants dn/dt are as follows:—347, 369, 340, 365, 351, 362, 356, 389, 349, 367, 362, 376, 364, 371, 356, 369, 376, 356, 380, and 371, all $\times 10^{-6}$. The mean value of the above figures is 0.000363 and the average deviation from the mean 0.000010, or 2.8%. The mean value for Wright's coefficients is 0.000751 and the average deviation from the mean 0.000023, or 3.1%.

The more conventional "coefficients of variation" $\frac{100}{M} \sqrt{\frac{\sum (x^2)}{n}}$

where M is the arithmetic mean of n results and x the difference of each from it, are 3.4 and 3.3 respectively.

It was, indeed, hardly necessary to put forward a new method of applying a temperature correction to refractive indices for practical purposes, as Tolman and Munson (*J. Amer. Chem. Soc.*, 1902, 754) had already demonstrated what is illustrated by the above figures, i.e., that dn/dt for different oils is very nearly a constant, the value of which they placed at 0.000365.

It appears to the present writer that Wright's method of correction does not introduce either increased simplicity or accuracy in practical work; it is, moreover, actually based on a hardly justifiable assumption. His formula, as stated above, assumes that the coefficients of thermal expansion of oils, and of fats and waxes, are approximately the same. But from a study of the figures given in a previous paper (this J., 1907, 514) it appears that these are in no closer agreement than the values dn/dt . Thus, for twenty of the substances dealt with in the above paper (neglecting those such as stearic acid and paraffin wax, which are not true oils and fats, and which give abnormal results) the mean value for the coefficient of thermal expansion is 0.000699 and the average deviation from it is 0.0000233, or 3.3%.

It is therefore suggested that correction of refractive index for temperature should be made by the simple method of adding or subtracting 0.00036 for every degree of temperature correction. Thus, in the example given by Wright, the refractive index of rape oil at 28.8° being 1.4696, the correction for 40° is $11.2 \times 0.00036 = 0.0040$, giving 1.4656 for the desired figure, as against 1.4655 using Wright's factor.

Wellcome Tropical Research Laboratories,
Khartoum, January 22, 1920.

Glasgow Section.

Meeting held at Glasgow on January 27, 1920.

MR. QUINTIN MOORE IN THE CHAIR.

WETTING POWER AND ITS RELATION TO INDUSTRY.

BY W. H. NUTTALL, F.I.C.

When a drop of liquid is placed on a level solid surface there are three possibilities; either the drop will retain its globular form, as, for example, mercury on glass, in which case there is little or no wetting of the solid by the liquid; or the liquid will distribute itself evenly over the surface to form a continuous film, as, for example, benzene on glass, which is an instance of complete wetting; or, lastly, the drop of liquid will flow to some extent and form a definite angle of contact between the surface of the solid and the air surface of the drop. Such a condition is to be regarded as partial wetting, and is exemplified by the behaviour of a drop of water on a slightly dirty glass or metal surface. The smaller the angle of contact which a liquid forms with a solid surface, the higher is its wetting power, and with complete wetting, the angle of contact becomes zero.

The ability of a liquid to wet a solid is a property which is made use of in many technical processes of great diversity. A method of comparing the wetting power of different liquids is a frequent necessity in such processes, yet the methods adopted show that the principles upon which wetting depends are not generally understood. Whilst the process of wetting is possibly a highly complex one and not yet completely understood, yet there are certain fairly well defined factors upon which wetting mainly depends, attention to which is usually sufficient to overcome most of the difficulties encountered in technical processes in which wetting in its broadest sense plays a part.

Quincke,* in 1877, was the first to point out the more obvious of these factors, and I will briefly reiterate them.

The spherical form assumed by a drop of falling rain is the result of the pressure exerted by the surface film, usually known as surface tension. A drop of oil suspended in dilute alcohol of the same density likewise assumes a spherical form as a result of the tensions exerted at the interface of the oil and dilute alcohol. This tension is usually known as interfacial tension, and will exist at the interface of any two immiscible liquids. Now consider the surface of a solid. The conception of a stretched film exerting a distinct tension at the surface of a solid is not so easy. Yet such undoubtedly exists, although its presence cannot be readily demonstrated. Further, in the case of a liquid in contact with a solid, judging from analogy, we should expect an interfacial tension to exist between the liquid and the solid.

Let us imagine that a drop of liquid has been placed upon the surface of a solid. Its behaviour will be mainly determined by the relative values of three distinct tensions. (If the drop is sufficiently small, the effect of gravity need not be considered.)

There is the surface tension of the liquid (T_1); the surface tension of the solid (T_2); and the interfacial tension of the liquid to the solid ($T_{1,2}$). The surface tension of the liquid (T_1) will be

exerted in the direction of the arrows at A (fig. 1), and will tend to maintain the drop in its spherical form. Similarly, the interfacial tension ($T_{1,2}$) will be exerted in the direction of the arrows at B, and this tension will also tend to roll up the drop

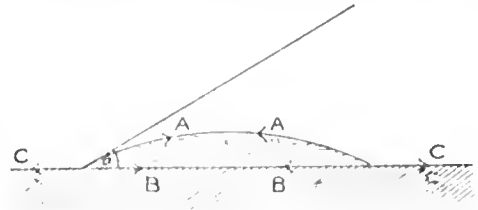


FIG. 1.

and maintain its spherical form. The surface tension (T_1), however, will have the opposite effect; it will be exerted in the direction of the arrows at C, and will strive to draw out the drop into a more and more lenticular form; the angle of contact of the drop will tend to become smaller and smaller until finally the latter forms a continuous film over the surface of the solid. For this to happen it is not difficult to see that the surface tension of the solid T_2 must exceed the sum of the surface tension of the liquid T_1 and of the interfacial tension $T_{1,2}$ —

$$i.e., T_2 > T_1 + T_{1,2}$$

Since two of these tensions, viz., the surface tension of the solid T_2 and the interfacial tension between the solid and the liquid $T_{1,2}$ are indeterminate, direct experimental verification of this inequality is not possible. Röntgen*, however, has succeeded by indirect means in establishing its validity for the system rubber/water. Certain indirect determinations which I have made are also in agreement with the inequality.

By a method to which I shall refer later, it is a comparatively simple matter to determine the interfacial tension of a thick oil to a soap solution. Both the surface tension of the oil and of the soap solution can be found by any of the usual methods. If a microscope slide is smeared lightly and evenly with the thick oil, the surface of the oil approximates sufficiently closely for the purpose of the experiment to a solid surface, the surface tension T_2 of which, as well as its interfacial tension to soap solution $T_{1,2}$, are known. In my experiments I used castor oil and two soap solutions of different strengths. The results obtained are tabulated below, the figures being given in dynes per cm.:—

Soap solution.	Surface tension of soap solution. T_1 .	Surface tension of oil. T_2 .	Interfacial tension of oil and soap solution. $T_{1,2}$.	($T_2 - T_1 - T_{1,2}$) Wetting power.
A.	34.6	38.1	0.4	+ 3.1
B.	40.8	38.1	10.4	-13.1

If the relation between the three tensions, stated above, is valid, since in the case of solution A the expression $T_2 - (T_1 + T_{1,2})$ is positive, we should expect that this solution would wet and form an even film over a slide prepared with castor oil; also, since with solution B the same expression is negative, we should not expect wetting. This was actually found to be the case. The greased slide was completely covered and wetted when solution A was poured over it; whilst a similarly prepared slide, on being treated in the same manner with solution B, was found to assert its greasy nature.

* "Ueber den Randwinkel und die Ausbreitung von Flüssigkeiten auf festen Körpern," Wied. Annalen, 1877, II., 145—194.

* "Mittheilung Einzelner Versuche aus dem Gebiet der Capillarität," Wied. Annalen, 1878, II., 321-28.

the soap solution running into drops and failing to wet.

From the above inequality it is evident that for a liquid to possess a high wetting power it must have a low surface tension (T_1) and also a low interfacial tension ($T_{1,2}$). Further, it is evident from fig. 1 that the effect of reducing the interfacial tension ($T_{1,2}$) will be to cause the liquid to spread over the surface of the solid, even if the surface tension T_1 is fairly high or, in other words, it is the interfacial tension $T_{1,2}$ rather than the surface tension of the liquid T_1 , which is the main determining factor in wetting. The figures in the above table lend support to this conclusion. In fact, in complete wetting, the interfacial tension $T_{1,2}$ is reduced practically to zero.

The process of wetting, however, is not so simple as would appear from a consideration of the above inequality or of its experimental verification which, after all, is purely of theoretical interest. In practice account must be taken of at least two other factors, which, under certain conditions, may cause the above relationship to fail entirely.

The first of these disturbing factors is concerned with the solvent properties of the wetting liquid. It usually happens in actual practice that the surface to be wetted is already coated with a waxy or greasy film. If the surface of the solid is to be reached, the wetting liquid must have a certain solvent action on this wax or grease. If the interfacial tension of the liquid is low, this solvent action need not be great, as, if the surface of the solid is reached in any one place, the waxy or greasy layer will be displaced by the wetting liquid, because its interfacial tension to the solid will be lower than that of the wax or grease to the solid. The detergent property of soap is due to this action. If, on the other hand, the wetting liquid has no solvent action on the wax or grease there is no actual contact between the liquid and the solid, and therefore no wetting. Thus, for example, it is probable that the reason why mercury fails to wet the surface of so many solids is not entirely due to its high surface tension, but also to the fact that it is incapable of dissolving the surface layer of moisture which adheres to most solids.

The second factor, liable to vitiate conclusions drawn from the expression conditioning wetting, is concerned with the phenomenon known as surface concentration or capillary adsorption. If, with increasing concentration, the surface tension of an aqueous solution of a substance decreases it will be found that the solute tends to aggregate in the surface layers of the solution.* Saponin, proteins, and various other substances possess this property to a marked degree. Aqueous solutions of dyes, kept in stoppered bottles so as to preclude evaporation, frequently show a visible accumulation of the dye at the surface. Lewis† has calculated that with a 0.25% solution of sodium glycocholate there is an extremely thin surface layer, the degree of concentration of which is about 160 times that of the bulk. The result of this surface concentration is the peculiar superficial viscosity or rigidity, first observed by Plateau‡. In the case of saponin this surface viscosity or rigidity may be easily demonstrated. If a bubble is blown with a 1% solution of saponin, the bubble film will be found to be imperfectly elastic and quite different from a soap film. If air is withdrawn whilst the orifice of the pipe is held in a vertical plane, so as to disturb the symmetry of the bubble, the shrinking film assumes a crinkled appearance, until finally there

results a vertical semi-solid cylinder with almost opaque walls.

Vermorel and Dantony* were the first to point out that a liquid exhibiting surface viscosity to a marked degree had in consequence a high wetting power. Solutions of saponin have marked wetting powers, although the surface tension of such solutions is comparatively high. Thus a 1% solution of saponin is capable of wetting a glass plate coated with paraffin wax, although a 5% solution of soap fails to do so. The wetting power of solutions of saponin, gelatin, etc., seems to depend largely upon their capacity to form liquid planes, the high superficial viscosity of which prevents rupture and running together to form drops.

The phenomenon of surface concentration is, however, of interest from another point of view. It has long been observed that the apparent surface tensions (static) of soap solutions are practically identical for all concentrations between 10% and 0.1%.† More recent investigations have, however, shown that this statement is only true if the surface tension determination has been carried out on an old surface. Rayleigh‡ succeeded in showing that, if the surface tension of a 0.25% solution of sodium oleate is measured within 1/400 of a second after the formation of the surface, the surface tension (dynamic) approximates closely to that of water. This initial tension, however, quickly falls until the ordinary value for the surface tension is reached. Rayleigh's results have been repeatedly confirmed by other workers, and a distinction is now drawn between what are known as the *static* and *dynamic* surface tensions. The static surface tension is the one obtained, when the determination is carried out on an old surface, as with the capillary rise and stalagmometer methods. The term "surface tension" as generally used in text-books refers to static surface tension. The dynamic surface tension, on the other hand, is the value obtained with determinations on perfectly fresh surfaces, as in the methods involving the use of jets, surface ripples, etc. The dynamic surface tension is always ill-defined, its value is dependent upon the age of the surface, and gradually passes to that of the static tension.

This strange behaviour of soap and other solutions is to be traced to the phenomenon of surface concentration or adsorption. Soap is a substance which is positively adsorbed, that is, it accumulates in the surface layers of a solution, and as surface tension is a function of the surface layers only, the presence of this concentrated surface pellicle of soap solution will be evidenced by a low surface tension. The peculiar behaviour of a soap solution referred to above would therefore find a ready explanation in the production of this surface pellicle of soap, the concentration of which is but little affected by that of the bulk of the solution. The formation of this surface pellicle is not instantaneous, but requires a certain lapse of time; hence the existence of the dynamic and static surface tensions and their gradual passage one into the other.

Having reached this stage we are now in a position to consider some of the more common methods which have been used in comparing the wetting power of different solutions. The method most frequently adopted is a determination of the surface tension of the solution. Can the surface tension of a solution afford a reliable indication of its wetting power? I think not, and for two reasons. Firstly, we have seen that the conditions for a

* Milner, "On Surface Concentration and the Formation of Liquid Films," *Phil. Mag.*, 1907 (6) 15, 96-110.

† An Experimental Verification of Gibbs' Theory of Surface Concentration, regarded as a basis of Adsorption, with an application to the Theory of Dyeing, *Phil. Mag.*, 1908 (6), 15, 489-526.

‡ "Statique des Liquides," 1873, II., Paris.

* "Tension superficielle et pouvoir mouillant des isocétides et fongicides. Moyen de rendre mouillantes toutes les bouillies euprique pour insecticides," *Comptes rend.*, 1912, 144, 1800-2.

† Maragoni, "Ueber die Ausbreitung der Tropfen einer Flüssigkeit auf die Oberfläche einer anderen," *Pogg. Annalen*, 1871, 143, 337-54.

‡ "On the Tensions of Recently Formed Liquid Surfaces," *Proc. Roy. Soc.*, 1890, 47, 281-87.

liquid to wet a solid surface are expressed in the inequality

$$T_2 > T_1 + T_{1,2}$$

From what has already been said, it is clear that of those three tensions it is the interfacial tension which is the predominating factor in determining wetting. Obviously some regard must be given to the nature of the surface to be wetted, and of the three tensions concerned, the surface tension of the liquid is the only one which has no connection with the surface of the solid. It is thus certainly the least desirable of the three upon which to base a judgment of wetting power. The frequently recurring statement that liquids with a low surface tension wet more readily than those with a higher one is not strictly true, for it does sometimes happen that a liquid with a high surface tension and low interfacial tension wets better than one with a low surface tension and a high interfacial tension.

Secondly, the surface tension of a soap solution cannot be accepted as a criterion of wetting power, because, as we have seen, the determination of the surface tension (static) is seriously vitiated by surface concentration effects. Solutions containing respectively 10%, 1%, and 0.1% of oleic soap all possess much the same surface tension (static), but their wetting power towards, say the leaf of a tree, is quite distinct.

Wetting power of sodium oleate solution towards liquid vaseline (at 20° C.).

Concentration of fatty acid.	No. of drops.	Surface tension of soap solution. T_1 .	Surface tension of oil. T_2 .	Interfacial tension of oil and soap solution. $T_{1,2}$.	$T_1 - (T_2 + T_{1,2})$ Wetting power.
		dynes per cm.	dynes per cm.	dynes per cm.	dynes per cm.
2.0	—	31.00	31.11	1.2	— 1.2
1.0	1300	29.35	31.11	5.87	— 4.11
0.5	1134	28.61	31.11	6.73	— 4.23
0.25	730	28.28	31.11	10.46	— 7.63
0.10	453	28.73	31.11	16.85	— 14.52
0.05	274	31.34	31.11	27.86	— 23.09
0.01	141	33.75	31.11	54.14	— 56.78
0.001	119	55.00	31.11	61.14	— 85.03
0.0001	117	70.99	31.11	65.24	— 105.12

Reference to the above table, in which figures are given for the wetting power of various dilutions of sodium oleate, will show that up to 2% of fatty acid there is a progressive increase of wetting power with the concentration. Further, whilst, except in the case of extreme dilution, there is but little difference in the values of the surface tension, the values for the interfacial tension go more or less hand in hand with those of the wetting power. If, therefore, it is convenient to estimate only one of the three tensions concerned in wetting, the interfacial tension is certainly preferable to the surface tension.

Another method of comparing the wetting power of solutions has been based upon a comparison of the amount of liquid which adheres to a solid object of standard size and shape, which is dipped into the liquid and then carefully withdrawn. But we have seen that the power to wet is dependent upon one or both of two main factors: (1) a low interfacial tension and (2) a high surface viscosity. One of the results of a low interfacial tension, where surface concentration effects are not particularly marked, is to cause the liquid to "run" easily. In the test under consideration the low interfacial tension of the liquid would cause it to wet the immersed solid, from which, on withdrawal from the fluid, all excess of liquid would "run" off very

rapidly leaving behind, however, a continuous film. The effect of a high surface viscosity would be in the opposite direction; as soon as a thin film of liquid was produced surface concentration would come into play, as a result of which draining would be largely hindered. The amount of liquid adhering to the solid would be the resultant of two opposite effects, and as such we should not expect it to differ in any marked degree, whatever the solution under investigation—a view which experiment confirms.

It is a matter of common experience that an emulsion of oil in any liquid always possesses good wetting properties, and one method of measuring wetting power is by a comparison of the emulsifying power of the liquids in question towards a standard oil. Theoretically the method is sound, and it may be of interest to point out why.

Donnan* has shown that a soap solution acts as an emulsifier by virtue of its low interfacial tension with respect to oil. The result of this low interfacial tension is two-fold. Firstly, the soap becomes concentrated at the surface of the oil globules, and owing to the surface viscosity produced thereby the film of liquid intervening between two oil globules resists thinning and the consequent coalescence of the globules. This explanation also probably holds for emulsifying agents other than soap. Certainly in milk, which is a good representative emulsion, the globules of fat are surrounded by a tenacious coating, which undoubtedly consists of adsorbed albuminous matter from the serum. It is so tenacious that it can partially protect the fat globules from the solvent action of ether, as it is impossible to extract the fat completely from milk by ether without drying the milk and so destroying the adsorbed film surrounding the globules. In fact, if the oil content in an emulsion is large, the surface rigidity or viscosity at the interface of each of the oil globules becomes visible in the thickening which results in the emulsion. The viscosity of a concentrated oil emulsion is considerably higher than that of the oil and of the emulsifying solution, and this increase in viscosity may be regarded as the result of the surface viscosity effects occurring at the interface of each oil globule and the aqueous matrix.

Something of the character of this adsorbed film which is formed at the surface of the oil globules may be gathered from the following simple experiment. If a fair-sized globule of thick mineral oil is placed on the surface of a somewhat concentrated solution of saponin, and by means of pipette saponin solution is introduced into the centre of the floating globule of oil, it is possible to produce a bubble of oil filled with saponin solution, the bubble itself floating within the external solution. The thin layer of oil forming the skin of the bubble, whilst of itself quite incapable of giving a tenacious film, has under the conditions of the experiment become so fortified, both internally and externally, by the saponin adsorbed on each interface that a comparatively rigid structure results. This bubble of oil is in fact a large scale representation of the condition of a minute globule of oil in an oil emulsion, except, of course, in the latter case, the globule of oil contains none of the emulsifying agent within itself.

The second reason why a liquid, possessing a low interfacial tension with respect to oil, should possess high emulsifying powers is a simple one. The force tending to break the aqueous film intervening between the globules of oil is that of the interfacial tension of the aqueous liquid to the oil, and if this is small, it is not sufficiently strong to withdraw the aqueous film separating the globules. Thus, the main requisite for a liquid to have a high

* "Ueber die Natur der Seifenemulsionen," Z. physikal. Chem., 1893, 31, 42-49.

emulsifying power to an oil is that it shall possess a low interfacial tension towards that oil. We have already seen that the main factor in determining the wetting power is a low interfacial tension towards the surface to be wetted, and, as in actual practice such surfaces are usually of a greasy character, it follows that a good emulsifier is also a good wetter, and that, therefore, emulsifying power is a very good indication of wetting power.

One other power of most wetting liquids has been suggested as of use in the comparison of wetting power; that is, foaming power. The property of foaming and the factors which lead to the formation of a permanent foam have formed the subject of a considerable amount of investigation by various workers, amongst whom may be mentioned: Plateau,* Quincke,† Rayleigh,‡ Donnan,§ Shorter,¶

A foam is now generally regarded as an emulsion of air in a liquid. Support is lent to this view by the fact that the requisite conditions for the production of a permanent foam are exactly analogous to those necessary for the formation of a permanent emulsion of oil in an aqueous liquid. These we have seen to consist in (1) a high surface viscosity of the intervening film, and (2) an interfacial tension so low as to be incapable of breaking this film. In the case of an oil emulsion the intervening matrix prevents the coalescence of the oil globules, and similarly, in the case of foam, it is the intervening matrix which prevents the coalescence of the air bubbles. With emulsions of oil, therefore, it is the interfacial tension of the aqueous liquid to the oil which is the determining factor; with foams, on the other hand, it is the interfacial tension of the aqueous liquid to air, i.e., the surface tension of the aqueous liquid, which is of importance. The property of giving a lasting foam, therefore, indicates that the liquid possesses (1) the property of surface concentration, such as saponin solution, which shows exceptional frothing power, and (2) a low surface tension, such as soap solutions. Since, however, wetting power is mainly dependent on a low interfacial tension, the ability to give a permanent foam does not necessarily indicate a high wetting power. As a matter of fact, I have examined liquids which, whilst possessing an extremely low interfacial tension towards oil and thus a high wetting power, were quite incapable of giving a foam.

It will now be recognised that whether we are concerned with the comparison of emulsifying power or of wetting power, it is the interfacial tension which is the deciding factor, and that a simple method of determining the interfacial tension would afford us a quantitative test of considerable practical value. Unfortunately, as I have already pointed out, there is no simple and direct method by which the interfacial tension of a liquid to a solid may be measured, but in the case of two liquids, as an oil and a solution of soap, the determination of their interfacial tension offers no difficulty. And since in the majority of cases the surface which we are concerned in wetting is a greasy or waxy one, there seems to be no valid reason why, for the purpose of comparison of wetting power, a thick oil should not be taken to represent the solid surface. This is a method which I have followed for some years with highly satisfactory results.

The determination of the interfacial tension between an oil and a solution of soap is carried out

by means of Donnan's "drop pipette" (*loc. cit.*). The pipette is filled up to a mark on the upper limb with a selected standard oil, lowered into a glass jar containing a given volume of the aqueous liquid under examination, and clamped in an upright position. The upper stopcock is then opened, and the number of drops of oil counted until the level of the oil in the pipette sinks to a mark on the lower limb. The size of the drop and hence the drop number is dependent upon (1) the interfacial tension, (2) the size of the aperture of the pipette, (3) the difference in density between the oil and the aqueous liquid, and (4) a constant depending upon the size of the aperture, the difference in density, and the interfacial tension. If absolute values are required, all these four factors must be taken into account. The calculations are somewhat tedious, but have been described fully by Lewis (*loc. cit.*).

Where comparative values only are required, as in the determination of wetting and emulsifying powers, the interfacial tension may be taken as inversely proportional to the number of drops since the diameter of the aperture remains constant, whilst the difference in density between the oil and the fluid examined does not usually vary greatly. Since the wetting or emulsifying power varies inversely as the interfacial tension, the wetting or emulsifying power is directly proportional to the drop number (compare table p. 69 r.). I have described this method more fully elsewhere in collaboration with Cooper.* We realise that the method is open to certain objections, but we have found by experience that it affords a ready means of determining and expressing in numerical value the wetting and emulsifying power of a solution. The apparatus required for the test is simple, and if attention is given to a few simple precautions reliable results are easily obtained. The method, unfortunately, has its limitations. When employed with solutions of gelatin, saponin, etc. these should be extremely dilute; with stronger solutions of such substances surface concentration at the interface of the oil and aqueous solution becomes so marked that quasi-solid surfaces are produced, with the result that the drop number and hence the value found for the interfacial tension are completely vitiated.

In conclusion, a few of the many industries in which wetting power plays a more or less important part may be mentioned very briefly.

Perhaps the most obvious is the flotation process for the separation of minerals, an industry which has made enormous strides during recent years, enabling, as it does, low-grade tailings, hitherto wasted, now to be extracted profitably. The principle upon which the process is based is the fact that the same liquid wets a mineral sulphide and quartz to a different extent, and further, that this difference in "wettability" is accentuated by the addition of a small quantity of a suitable oil, which, by being selectively adsorbed on to the surface of the sulphide, renders it more difficult to wet. Similarly, by the addition of an acid or alkali, the surface of quartz is rendered more easily wettable. At the recent Scientific and Industrial Exhibition held in Glasgow a process was shown working in which fine particles of coal were separated from earthy matter through their difference in "wettability" by a common solution.

There are numerous other industries in which wetting power and emulsifying power play an important, though perhaps not such an obvious, part. Take the case of coal-tar disinfectants. Why should an emulsified disinfectant possess a higher disinfecting coefficient, as measured by the Rideal-Walker or similar test, than a clear soap solution containing the same content of tar acids? I do

* *loc. cit.*

† Ueber periodische Ausbreitung an Flüssigkeitsoberflächen und dadurch hervorgerufene Bewegungserscheinungen. Wied. Ann., 1888, 35, 580-642.

‡ "On Foam." Proc. Roy. Inst., 1890, 13, 85-97.

§ *loc. cit.*

¶ Note on the elasticity of Films and the Foaming of Solutions. Phil. Mag., 1912 (6), 27, 629-32; "Classification of Foaming Solutions. Phil. Mag., 1914 (6), 27, 718-720.

* "Wetting power and its Determination." J. Agric. Sci., 7, 219-239.

not suggest in this case that it is due to a higher wetting power, although a high wetting power in a disinfectant is very desirable so that it may thoroughly wet the surfaces which it is intended to disinfect, and, as we have seen, a good emulsion is a visible indication of high wetting power. I think that Chick and Martin's² explanation of the superiority of an emulsified disinfectant is correct, viz., that the bacteria are adsorbed on to the fine emulsified particles of the tar acid, and are thus brought into direct contact with the germicidal agent in a highly concentrated form. And, if this be the case, the larger the surface of tar acid exposed, the more highly efficient will be the disinfectant; or in other words, the globules of emulsified oil must be made as small as possible. The lower the interfacial tension between the solution of soap and the tar acids used, the finer will be the emulsion.

An almost identical problem is offered by the use of Twitchell's reagent for the saponification of fats. Twitchell's reagent owes its efficacy to its great emulsifying power, whereby the fat is broken up into innumerable fine globules, so that as large a surface as possible is exposed to hydrolysis. It might easily be determined if the reagent is up to standard and if it is the most suitable form for the particular kind of fat in use by a determination of the interfacial tension of solutions of the reagent and the fat, although I have never determined such myself.

Sometimes problems of the opposite character are encountered, where it is the object to avoid the formation of emulsions. The use of oil in turbines is a case in point. Some time ago it occurred to me that the drop pipette might be employed in this connection also, as an oil with as high an interfacial tension as possible would be the most suitable for turbine work. One of the large oil companies was good enough to supply me with a dozen specimens of oil, some of which were known to be unsuitable and some suitable for turbine work. I graded them according to their interfacial tension towards water, and in only two cases was I in error. I believe that if, instead of carrying out the determination of the interfacial tensions at ordinary temperatures, I had worked at one more nearly approaching working conditions these two discrepancies might have been avoided.

In every process in which any liquid is applied to a solid surface the question of wetting must play some part. Take the case of the application of contact poisons as a means of eradicating or at least controlling insect parasites. In various parts of the world ticks cause an enormous annual loss to cattle breeders. The usual means of dealing with ticks is by dipping the cattle in a dilute solution of sodium arsenite, but in the case of the "bont tick" it was found that the concentration necessary to kill the tick frequently led to the destruction of the cattle through bad scalding. By incorporating an oil emulsion with the dip, and thus ensuring a high wetting power, Cooper showed that it was possible materially to reduce the arsenic content of the dip and yet ensure the destruction of the tick without injury to the cattle. This application of wetting power alone has been instrumental in clearing large tracks of tick-infested country in various parts of the world.

Wetting power is of equal importance with all forms of tree and horticultural sprays as well as with insecticides. The obvious necessity of a contact poison is that it shall come into contact with that which it is intended to kill. And there is no real contact unless there is real wetting. The leaf of a tree is not an easy thing to wet; the chitinous integument of an insect is still more difficult; and the protective woolly secretion of the aphids of

American blight most difficult of all. Everyone knows that an emulsion of paraffin in soap makes a very fair tree spray, but perhaps few realise that the mixture owes its efficacy to the high wetting power conferred by the soap. Such an emulsion, however, usually fails to subdue American blight, either because its wetting power is not sufficiently high or because it lacks sufficient solvent action on the woolly secretions of the aphids. If such secretions are touched with a drop of alcohol, which possesses both wetting power and solvent action, the pest quickly succumbs.

For the valuation of soaps for technical purposes we usually rely upon a determination of the fatty acid content, and make no attempt to differentiate between different kinds of fatty acids, although experience often shows that certain fatty acids are more suitable for some purposes than for others. I would suggest that the drop pipette might also be used for determining the detergent action of different kinds of soap under the particular conditions in which they are used. We have already seen that the detergent action of soap is largely determined by its power of displacing grease and other matter from a surface, by virtue of the fact that the interfacial tension between the surface and the soap solution is less than the interfacial tension between the greasy matter and the surface. Thus, generally speaking, the detergent action of a soap is inversely proportional to its interfacial tension towards an oil.

In the varnish and paint industry interfacial tension also plays some part. When certain varnishes are applied to paper the interfacial tension of the thickening varnish towards the paper is so high that as the spirit evaporates the original film breaks and the varnish is drawn into drops. Much the same effect is produced by painting on a wet surface. Lack of attention to interfacial tension effects in the doping of aeroplane wings led to serious accidents in the early days of the industry. Acetylcellulose dopes are generally made up with a solvent which is not miscible with water. Cotton naturally contains over 7% of water, and its surface is undoubtedly coated with a fine film of moisture. Since an organic solvent would have little or no solvent action on this superficial layer of moisture, the dope would be unable to penetrate to the surface of the cotton itself; there would be no real wetting, no real contact, and so no good adhesion. It is not surprising therefore to find that dope applied under such conditions showed a great tendency to strip off in large sheets. Later, when care was taken to dry the cotton before applying the dope, there was perfect adhesion.

With the advent of the modern form of gas mask means had to be taken to prevent the deposition of moisture in globular form from the eyes and face on to the eye-pieces of the mask, which would impair vision. From what has been said it is obvious that all that is required is a substance which has a low interfacial tension towards glass or celluloid; the application of such would cause any moisture to be deposited in an even film, so that there would be little or no interference with vision. Much work was done on the subject by the Americans, and it is interesting to note that attempts were made to compare the efficacy of various "anti-dimming" compositions by determinations of their surface tension, with not always very gratifying results.*

Such are some of the more obvious applications of wetting power. Doubtless many more could be discovered, but perhaps these will suffice to show that the wetting power and interfacial tension are factors well worthy of the attention of the practical man, for they play no small part in many branches of industry, widely diverse in character.

* J. Hygiene, 8, 698-703.

* See J. Ind. Eng. Chem., 1919, 11, 1105, 1111.

Manchester Section.

Meeting held at Grand Hotel on December 6, 1919.

MR. JOHN ALLAN IN THE CHAIR.

A METHOD OF DETERMINING THE RELATIVE TEMPERATURES OF SPONTANEOUS IGNITION OF SOLID FUELS.

BY F. S. SINNATT, M.B.E., M.SC. (TECH.), F.I.C., AND
BURROWS MOORE, M.B.C. (TECH.) B.SC. (ENG.) A.I.C.

The investigation was designed for the purpose of obtaining information of the relative spontaneous inflammabilities of certain finely divided fuels, and to examine the influence of physical and chemical variations of the fuels upon the temperature of ignition.

The conditions under which the experiments were carried out were considered to approximate to the treatment received by fuels previous to combustion in a stream of air, when used for pulverised fuels. It seems possible, however, that the temperatures recorded may also be of interest in the study of those properties of coal which lead to ignition when brought into heaps, either in the mine, or during storage. With the exception of the method designed by Wheeler (Chem. Soc. Trans., 1918, 113, 945), no very clear indication can be obtained by means of tests of the liability of coals to undergo heating, either during storage or in the gowf. It will be seen that if pulverised fuels are to be widely used, it is desirable to have a rapid method available for measuring the relative ease with which the fuels will ignite. Upon this property the completeness of the combustion in a given space will depend largely, and also the degree to which the air can be pre-heated without the danger of pre-ignition occurring.

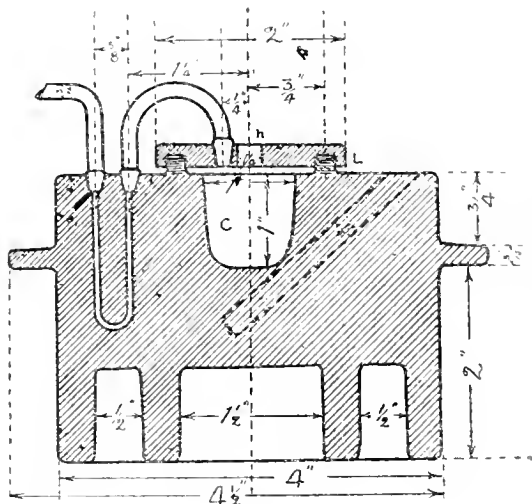


Fig. 1.

It appeared of interest to attempt to elaborate a simple means, necessarily empirical, by which some information upon this intricate subject might be gained, and, although the investigation is still

proceeding, we feel that the results obtained warrant our bringing them forward at the present stage.

It is not our intention to enter into a discussion of the literature of this subject; comparatively few experimenters deal directly with the temperature of spontaneous ignition of coal, and we are only able to find a few papers which actually deal with the determination, one of the most valuable being that of Wheeler. There is, however, a very wide literature dealing with the chemical aspect of the oxidation of coal.

The experiments to be described were planned (a) to determine the relative tendencies to spontaneous ignition in oxygen of certain finely powdered fuels; (b) to examine the influence of the degree of fineness of the particles on the temperature recorded; (c) to examine broadly the influence of the volatile matter upon the temperature recorded. For the purposes of this paper, the temperature of spontaneous ignition in oxygen is defined as that temperature to which a fuel must be raised for ignition to take place without the aid of external agents of inflammation.

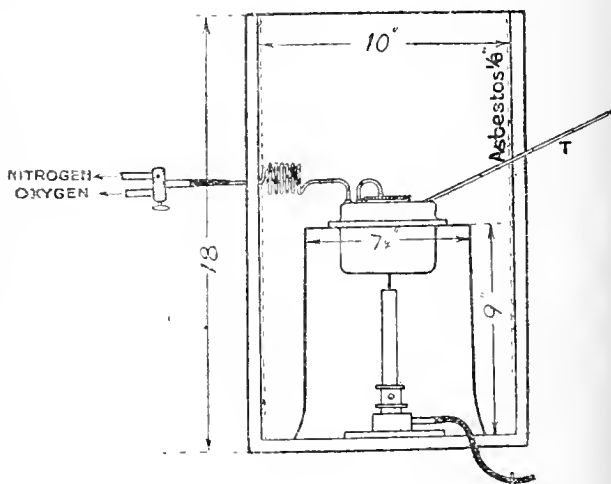


Fig. 2.

The apparatus used for the experiments was similar to the ignition meter designed by Harold Moore, and successfully used by him to determine the relative ignition temperatures of liquid fuels (this J., 1917, 109).

The apparatus consists of a heavy cast iron vessel, in which an enclosed nickel crucible may be raised to any desired temperature. It is shown in section in Fig. 1. Fig. 2 shows the arrangement used by us, in which the apparatus is contained in an asbestos-lined box to eliminate the influence of draughts. The oxygen or other gas, before entering the nickel combustion chamber, traverses the U-shaped tube, bored into the casting. The length of this pre-heating tube is sufficient to raise the temperature of the oxygen to that of the casting, before it comes into the combustion chamber. A thermometer is placed in a cylindrical chamber, so arranged that the mercury is practically directly below the combustion chamber. The casting is deeply grooved at the bottom, in order to increase the surface exposed to the flame of a large bunsen burner, which is used as the source of heat. The oxygen was dried by passing it through concentra-

ted sulphuric acid. In certain experiments nitrogen was needed to sweep out the crucible, and a three-way tap was attached to the ignition meter, so that nitrogen or oxygen could be passed into the combustion chamber as required. Throughout the experiments, the oxygen was supplied to the instrument at the rate of three bubbles per second, as measured over a period of 60 seconds.

Preliminary experiments indicated that approximate constancy in the weight of the charge of fuel was necessary in order to secure reliable comparisons, and it was therefore decided to introduce the fuel into the combustion chamber from a "dropper" made of glass tubing, 8 in. long and $\frac{1}{4}$ in. internal diameter, with one end bent at right angles. The most suitable shape of cup was found by experiment to have a diameter about $\frac{1}{4}$ in., and depth about $\frac{1}{2}$ in. The cup was filled by dipping it into pulverised fuel, the sharp edge of a spatula was then drawn across the ground edges, and any loose particles adhering to the outside removed. With solid fuels, sifted through a certain size of mesh under similar conditions, the volume contained in the cup is approximately the same. Each of the samples of dust examined was examined microscopically, and, with the exception of the Irish peat and the Fusain, particles which have passed through the same size of sieve were found to have approximately the same size.

The weight of fuel contained in the cup was ascertained for two widely different types of coal, namely, Wigan Arley coal and Hoo canal coal. With Arley coal (graded to pass through a 1/200 sieve), the average weight of coal contained in the cup of the dropper for 14 determinations was 0.0052 grm., the greatest weight recorded was 0.0054 grm., and the lowest 0.0050 grm. With Hoo canal coal, similarly graded, the corresponding figures were 0.0057, 0.0061, and 0.0055 grm. respectively. It will be observed that the weight contained in the cup is fairly constant, although it cannot be considered free from error. An actual experiment was carried out in the following manner: The temperature of the ignition meter was raised until the reading of the thermometer was constant and the flow of oxygen exactly three bubbles per second. A charge of coal from the dropper was then introduced into the crucible, and a time record was kept of the following phenomena: (1) Commencement of visible active combustion; the mass glows. (2) Ignition, as manifested by an explosion or production of flame. (3) Extinction of visible combustion.

The especial points noted were the time interval before visible combustion or glowing occurs and the time interval before explosive or inflammatory ignition occurs. The progress of the combustion was watched through the central hole in the lid, and the residue, after visible combustion had ceased, was examined preparatory to the introduction of a fresh charge.

When a determination was complete the delivery tube for oxygen was detached, the lid covering the combustion chamber unscrewed, and the nickel crucible taken out, and its contents removed. Thus, for each determination the rate of supply of oxygen and the condition of the combustion chamber were appreciably the same.

The coals examined were: Wigan Arley coal, Wigan Yard, anthracite, Hoo canal (26% ash), and two coals liable to produce gob fires (Nos. 1 and 2).

The coal was pulverised so as to pass through a 1/200 mesh sieve and was air-dried; the samples, with the exception of Hoo canal, were fairly freshly mined, *e.g.* not mined longer than three weeks. The results are shown in the following tables:—

Wigan Arley coal.

Time in seconds.			Temp. ° C.	Remarks.
Extinction.	Glow.	Ignition.		
90	70	—	184	
100	72	—	187	
200	80	150	188	Yellow flame.
150	60	100	190	
80	30	40	210	Yellow flame.
70	20	30	220	Fairly powerful explosion
65	15	25	210	Yellow-white flame.
60	13	23	219	
60	12	16	259	Fairly powerful explosion
65	10	12	272	Yellow-white flame, fairly powerful explosion.
50	10	14	279	
54	10	16	280	
50	10	16	296	
66	8	38	309	Feeble explosion.
65	4	35	313	Feeble ignition.
39	3	11	320	Powerful explosion.
36	2	2	335	Feeble explosion (blue-yellow-flame).
27	2	4	345	
40	2	2/14	355	
42	1	13	366	Feeble explosion, blue flame at 11.
40	1	1/11	375	Blue flame at 11, yellow-white flame at 1.
48	1	1/18	396	
42	1	1	396	

Wigan yard coal.

Time in seconds.			Temp. ° C.	Remarks.
Extinction.	Glow.	Ignition.		
50	17	35	219	
48	13	30	233	Yellow flame.
45	11	27	246	Yellow flame.
42	8	28	269	Yellow-white flame.
50	7	7/9	271	Yellow-white flame.
46	4	23	280	
55	4	26	290	
45	3	28	300	Very powerful explosion.
50	3	18/21	310	Very powerful explosion.
49	3	3	328	Blue flame at 21.
30	2	2/25	342	Yellow-white flame at 2.
42	2	36	364	Blue-white flame at 25.
35	1	20	372	
31	2	18	380	Yellow-white blue flame.
45	2	2	392	
45	1	1	396	

Anthracite.

Time in seconds.			Temp. ° C.	Remarks.
Extinction.	Glow.	Ignition.		
—	—	—	246	
—	—	—	248	
113	45	—	250	
125	32	32	256	
140	31	31	258	Yellow flame
136	29	29	267	
120	23	23	278	
130	17	17	287	
90	19	—	296	
105	17	20	300	
110	15	45/50	310	
100	12	41	321	Yellow-white flame.
110	12	14	332	
103	9	12	342	
90	6	6	350	
100	6	28/35	362	
102	6	—	368	
120	7	50/58/70	384	Yellow-white flame at 50.
83	4	38	388	Blue flame at 70.
106	5	52	390	Yellow white blue flame.
121	4	35	391	
111	6	17	400	Very powerful explosion.
90	5	5	412	

Hoo canal.

Time in seconds.			Temp. ° C.	Remarks.
Exinction.	Glow.	Ignition.		
77	33	—	225	
90	31	—	230	
64	22	59	244	Yellow flame.
45	19	—	253	
67	19	50/60	253	Yellow flame at 50. Blue flame at 60.
60	18	45	267	Yellow flame.
48	16	—	274	
35	15	35	277	Yellow flame.
44	13	—	290	No ignition.
40	10	—	300	No ignition.
35	10	—	310	No ignition.
58	12	—	320	No ignition.
53	10	—	334	No ignition.
63	10	—	343	No ignition.
60	7	38	350	Yellow-white flame.
97	6	—	360	
45	3	3	374	
45	5	—	382	
57	2	2	391	
63	2	2	398	
40	1	20/30	400	Blue-yellow flame at 20.
46	2	20/30	402	Blue flame at 30.
55	3	3/25	404	Blue-violet flame at 3. Blue-white flame at 25.

Coal liable to produce gob fires, No. 1.

Time in seconds.			Temp. ° C.	Remarks.
Exinction.	Glow.	Ignition.		
52	29	35	228	
55	20	35	230	
45	13	15	243	Very powerful explosion.
44	13	14	250	Powerful explosion, yellow flame.
40	11	14	267	Very powerful explosion, yellow flame.
40	10	14	278	
38	10	14	290	
39	10	14	300	Very powerful explosion, yellow-white flame.
38	9	13	310	
28	8	14	320	Powerful explosion, yellow-white flame.
25	6	9	330	
37	4	10	340	
35	3	3/14	350	Very powerful explosion at 14.
36	5	10	362	Very powerful explosion, yellow blue white flame.
30	2	2	378	
40	1	1	386	

Coal liable to produce gob fires, No. 2.

Time in seconds.			Temp. ° C.	Remarks.
Exinction.	Glow.	Ignition.		
55	25	30	223	Feeble explosion, yellow flame.
40	17	17	238	Feeble explosion, yellow flame.
30	12	25	256	
30	10	13	265	Powerful explosion, yellow-white flame.
40	10	14	290	
39	11	13	308	
40	5	11	318	Powerful explosion, yellow-white flame.
35	4	13	329	
35	2	2	338	Feeble explosion, yellow-white flame.
35	2	2	345	
36	3	3	351	
36	3	12	360	Powerful explosion, yellow-white flame.
33	12	2	372	
35	12	2/10	379	Fairly powerful explosion
35	12	10	389	Feeble explosion, blue white yellow flame.
37	12	2	391	
39	3	3	394	
40	12	2/15	397	Feeble explosion.
35	3	11	400	
29	12	2	404	
45	1	1/15	408	Feeble explosion at 1, powerful explosion at 15, blue-white flame.

The above values are plotted in the graphs, figs. 3—8.

The following is a brief analysis of the above results:—

In all the cases, except at the higher temperatures, glowing in the mass of coal commences before ignition takes place, and the time interval before the former occurs decreases with rise in temperature. The variation of the "time intervals" before ignition takes place, compared with the intervals for glowing to appear, are somewhat irregular. There is a well-defined temperature below which no glowing occurs within a time limit

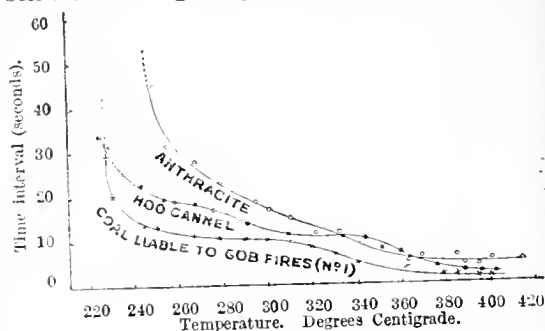


Fig. 3.—Glowing.

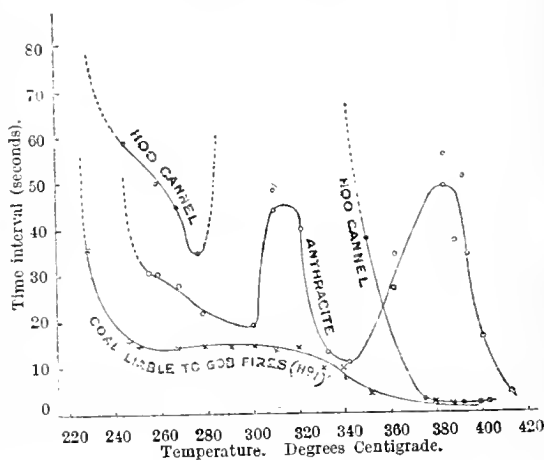


Fig. 4.—Ignition.

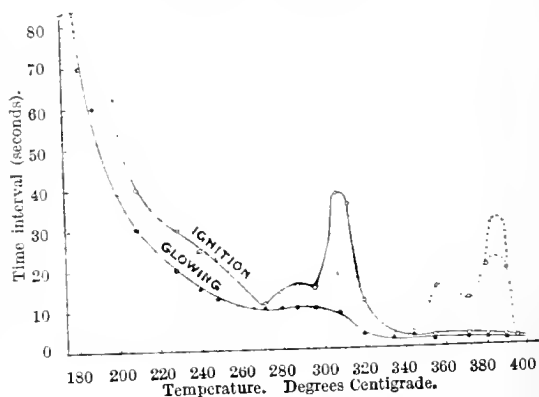


Fig. 5.—Arley.

of four minutes, and, with the exception of Hoo canal, this also applies to the ignition. In the case of Hoo canal (which contains 26% of ash) an intermediate range of temperature exists, at which no active ignition occurs.

In the coals examined, with the exception of the two which are known to be liable to gob fires, there appear to be zones of temperature at which spontaneous ignition occurs after a much longer interval, and it would appear that coal yields volatile compounds at these ranges of temperature, which compounds have a higher temperature of ignition (compare figs. 3 and 4). It is suggested that such coals may possess the property at these temperatures of tending to extinguish any heating which may be taking place, especially where the coal is in masses, and the oxygen only in contact with the surface. Where, however, coal does not evolve such compounds, no such self-damping action is possible, and the result of the oxidation would be an additive one consisting of glowing of the solid substances, and ignition of the volatile constituents. This feature of the work requires more exhaustive examination.

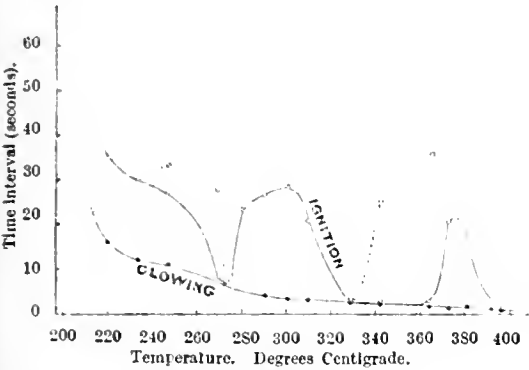


Fig. 6.—Yard coal.

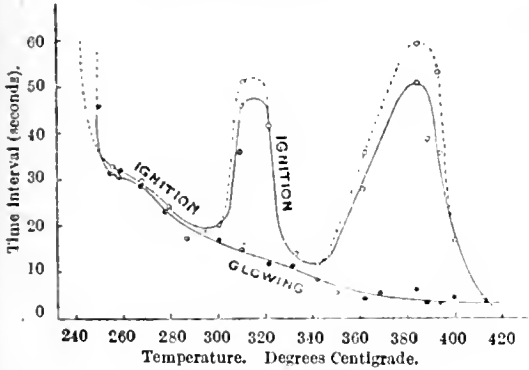


Fig. 7.—Anthracite.

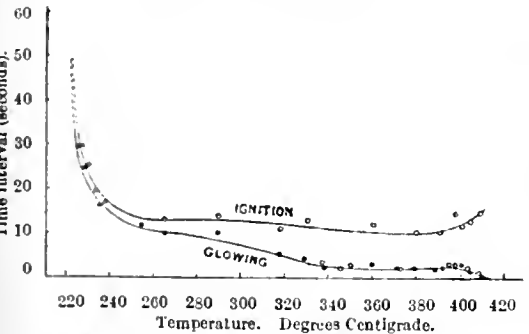


Fig. 8.—Coal liable to gob fires, No. 2.

It will be noticed that in the case of the two coals liable to gob fires, the curves of spontaneous glowing and ignition are practically parallel (compare figs. 3, 4, and 8), but there were very clear

evidences of the spontaneous ignition occurring practically at the same interval at which the glow was visible; in other words, the volatile compounds produced from these coals were more easily inflammable than was the case with the other coals examined.

In order to obtain an idea of the minimum temperature at which substances would either glow or ignite, a time limit of four minutes was adopted, and the lowest temperature at which substances would glow or ignite within this period was taken as the temperature of glowing or ignition. The time interval was decided upon as being sufficiently long for most practical purposes.

The values obtained for a number of substances are given below:—

Substance.	Minimum temperatures in oxygen, °C.			
	Glowing.	Ignition.	Holm's figures.	Brame's figures.
Anthracite coal ..	250	258	440	500
Hoo canal coal ..	225	230		
Wigan Arley coal ..	184	188		
Wigan yard coal ..	219	219		370
Indian coal ..	242	242		to 425
Coal liable to gob fires, No. 1 ..	228	228		
Coal liable to gob fires, No. 2 ..	228	228		
Gas coke ..	Above 398	Above 398		
Coalite ..	Above 396	Above 396		
Wood charcoal ..	248	248		
Irish peat ..	300	300		
Cellulose ..	324	324	360	
Fusain ..	367	367		
Paraffin wax ..	246	246	310	

Influence of the degree of fineness of the particles of the coal upon the minimum temperature at which spontaneous glowing occurs.

Early in the experiments it was found that the degree of fineness of the fuel had a considerable influence upon the minimum temperature at which spontaneous glowing would occur, and preliminary observations have been made to ascertain broadly the influence of the degree of fineness of the fuels examined. The results are recognised as being quite preliminary, but as they show that the degree of fineness is a most potent factor, it is desirable to place them on record at once.

Minimum temperature at which spontaneous glowing occurs.

Description.	Degree of Fineness.		
	1/200	1, 100 to 1/200	Mixture of 2 parts 1/200 & 1 part 1/100—1/200
	Temp. °C.	Temp. °C.	Temp. °C.
Indian coal ..	240	Above 380	242
Hoo canal ..	229	Above 390	277
Yard coal ..	219	Above 396	253
Coal liable to gob fires, No. 1	232	346	—
Do, do, No. 2	225	298	—

It will be observed that coal pulverised to pass through a 1/100 to 1/200 mesh sieve did not commence to glow at a temperature below about 400° C. within four minutes, whereas, when pulverised to pass through a 1/200 mesh sieve, glowing commenced at a much lower temperature. Further, that mixtures of fine and coarse material appear to commence to glow at a temperature approximating more closely to the glowing temperature of fine material than to that of the coarse material.

The influence of the volatile matter present in the fuel upon the temperature at which spontaneous ignition occurs.

The results recorded up to the present indicate that the inflammatory or explosive ignitions which coincide with, or follow, the initial glowing of the fuel, are associated to a degree with the volatile matter present in the fuel. It was therefore thought desirable to place on record some experiments we have commenced upon the influence of the volatile matter upon the liability of the fuel to glow or ignite spontaneously. It was thought that if a charge of fuel could be maintained at a fixed temperature in the crucible, under conditions which would simultaneously preclude the oxidation and assist the expulsion of the volatile matter, and the residual material at once examined to determine the temperature of spontaneous inflammation and glowing, that the resulting observations might be compared with those obtained by a blank experiment, using oxygen alone at the same temperature. The apparatus was therefore arranged so that nitrogen could be passed into the combustion crucible, and by merely reversing the three-way tap a supply of oxygen could be at once introduced. The nitrogen used was freed from oxygen by passing it through two bottles containing alkaline pyrogallie acid, and the rate of flow was six bubbles per second. The nitrogen was passed in at this rate in order to have a distinct sweeping action and yet not disturb the charge in the crucible. The ignition meter was raised to any required temperature, the nitrogen turned on, and a charge of coal introduced from the dropper. The coal was kept for 15 minutes in the supply of nitrogen. At the end of the period determined upon the supply of nitrogen was interrupted and oxygen introduced into the crucible at three bubbles per second. The time interval before glowing or ignition occurred was then determined. The crucible was at once emptied from any residue, and a fresh charge or blank was dropped into it, having the oxygen continually flowing. The process was repeated for different temperatures, and the results are given in the following table:—

the fuels (except Hoo canal) which had been previously exposed to nitrogen failed subsequently to glow or ignite in oxygen within the time interval of four minutes, whereas the control samples examined immediately afterwards behaved exactly as we have previously recorded. In the case of Hoo canal, glowing and sometimes ignition was observed for the charges exposed to nitrogen at temperatures of 330° to 380° C., i.e., considerably above the minimum temperatures previously recorded. In all cases the actual ignition which occurred was of a less inflammatory or explosive character when the coals had been heated in nitrogen.

The conclusion to be deduced from these observations is that the volatile matter of the coal has a considerable influence upon the liability of the fuel to glow. The non-appearance of glowing in the case of the nitrogen-treated charges appears especially important where the material has only been heated to temperatures not appreciably above the minimum temperatures previously recorded for glowing to occur, and it would therefore appear that the oxidation of the volatile constituents evolved contributes very largely to the heating effect, which leads to the mass of coal becoming heated, and consequently to the production of a glow.

We wish to suggest that the minimum temperature for glowing to occur in coals may be of use in comparing the relative ease with which coal will undergo oxidation. It is our intention to examine coals which are liable to undergo ignition during storage, and others which produce gob fires. It seems possible that the curves indicating the time interval at which coals will glow at different temperatures may be a means for characterising coals. Further, the experiments have afforded direct evidence that fineness of the material has an influence upon the tendency to spontaneous ignition and glowing, and that if the particles are present in sufficient proportions the temperature at which the mass will glow is determined largely by the proportion of the fine material present. The

Air-dried sample.	Exposed to nitrogen for 15 mins. before admission of oxygen.			Temp., °C.	No previous exposure to nitrogen before admission of oxygen.	
	No. of seconds before glowing.	Remarks F = flame. E = explosion.			No. of seconds before glowing.	Remarks.
Arley coal 200 mesh.	20	Feeble E at 20 secs. Yellow F.	398	1		More powerful E at 42 secs. Blue F.
	10	Feeble E at 10 secs.	378	1		More powerful E at 25 secs.
	35	Residue partly ash.	285	3		Explosion at 25 secs.
		No glowing.	240	13		
		No glowing; unburnt residue.	226	25		
Anthracite coal 200 mesh.		No glowing.	260	8		
		No glowing.	388	7		Yellow F.
		No glowing.	362	12		Feeble E.
		No glowing.	325	22		
		No glowing.	300	30		
Hoo canal 200 mesh.	8	Feeble E at 12 secs.	382	1		Fairly powerful E.
	10	Very feeble E at 12 secs.	363	3		Fairly powerful E.
	11	Very feeble E.	340	3		No ignition.
Irish peat 200 mesh.		No glowing.	270	16		No ignition.
	20	No glowing; residue charred.	390	1		
		No glowing; residue charred.	385	1		
Cellulose 200 mesh.		No glowing; residue charred.	350	2		
	15	No glowing; residue charred.	396	1		
		No glowing; residue charred.	369	1		
Fusain 200 mesh.		Feeble E at 15 secs.	340	30		Powerful E at 30.
		No glowing.	389	6		Yellow F.
		No glowing.	370	10		

It may be observed that at temperatures only slightly above the minimum temperatures previously recorded for the occurrence of glowing, all

results also indicate that the volatile constituents evolved at any particular temperature have a considerable influence upon the temperature at which

spontaneous glowing occurs. The work is being continued along a number of lines.

We have to thank Miss P. Wray for help during the final preparation of this paper, and are indebted to the Lancashire and Cheshire Coal Research Association for supplying us with the samples of coal used in the work.

DISCUSSION.

MR. J. DRUMMOND PATON said that if reference was made to the work done by Mr. Lomax and himself (the speaker) it would be found that a solution for certain of Captain Sinnatt's curves was already provided by an examination of the halations of the volatile constituents as manifested in photomicrographs. The various agents from which these halations were obtained had already been stated, and the three peaks on the curve shown were dependent upon three regions from which the volatile matter was evolved, namely, the paraffinic, the cresylic, and benzenoidal. An examination of the oxidation effected and the ignition temperatures of the gases evolved and the sources of production would probably suggest many reasons for the glow and the explosive effects. It was well known that in gob coals, or coals which created gob fires, these effects were usually accompanied by a high percentage of oxygen, and this was necessarily the first source of supply for spontaneous ignition. With regard to the damping-down effect, it was well known also that any of the constituents mentioned could be emitted from a coal on a red-hot bed without being ignited, this being dependent upon the rate of emission of the same gases. The structure of coal, as shown by photomicrographs, very often explained the rate of emission, and the halation and colour effect would also define the zone in which they existed and the temperature at which they would be emitted. A damp or saturated hydrocarbon gas would not ignite in an atmosphere where it was in excess, and especially in regions and atmospheres with a large amount of vapour or steam accompanying the emission of the hydrocarbon.

MR. GIBSON asked whether the same time was taken in connection with the coarse material and whether the method of grinding and pressure were identical; also whether the same sieves had been used, whether they were standard gauze, and whether the method of sifting was standardised. The degree of coarseness of the coal was extremely important. He suggested the advisability of tests to ascertain whether the fineness or coarseness agreed by using a totally different method.

MR. W. PAYMAN suggested that instead of leaving the coal at the bottom of the crucible it might be permitted to fall through the hot gas. Gases evolved from the dust were not always present in dust explosions, these being obtainable with charcoal. With coal that was falling through a gas mixture it might be possible that ignition would take place at a much different temperature. In the case of coal burning in air in any type of industrial burner there was quite an efficient mixture of the coal dust and the air. He thought there was not such a free access of oxygen to a gob fire when it was starting as there was in the case of the experiments under review.

MR. TIDSWELL asked whether the absence of glowing, observed with certain coals after previous treatment in nitrogen, could not be more easily explained by the changed state of the coal after heat treatment, reducing its capacity for absorption of oxygen, rather than by the assumption that the glowing was caused by the combustion of the volatile decomposition products of the coal.

DR. H. F. COWARD said that, so far as he was aware, no other serious attack on the present problem had been attempted. The most severe

criticism he could make was that the authors were not measuring the ignition temperature of coal; they were measuring the ignition temperature, in the defined circumstances, of the most inflammable portion of the contents of the vessel, and these consisted of gas, vapour, and partially carbonised coal. The measurement of the ignition temperature of coal itself was an extremely difficult problem. Coal underwent slow exothermic oxidation in contact with air at low temperatures, and if it were contained in an enclosure the heat conductivity of the walls of which was zero, the coal must ultimately inflame. That was an extreme case unattainable experimentally, and in practice a compromise had been adopted in the definition of the ignition temperature as that temperature at and above which very rapid self-heating occurred in the reacting mixture, so that inflammation was observed within a small fraction of a second after the materials had been brought to that temperature. The authors had not been observing this, but they had been determining another very important constant connected with the decomposition and ignition of coal, and this might perhaps be better described by another name than ignition temperature, but it would lose none of its significance thereby. He (the speaker) had just used the word "constant," which he understood from Mr. Sinnatt was applicable if the standard apparatus were used in the standard way. In addition to those details of standardisation mentioned by the authors, they would perhaps find that other observers using exactly the same methods would get different results for one and the same coal, even if each used sieves of the same nominal size for grading the coal. The fineness of the sifted coal depended on the dimensions of the spaces between the wires, and these were governed by the diameters of the wires themselves, as well as by the number of wires per inch.

CAPT. SINNATT said that it was intended to examine the effect of the chemical composition of the coal upon the results, and it would be necessary to include an examination of the structure of the coal as indicated by the microscope. As the method was empirical in character, every detail was carried out under similar conditions, the same sieves and manner of grinding and sifting were used throughout the experiments. The coal used was freshly mined and the moisture present was standardised. In one case a sample of coal (Arley) was exposed to the air for three months in a fine state of division and then tested, when values much higher than those given by the freshly mined coal were obtained. Until further evidence had been brought together it was difficult to explain the increase in temperature of glow and ignition found after heating the coal in nitrogen in view of the fact that the temperature to which the coal was heated never exceeded about 350° C. When two points were found on the ignition curve two explosions had occurred. Their object in devising the test had been to keep the apparatus and method of manipulation as simple as possible, as, in their opinion, the determination of the temperature of glow (and of ignition) must in its essentials remain an empirical one until our knowledge of the constitution of coal is much more extensive than it is at present.

MR. HAROLD MOORE, referring to tests made by himself when designing the instrument, said that it was surprising how little the results were affected by such variations as halving or doubling the quantity of the charge put in, using from one quarter to four times the normal quantity of oxygen per unit of time, and also by the catalytic effect of the surface of the ignition chamber. The original crucible was made of platinum, while later on porcelain, silica, aluminium, copper, and other crucibles were used. In no case did the variation exceed 2° on

the ignition readings when the readings were somewhat about 300°C ., so that, though the test was empirical, there was something to be said in favour of that type of apparatus. He thought the peaks on Captain Sinnatt's curves could be explained by the generation of certain hydrocarbons or other organic compounds. The simple hydrocarbons, methane, ethane, and so on, had considerably higher ignition temperatures than the temperatures at which Captain Sinnatt was working, and might "blew out" the flame or "dilute down" the oxygen mixture until there was such a low concentration that the ignition point was raised. Incidentally, it could be expected that dilution of the oxygen from 100% down to 50% would greatly affect the result. During the course of experiments with aircraft engines, in which the exhaust gases were being trapped in the cylinder in order to raise the ignition point of the mixture, a certain number of tests were made, and he was surprised to find that on diluting the oxygen from 98% to 30% or 40% the ignition point was only affected about 2°C . The carbon dioxide was in these experiments supposed to represent the exhaust gas. Experiments conducted in air gave much less regular results. The ignition point of a mixture of two volatile compounds was a much more complex matter than might appear at first sight.

Meeting held at Grand Hotel on January 9, 1919.

MR. JOHN ALLAN IN THE CHAIR.

A NEW INSTRUMENT FOR MEASURING VAPOUR TENSION.

BY HAROLD MOORE, M.SC.TECH.

The determination of the volatility of motor spirit has hitherto been carried out by fractional distillation, but this test is only an indirect indication of the behaviour of the spirit in the engine. In the jet carburettor of the present day the ratio between the amounts of fuel in the liquid and in the gaseous state determines the ease of starting under standard conditions, and the "flexibility" of the engine whilst running at varying loads and speeds.

The proportion of fuel volatilised is dependent upon the vapour tension of the fuel, and the vapour pressure also requires to be determined in the valuation of motor spirits. The standard method of determining vapour pressures of liquids is by means of a barometer enclosed in a water jacket to enable the temperature to be regulated. The liquid to be tested is introduced under the lower end of the barometer tube by means of a pipette, the end of which has been bent. The fall in the mercury column after equilibrium has been established gives the vapour pressure at the temperature of the experiment.

This method is accurate and reliable and is most suitable when a very low degree of experimental error is essential. It is, however, very tedious to clean out and re-assemble the apparatus, and for this reason the method has not found use in technical work. Redwood and Thomsen devised a simple form of instrument for gauging the relative vapour pressures of motor spirits,* but this does not give true vapour pressure readings.

After experiments with various types of instrument, one of which is described in the "Automobile Engineer" of September and October, 1918, the author devised the apparatus shown in the accom-

panying figure. This consists of two tubes of about $\frac{1}{2}$ in. bore and about 800 mm. long, connected to each other and to a levelling bottle by means of a Y-piece and thick-walled rubber tubing. The two tubes are clamped in a vertical position and have mercury-sealed cocks at their upper extremities. One of the tubes is surrounded by a water jacket in which a thermometer is suspended.

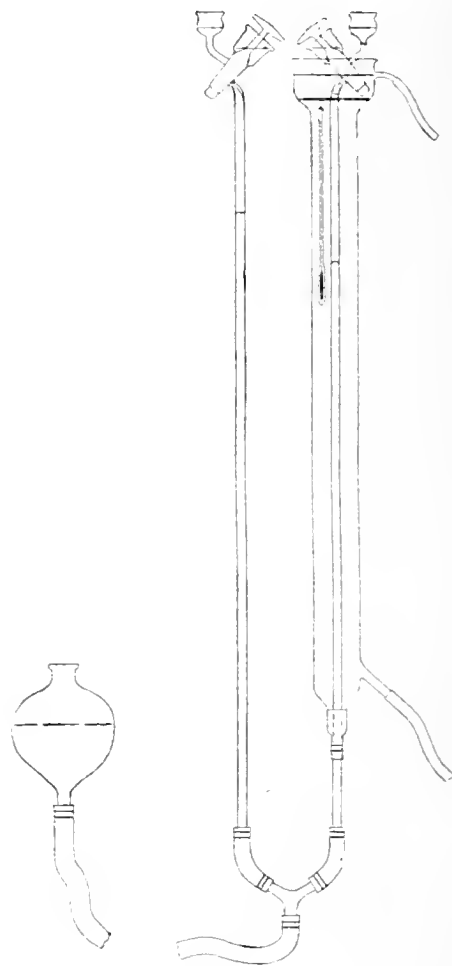


Fig 1.

When the levelling bottle is raised, the two cocks opened, and mercury flooded into the cups attached to the cocks, and then the cocks closed and the levelling bottle sufficiently lowered, two barometers are formed. The method of making a determination is as follows: One cup is filled with motor spirit; a small amount (measured by a mark below the cock) is passed into the tube, and sufficient mercury afterwards placed in the cup to ensure an effective seal. The levelling bottle is then lowered, when the barometric height may be read from the empty tube, whilst the jacketed tube containing the motor spirit gives a reading which is the barometric height minus the vapour pressure of the motor spirit at the temperature of the experiment. The difference in height between the two tubes is the vapour pressure reading. It is necessary for accurate readings to make a correction for the density of the mercury, but on the motor spirit tests in question this was not done, as the quantity was

* See V B Lowes, J. Soc. Arts, 1915, 63, 760.

considerably less than the normal experimental error.

When it is necessary to make a trial of a new substance the tube is best cleaned by repeated washing with the substance about to be tested. After each wash the mixture is removed by flooding it into the cup and absorbing in filter paper. Where two substances are insoluble in each other the washing method may be modified by the use of an intermediate substance which is soluble in both.

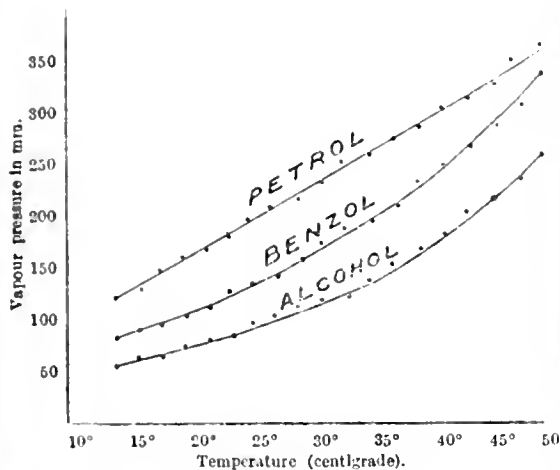


Fig. 2.

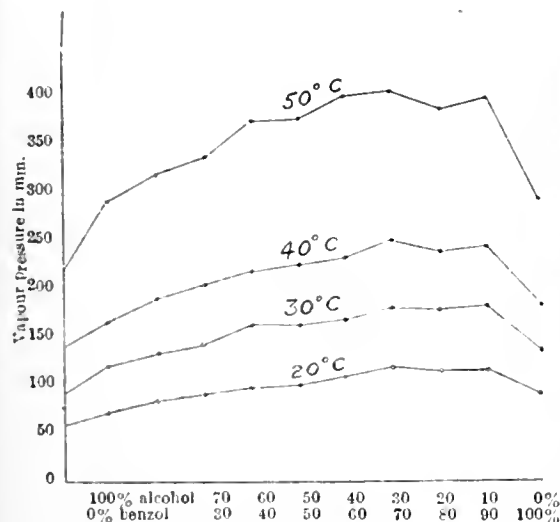


Fig. 3.

The vapour pressure increases with rise of temperature, and the results are best shown in the form of a graph, in which temperature and vapour pressure are the components.

The laws governing vapour pressures of mixed substances are somewhat complex, and a mixture of two soluble substances is liable to give a vapour pressure either lower than that of either constituent, or greater than that of either constituent, and may approach the sum of the vapour pressures of the substances at the given temperature. Cases in which the latter phenomenon is shown are of ex-

treme importance to the student of motor fuels, as any method of raising vapour pressure is of great value. In cases where the vapour pressure exceeds the necessary value there may be found plenty of cheap fuels which can be added to bring about the necessary reduction in volatility.

From these considerations it was deemed advisable to work out the vapour pressures of mixtures of important fuels with varying compositions and at varying temperatures. The temperature was controlled by passing the water through a heating coil before it entered the jacket until a temperature slightly in excess of that desired for the highest reading had been attained. The current was then discontinued and the readings taken as the apparatus slowly cooled.

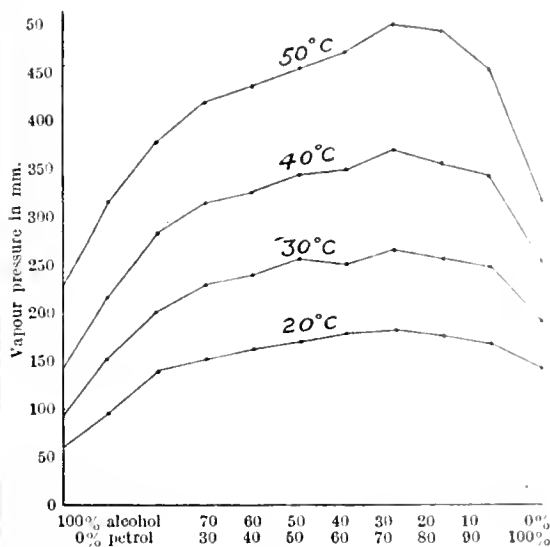


Fig. 4.

The vapour pressure/temperature graphs for benzene (crystallisable), petrol (Pratt's "Perfection"), and alcohol are shown in Fig. 2. The diagram for mixtures of 98% alcohol with benzene (crystallisable) at various temperatures is shown in Fig. 3, the vapour pressure being plotted against composition. Similar curves for alcohol-petrol mixtures have been prepared, and the results are shown in the curve in Fig. 4.

The experimental error when making determinations on water was only about 3 mm., but with these mixed motor spirits the error is undoubtedly much greater. In mixtures containing petrol a slight amount of gas is formed, which is not re-absorbed by increasing the pressure. This gas cannot be formed by leakage of the cocks, as it forms within one minute of taking readings, whereas the apparatus can be left charged under vacuum for many hours without any leakage becoming apparent. The results have not the degree of accuracy which is desirable for this class of work, but the essential point was to find the direction of the curves of pressures for various mixtures, and the general tendency is clearly indicated in the graphs.

DISCUSSION.

MR. MORTON said in regard to the permanent gas dissolved that he had found it necessary to take two

readings on a barometer tube—one where the liquid was at the top of the tube and one where it was at the bottom. The amount of gas dissolved could then be calculated when dealing with a pure substance. To obviate errors due to dissolved air, some of the liquid should be introduced into the barometer tube and the mercury lowered until it was at the bottom of the barometer tube and most of the dissolved air had been released; the liquid and the mercury should then be made to rise rapidly and a small amount of the liquor allowed to pass through the top, when the cock should be closed. A repetition of this procedure would remove all dissolved air. He asked how the difficulty in regard to grease on the stopcocks was overcome. He had had to devise an apparatus without stopcocks. In most of the determinations the chief difficulties were caused by the amount of dissolved air always present in any liquid and by using too small an amount of liquid for the estimation.

MR. GIBSON said that the approximately straight-line petrol graph did not agree with results he had obtained within the past few months, using an ordinary tube method, dealing with a petrol containing in some cases 5% of very light volatile constituent, and up to 20, 30, and 40%. The stopcock question was a difficult one. What guarantee was there that no leakages or evaporation occurred with very light constituents?

CAPTAIN F. S. SINNATT asked if the effect of naphthalene upon the vapour pressure of motor spirits had been tested. What was the effect of a small quantity of heavy or high boiling material upon the curves obtained?

MR. MOORE, in reply, said he had entertained the opinion that the gas formed was natural gas in solution. It was quite possible, even if compressed natural gas had not been used in the mixture to make up the motor spirit (which he did not think was so in the case of Perfection spirit), that some natural dissolved gas might come through in the distillation process. He had not analysed the gas itself. He had tried to remove the gas by subjecting the spirit to a vacuum and then blowing it out afterwards, and certainly after the gas was once removed no more was formed. When a small bead of gas was left in the top, if the mercury were raised a little higher and the gas blown out sufficient liquid was left in the tube to give a reading, and no gas would be formed. He questioned, however, whether such a course was permissible, since it was desired to test the spirit as it actually existed. A similar curve with a slightly higher vapour pressure was obtained if the gas was not extracted. The stopcocks were sealed with mercury, and even if the apparatus were left for four or five hours there was less than $\frac{1}{2}$ mm. loss. When the cocks had been well ground in, even after the lapse of a month, the leakage was mercury leakage and not air leakage. A small quantity of a very volatile constituent raised the vapour pressure a great deal. The measurements of level were taken by a rule placed directly behind; there was no necessity to use measuring instruments, as most of the experimental error was much greater than the error in actually reading the columns. He had not prepared curves in the case of naphthalene mixtures. The vapour pressures of naphthalene itself had been published in Schnitz's book on liquid fuels. A small quantity of heavy substance did not appear to affect the readings appreciably. Evaporation did not affect the results once the instrument was filled. In his experiments the material was only exposed for 10 to 30 seconds prior to the beginning of the experiment. The duration of the actual experiment was three hours. During the whole of that time no more of the volatile liquid was taken from the upper cup into the tube, it acting only as a seal.

Nottingham Section.

Meeting held on February 4, 1920.

MR. F. H. CARR IN THE CHAIR.

NOTE ON THE REICHERT-MEISSEL-POLENSKE METHOD.

BY H. DROOP RICHMOND, F.I.C., AND G. F. HALL, M.B.E.

The directions given by Polenske (*Z. Unters. Nahr. Genussm.*, 1904, 7, 273; this J., 1904, 387) are very explicit and appear to be adhered to generally, but in two respects analysts are prone to introduce slight modifications, *i.e.*, in the time of distillation and the temperature to which the water in which the flask containing the distillate is cooled. Indeed it appears general to depart from Polenske's prescribed temperature of 10° C., and most of the text-books give 15° C. as the temperature of cooling. Both these points have been investigated. The samples of coconut oil used in the two series were not identical.

Experiments on time of distillation.

Substance.	Time.	Soluble acids.	Insoluble acids.
	Mins.		
Butter	19½	31.0	2.25
"	20½	32.05	2.4
"	38½	32.0	2.35
Coconut oil ..	20	8.45	18.3
"	20½	8.3	17.9
"	24½	8.5	17.55
"	27	8.45	16.9
"	36	8.35	16.45

It is seen that with butter the time of distillation does not greatly matter, but with coconut oil it is essential to adhere to Polenske's directions (18 to 22 minutes).

Experiments on temperature.—Coconut oil.

Time of distillation. Mins.	Temperature of cooling. ° C.	Soluble acids.	Insoluble acids.
21	5	8.3	16.4
18½	10	8.0	16.6
20	10	8.5	16.0
20	10	8.5	16.4
19	15	8.5	16.1
20½	15	8.55	16.1
20	20	8.5	16.2

It is seen that the temperature to which the distillate is cooled does not greatly affect the results.

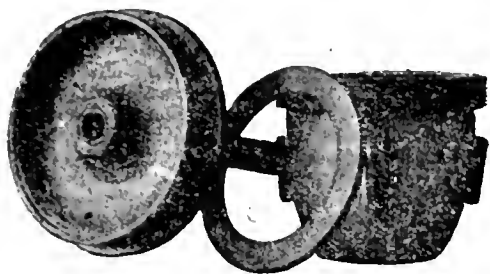
This work was done in the Analytical Laboratory of Messrs. Boots Pure Drug Co., Ltd., to whom our thanks are due.

SODIUM PEROXIDE FUSIONS.

A. E. MUSGRAVE.

When using the Roland Wild calorimeter the estimation of sulphur may be conveniently combined with that of the calorific value of the fuel. In many cases, however—as, for instance, foundry coke—it is desirable to estimate the percentage of sulphur without determining the calorific value. For this purpose—sulphur estimation—the Roland Wild calorimeter is unwise, as the top of the crucible is attached to the top and stirring gear of the calorimeter vessel. A very simple and convenient crucible cover was therefore made, so that

the crucible may be much more easily handled when carrying out sodium peroxide fusions with solid fuels and other substances.



The crucible and cover are shown in the illustrations. The crucible is of nickel; both cover and tube are of brass; ignition is effected by dropping down the tube a piece of red-hot nickel wire about 10 mm. long and 1.25 mm. diameter (17 to 19 gauge). The crucible top may be screwed down tightly on the rubber gasket, and the whole crucible immersed in a beaker of cold water to about 1 in. above the top of the crucible. If it is feared that any sulphur contamination may result from the rubber gasket (which has not been found to happen) this may be avoided by omitting the rubber gasket, screwing the top on the crucible and immersing the latter in the cold water to within about 3 mm. of the crucible top, then igniting, etc., as previously described.



The melt always leaves the crucible cleanly, and after several hundred fusions the crucible and cover appear as good as new. The brass tube in the crucible cover is 185 mm. long, 9.5 mm. diameter, and 7.5 mm. bore. It will be noticed that there is no trap at the top; this facilitates the introduction of the red-hot nickel wire, and the loss by spurting is negligible.

Communication.

A NEW TEST FOR THE DETECTION OF LIGNEOUS IMPURITIES IN COTTON AND COTTON WASTE FOR NITRATION PURPOSES.

BY FRANK LESLIE BARRETT.

The older method of detecting woody impurities in cotton to be used for making nitrocellulose, con-

sists in dyeing samples of the material with a basic dye, when these impurities appear as deeply dyed specks on a coloured background. This is known as the "dye test," and unevenness of dyeing is ascribed to the presence of oxycellulose in the fibres. The dye solution employed is sufficiently strong, however, to colour the fibres deeply, and the detection of the ligneous matter is thereby rendered more difficult.

The usual method of carrying out this test is to boil 2 grms. of the cotton with 100 c.c. of a 0.2% solution of rosaniline hydrochloride, and then to heat the material with repeated changes of water until the wash water is free from dye. The complete removal of the loose dyestuff from the fibre is difficult, and the test is therefore unsatisfactory as a means of detecting oxycellulose.

In investigating the efficiency of the dye test several cotton wastes cleansed by different processes were examined, and it was found that where small traces of certain impurities were present in the fibres, the cottons dyed to deep shades, whereas the soda solubility and copper values were normal. These impurities, which are probably phenolic in character, appear to act in a manner similar to that of the tannin compounds used in cotton dyeing, and assist in the fixation of the dye. When these cottons were deeply dyed, the detection of the woody impurity, which was only very slightly darker, was difficult. It was also found that the presence of local grease on the cotton causes uneven dyeing, and might be mistaken for oxycellulose.

Attempts were therefore made to devise a more satisfactory test for the presence of ligneous matter in cotton. A large number of experiments were made with various microscopical staining reagents for lignified matter. Such reagents as those suggested and discussed by E. Grandmougin,* Wheeler,† and Cross, Bevan and Briggs‡ were also tried and found to be unsuitable for the detection of woody matter in cotton waste by visual examination. These reagents included aniline, toluidines, xylydines, phenylenediamines, diphenylamine, naphthylamines, their various salts, methyl and nitro derivatives, phenols such as carbolic acid, cresols, dihydric and trihydric phenols, naphthols, and also nitro- and aminophenols. Attempts to develop Aniline Black on the ligneous matter were unsuccessful.

Attempts to make use of the specific reactivity of the lignone groups were therefore abandoned, and work was confined to the dyeing of the woody matter with basic dyes. It was found that Malachite Green, the oxalate or zinc double salt of tetramethyldiaminotriphenylcarbinol, is readily absorbed by woody matter. Endeavours were made to heighten the contrast between the dyed ligneous particles and the tinted cotton by the use of a second dye, Bismarck Brown, to counteract the green tint imparted to the cotton with production of a neutral shade, but such methods were rejected after extended trial.

Satisfactory contrast was obtained by treating the cotton waste dyed with Malachite Green with an oxidising agent, which immediately discharged the dye loosely held by the cotton fibres, whilst the retention of the colour by the particles of wood was more persistent. The addition of formaldehyde to the dye-bath assists the fixation of the Malachite Green by the ligneous matter, and the colour is not discharged by treatment with strong oxidising and reducing agents. Further, it was found that the colour could be conveniently discharged from the cotton by adding a solution of bleaching powder to the dye-bath in presence of a trace of acid.

* Z. Farben u. Textilchem., 1906, 5, 321-323.

† Ber., 1907, 40, 1883.

‡ Ber., 1907, 40, 3119.

Method of carrying out the test.—The dye solution is prepared by dissolving 0.1 gm. of pure recrystallised Malachite Green in hot distilled water or good tap water (about 100 c.c.), diluting to 500 c.c., adding 50 c.c. of 40% formaldehyde solution and 1 gm. of sodium bisulphate (NaHSO_3) dissolved in a little water, and making up to 1 litre.

The bleaching powder solution is made by shaking 20 grms. of bleaching powder (containing about 35–37% of available chlorine) with 1 litre of good tap water, allowing to stand, and pipetting off as required.

Dyeing is conveniently carried out in 500 c.c. porcelain beakers, eight of which can be heated in a convenient-sized bath and run in a series. 300 c.c. of dye solution is placed in each beaker. After the beakers have been heated in the water bath for a few minutes, 3-grm. samples of cotton are placed in each and allowed to remain in the dye liquor at the temperature of boiling water for 10 minutes, with occasional stirring. At the end of that time, 25 c.c. of the bleaching powder solution is added to each bath from a pipette, and after addition of the bleach solution the baths are stirred rapidly.

On addition of the bleaching solution the colour of the bath is immediately discharged. The cotton is allowed to remain in the hot liquor for a further 5 minutes; the liquor is then poured off, the cotton being used as a filter to catch any small particles of husk etc. The treated material is rinsed first with tap water and then with distilled water, and examined wet for green specks. It is important to use pure Malachite Green and to keep the baths well stirred during dyeing and bleaching.

Experimental.

Carded American cotton (good middling quality), which had been cleansed by boiling with caustic soda solution under pressure, was selected as a typical pure cotton. Samples of this material submitted to the test remained white. On careful examination minute specks of green-coloured woody matter could be detected occasionally in the samples.

Effect of typical ligneous impurities.—To investigate the efficiency of the test, a large number of experiments were made in which samples of the purified American cotton were adulterated with impurities commonly found in cotton waste, such as cotton husk, leaf and stalk, cotton seed, straw, including common cereal straw and the eastern grass used in packing bales, hemp and jute fibres, and teased fibres from the ordinary Hessian bale covers. These impurities were examined in the unboiled state and after they had been subjected to a pressure boil with caustic soda as in the ordinary process of cotton waste purification.

In every case the cotton remained perfectly white after treatment, whilst the impurity took the characteristic green colour and retained it. Where foreign vegetable impurity could not be detected by visual examination in the untreated materials, its presence was clearly disclosed by the test, the green dyed particles being very conspicuous on the white background, and on examination under the microscope particles of broken dyed fibre could be seen intermingled with the cotton.

Further experiments showed that, if the test is properly and carefully carried out, the dyed woody

material is not decolorised by prolonged exposure to the action of the bleach solution, and also that the test is unaffected by the presence of grease on the fibres.

Comparative tests on typical cotton wastes.—A large number of cotton wastes, purified by boiling with 2–4% caustic soda solution at different pressures, have been examined by the older dye test and by the test suggested above. The latter indicated the presence of the ligneous material much more clearly, and enabled a better discrimination to be made between the different samples. Thus, relatively large proportions of woody impurity were found in samples boiled at atmospheric pressure, somewhat less in samples boiled for short periods under low pressure, while samples boiled for 10 hours or more under pressure were found to be reasonably clean. As expected, cottons boiled at 80 lb. per sq. inch appeared to be more free from woody matter than those boiled at 40 lb., whilst those boiled at 40 lb. contained less ligneous impurity than those boiled at 20 lb. and at lower pressures. Again, materials boiled with 4% caustic soda appeared cleaner than those boiled with 2% caustic soda and 2% sodium carbonate.

Examination of a large number of cotton wastes prepared for nitro-cotton manufacture showed that in cases where the material had not been bleached the detection of the woody impurity was difficult in samples dyed with rosaniline hydrochloride on account of the original dark colour of the waste, whereas in samples dyed by the Malachite Green method the impurity was easily recognisable, as the dark shade disappeared on treating with the bleaching powder solution. When the particles were extremely small and unrecognisable by the routine test, they could easily be found by the modified method.

The test has been devised for the detection of woody impurity in cotton waste and not, of course, for the detection of oxycellulose. About 300 to 400 samples of wastes have been examined by the method, over 200 of these in comparison with the well-known rosaniline dye test, and in every case the former has been found to be much more discriminative. In no case could undyed woody matter be detected.

It has, for some time past, been employed as a routine test in a number of works and laboratories, and has been found to give excellent results.

It is desired to thank the Director of Artillery for permission to publish those results, Lieut.-Colonel R. A. Craig, C.M.G., R.A., and Sir R. Robertson, K.B.E., F.R.S., for facilities afforded in carrying out the experimental work, and Drs. W. H. Gibson and A. Forster for valuable suggestions made during the carrying out of the work, which formed part of an investigation undertaken at the Research Department of the Royal Arsenal, Woolwich.

ERRATUM.

SUGAR IN OAT STRAW AND CATTLE FOODS.

BY S. H. COLLINS AND A. SPILLER.

(This J., Mar. 15, 1920, p. 66 r.)

The last line but one of the first paragraph should read "which are relished by cattle, but it is doubtful."

Manchester Section.

Meeting held at the Grand Hotel on Feb. 6, 1920.

MR. JOHN ALLAN IS THE CHAIR.

A NEW CHARACTERISTIC FOR COAL: THE AGGLUTINATING POWER CURVE.

BY F. S. SINNATT, M.B.E., M.Sc. (TECH.), F.I.C., AND
A. GROUNDS, B.Sc. (TECH.), A.I.C.

This work is part of the investigation of Lancashire coals being carried out under the auspices of the Lancashire and Cheshire Coal Research Association.

The present paper contains a study of the agglutinating power of coal under certain special conditions, and affords an auxiliary means for the identification and valuation of coals, with especial reference to their caking qualities. Although the test for the agglutinating power of coal has been studied fairly completely by certain firms for industrial purposes, the literature of the subject is somewhat scanty, the following being the two chief contributions: Campredon* describes a method for measuring the caking value of coals by heating a standard weight of coal with varying weights of inert matter (Calais sand) in a platinum or porcelain crucible. The test was repeated until the maximum amount of inert matter which could be sustained by a coal was found, the coal still yielding a coherent button of coke. J. T. Dunn† observed that, occasionally, coals of high ash value had given higher agglutinating values than coals of a similar nature but of low ash value, the latter coals being obviously much more suitable for the manufacture of coke. He found that much more consistent results could be obtained by the use of anthracite in place of sand as the inert matter. The coal to be tested was passed through a $\frac{1}{2}$ mm. sieve (approximately 1/50 sieve), whilst the anthracite was graded from 1 mm. to $\frac{1}{2}$ mm. (i.e., while it passed through a 1/25 sieve it would be retained by a 1/50 sieve). The proportion of the weight of inert matter to the weight of coal was taken as a measure of the agglutinating power. In the discussion which followed this paper it was suggested that retort carbon might be substituted for anthracite, and one speaker also mentioned that particular care should be taken to specify the size of the anthracite. Further, it was stated that the coking test should be carried out in a muffle, and not over a Bunsen burner, and that the period of carbonisation should be controlled. It was considered that some definite mechanical means should be adopted to measure the strength of the resulting coke.

No further attempt, as far as we are aware, has been made since the publication of the above paper to standardise a method for determining the caking value of coals.

The temperature at which the coking or caking determination is carried out is usually lower than that at which the ash of coal becomes fusible, but there is a possibility (as suggested by Dunn) that the inorganic substances present in the coal may fuse during the carbonisation. It is well known that certain coals contain ferrous carbonate, and if the temperature at which the test is carried out is high, slight slagging may occur between the sand and the inorganic constituents of the coal. It is therefore desirable to choose an inert material which has no agglutinating power, either when

heated alone, or when heated with coal. Further, a substance which does not give rise to the evolution of gaseous products is preferable, as these have a distinct influence upon the rate at which the volatile constituents of coal are eliminated from the sphere of the reacting mass. The greater the volume of gas evolved by the inert matter and the greater the proportion of the latter present, the shorter the period the volatile constituents are in contact with the coal during the carbonisation. For these reasons it is considered that sand and anthracite are not entirely suitable. As it is desirable to measure only the caking power derived from the decomposition of the organic compounds present in the coal, which bind together the coke formed during the carbonisation, the inert material should resemble coke as far as possible. The inert material should not be porous, or it tends to absorb the binding or agglutinating substances produced during the carbonisation of the coal, the effect of which will be lost. Further, the particles of the material chosen should be spheroidal in shape, thus eliminating binding together of the substance by interlacing of the particles, either alone or when mixed with the coal or coke. The substance finally chosen by us was pulverised electrode carbon, which appears to satisfy most of the above conditions. "Flame-arc" carbons should not be used, as these are impregnated with sodium and calcium salts, which might act as a flux for the ash of the coal, thus producing a binding effect and influencing the agglutinating value of the coal.

In order to study the influence of the degree of fineness of the powdered inert matter, a quantity of carefully graded electrode carbon was prepared; 14 lb. of electrode carbon was pulverised so as to pass through a 1/10-mesh sieve. It was then intimately mixed and carefully sampled and analysed, with the following results:—Moisture, 0.17%; volatile matter, nil; ash, 1.4%; carbon, 98.35%. This material was then sifted through the following tiers of sieves: 1/10 to 1/30; 1/30 to 1/60; 1/60 to 1/90; 1/90 to 1/120; 1/120 to 1/180; 1/180 to 1/200; and through 1/200. Considerable difficulty was experienced in obtaining sieves of accurately sized mesh, and the above sizes had to be adopted finally, and the same material was used throughout. In our opinion, it is desirable to have the inert matter of such an approach to uniformity of fineness as will pass through, say 1/80 and be caught by a 1/90. The difference in size between the 1/60 and 1/90 is too large if great accuracy is aimed at, as it is possible for one specimen of the material to consist of a large proportion of 1/60 to 1/70 and a much smaller proportion of 1/80 to 1/90, and a second specimen to have the proportions reversed. In fact, the gradation in size of a 30's difference in mesh is not, in our opinion, sufficiently precise. It is hoped at a later date to repeat the experiments, using particles of inert matter more uniform in size.

A sample of Arley coal was carefully picked so as to be as free as possible from inorganic constituents, and the whole of the sample was ground to pass through a 1/90 mesh sieve. Determinations of the agglutinating value were then made by mixing one unit of coal with increasing proportions of sand (graded 1/60 to 1/90) and adopting the standard American method for the determination of volatile matter in coal (i.e., 1 gram. of coal or mixture is placed in a platinum crucible weighing 20–30 grms., on a silica triangle, over a Bunsen burner, and allowed to remain in the full flame for exactly seven minutes).

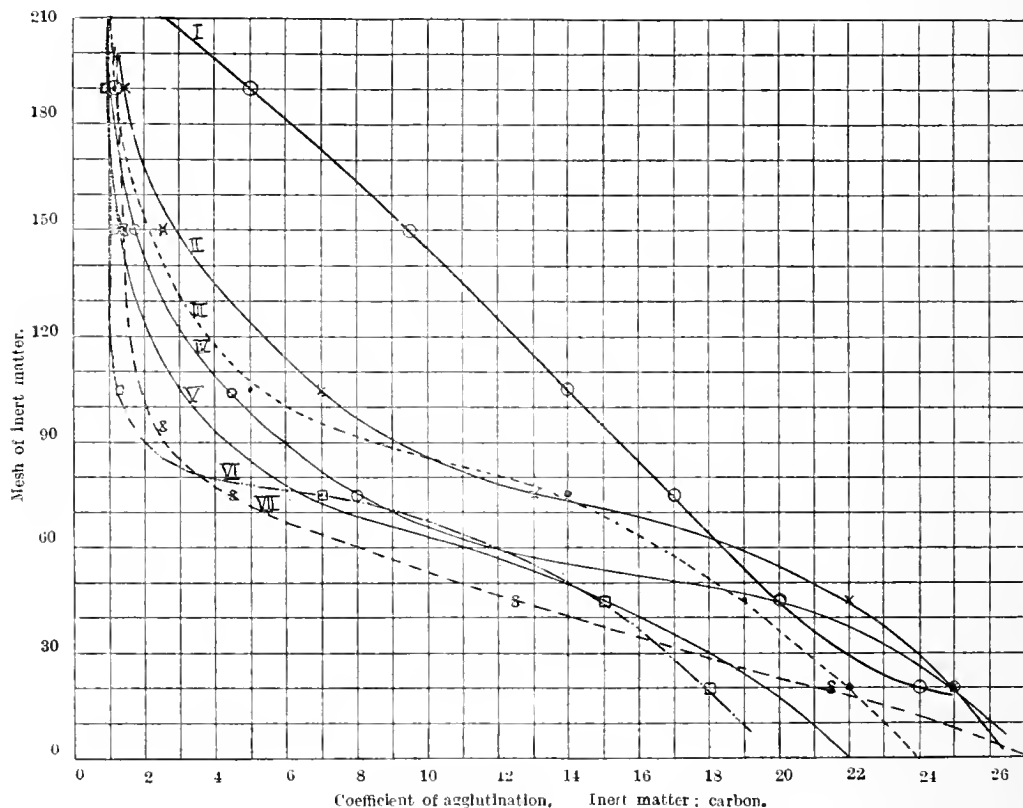
The coherence of the coke obtained from the volatile matter test was examined by removing the button and placing upon it a 100-grm. weight, the surface of which was equal to that of the button ob-

* *Comptes rend.*, 1895; see this J., 1896, 186.
† This J., 1913, 397.

tained. When the button crushed to powder under the pressure of the weight the maximum value was considered to have been attained. The term "agglutinating value" is used to describe the proportion of the weight of inert matter to unit weight of coal. With an Arley coal ground to pass through a 1/90 sieve the agglutinating value with sand 1/60 to 1/90 was 15, while with gas carbon 1/60 to 1/90 it was 7.5. It would thus appear that the relative weights of sand and electrode carbon used are directly proportional to the specific gravities of the two substances, and that the 1 grm. of coal requires equal volumes of sand and electrode carbon. It was therefore concluded that the coefficient of agglutination of a coal is a function of the volume or surface of the inert material used. This observation led us to examine the influence of the size of the particles of inert matter upon the agglutinating value of coals, and it was found that with decreasing size of particle the weight of inert material required to destroy the caking power of a coal was diminished. It was therefore decided to examine a number of typical Lancashire seams, and to plot as graphs the results of the agglutinating power determinations. The size of the inert matter experi-

standard American method in this work was to make the results of greater interest, as it enables chemists who do not possess an electric furnace or a standard muffle to compare the results with those we have obtained. At a later date it is proposed to publish the results obtained by carbonising the coal in an electrically heated tube, the temperature of which is controlled by a thermo-couple. The results for the different coals are given in the following table, and the figures are plotted in the diagram:—

No.	Name of coal.	Fineness of electrode carbon.					
		1/180 to 1/200.	1/120 to 1/180.	1/90 to 1/120.	1/60 to 1/90.	1/30 to 1/60.	1/10 to 1/30.
		Weight of electrode carbon per one unit of coal.					
1	Mountain Mine ..	5.0	9.5	14.0	17.0	20.0	24.0
2	Garswood, 9 ft. ..	1.5	2.5	7.0	13.0	22.0	25.0
3	Bacon Mine, tops ..	1.2	2.5	5.0	14.0	19.0	22.0
4	Arley ..	1.2	1.7	4.5	8.0	20.0	25.0
5	Ravine, tops ..	1.0	1.3	3.0	7.0	15.0	19.5
6	Hell Hole, middles ..	1.0	1.1	1.3	7.0	15.0	18.0
7	Chanters, 4 ft. ..	1.0	1.3	1.5	4.5	12.5	21.5



mented with was as previously stated, and in the graphs the size of the particle has been assumed to be the mean between the sieves used, *e.g.*, particles between 1/60 and 1/90 are taken as having an average size of 1/75 mesh.

Average samples of coal were obtained from a number of different seams, each of which was treated in exactly the same manner. The sample was ground till it would pass through a sieve of 1/90 mesh, and the agglutinating value determined. Each experiment was carried out in exactly the same manner, and the reason for the adoption of the

The graph for No. 1, which is practically a straight line, shows no evidence of any bend when the size of the inert matter becomes extremely small. This is interesting in view of the fact that No. 1 coal yields by far the most voluminous and perfect foundry coke of any of those examined.

An examination of the curves leads to one point of importance, namely, that not only does the fundamental property of caking of different coals vary considerably, but the property is influenced by the degree of fineness of the inert substance until, when inert material finer than 1/90 mesh is taken,

the caking property of the coal may be entirely destroyed by less than its own weight of inert matter. Attempts were made to determine the agglutinating value with less than one unit of inert matter, but the results have not, up to the present, led to a satisfactory conclusion, and are not included in this paper. There appears to be no reasonable objection to the conclusion that, if finer inert particles were used than those already experimented upon, the weight of inert material required would be reduced. It is suggested that the most satisfactory manner of determining the agglutinating power of coal is to prepare a graph, showing the relationship between the size of mesh of the inert matter used, and of the weight required to destroy the caking power of a unit weight of coal. This we have called the "agglutination curve"; it will be seen, however, that this determination is rather laborious, and as an alternative we suggest that the agglutinating powers of coal should be measured by the fineness of the inert matter required to destroy its caking properties. The manner of carrying out this test would be to mix one part of coal intimately with, we suggest, three parts of inert material the size of particle of which should be varied, until the coal will no longer yield a coherent button with the inert matter. The caking index would then be expressed in the degree of fineness of the inert matter, *e.g.*, one coal would be, say, 1/200 mesh, the next would be 1/75. It has been found by us that the size of the particle of inert matter does not influence the percentage of volatile organic matter evolved when the coal is heated as shown by the following series of experiments. In these experiments one unit of coal, powdered to pass through a 1/60 sieve and be caught by a 1/90-mesh sieve, was mixed with four units of inert matter (electrode carbon) of the following degrees of fineness and one grm. of the resulting mixture was carbonised under constant conditions:—

Size of inert matter (electrode carbon).	* Percentage of volatile organic matter evolved, calculated on coal taken.
1/10—1/30	28.6
1/30—1/60	29.0
1/60—1/90	29.4
1/100—1/200	29.4
1/200 and finer.	29.6

* Mean of three determinations for each experiment.

It can hardly be hoped to discuss the subject from any but a limited number of aspects in this paper, but the following points may be considered.

It is well known that during the washing of coal a considerable proportion of fine material is produced, part of which contains a high percentage of inorganic matter and fusain. This material has to be disposed of in one form or another, and where the intention is to manufacture coke the fine material or dant is distributed throughout the mass of the coal. It would appear that the addition of this fine dant, which usually possesses a low caking value, and in some cases is entirely without caking properties, although produced from a caking coal, will lead to a distinct difference in the caking value of the coal itself. Further, it is clearly important that the fine material should be mixed throughout the mass of the coal as perfectly as possible, as its accumulation in certain parts of the masses of coal would lead to the production of coke possessing distinct lines of weakness, due to the presence of sufficient finely-divided inert matter to render the coal at this particular point entirely non-caking.

The above observation applies to some extent to the processes in which coal and coke are mixed for use during steam raising, but it would seem that the addition of a small proportion of very finely-divided coke might have a distinctly beneficial

effect in reducing the caking power of a coal which otherwise might be unsuitable for steam raising.

The influence of the inert matter also arises in the stone-dusting of mines; finely-pulverised stone dust is sprayed into mines with a view to eliminate any chance of the fine coal dust in the mine exploding; the mixture of coal and stone dust deposits on the floor, walls, and roofs in the pit. The addition of a small proportion of this very fine material to caking coals would cause the coal to act as a non-caking coal.

It should be mentioned that the figures given in this paper were obtained under conditions which were as uniform as possible and represent the agglutination curves of the coals at one particular temperature. It appears that the shape of the curves may vary considerably with alterations in the temperature, and it is proposed at a later date to prepare curves for certain characteristic seams at a series of constant temperatures.

DISCUSSION.

The CHAIRMAN said that the subject presented many difficulties because of the extraordinary number of variables which had to be reconciled into something approximating to constants, and it was open to doubt whether some of the variables had been entirely eliminated. Had any precautions been taken to avoid prolonged contact of the products of combustion with the walls of the vessel, and to what extent was the vessel packed with the mixture used in determining the curve?

Dr. J. A. WEIL said that in using the so-called "sand" test he had found it necessary to standardise the size of inert material in exactly the same way as described in the paper. Even then the test was rather unsatisfactory, inasmuch as it did not accurately tell how a coal would behave in either a coke oven or a gas producer. The length of time taken in raising the temperature of the mixture of inert material and coal had a very considerable influence upon the strength of the resulting product. This was borne out by experience on a large scale. Coals which were used in coke ovens, heated very slowly under considerable pressure, and without the passage of gas through them, would make a good metallurgical coke; whilst the same coal in moderately small pieces heated moderately slowly in a gas producer, and with a large volume of gas passing through, did not cake to any extent. Owing to the "sand" test being so unreliable, even with the utmost standardisation it was not possible to get any real indication of how the coal would behave in a gas producer. For coke-oven work it was much more easy to guess from the indications given by the sand test, but for gas-producer work it was almost impossible to tell what would happen. It was possible that iron-bearing ash, when heated with an excess of carbon, might yield an ash fusing at a very much lower temperature than would the final fully-oxidised ash of the coal.

Mr. W. THOMSON said that when burning coal in oxygen in the calorimeter nearly all the ash was left in small fused pellets. He supposed that the sand used for mixing with the coal would be taken in its ordinary form, in which case there might be possibly a certain amount of combined water and moisture which might modify the results of the experiments.

Mr. W. H. H. NOMIS suggested that the actual nature of coking might be elucidated by considering the authors' data in the light of simple mathematical reasoning. If coal at the time of coking fused and merely penetrated the natural interspaces between the particles of inert matter, its agglutinating power should be independent of the size of the particles in the test described, because the interspace volume of packed spheres of the same size was independent of their diameter,

viz., 48% for rectangular or "pressure" packing and 26% for normal packing. The latter would only account for values up to 3, and coking would take place with contraction of volume. If, however, coking were considered as a surface effect, the agglutinating power would be reduced as the division or fineness of the inert matter increased, and coking would take place with increase of volume. Both of these were found to be the case. The total surface of the inert matter increased proportionally to the fineness. This was not sufficient to account for the remarkable influence of the size of the inert matter. In one sample, reduction in size from 1/150 to 1/190 mesh should have reduced the binding power of the coal from 4 to 3 instead of from 8 to 1 as found. The explanation for this result might be indicated by the microscope, and connected with the large increase in volume noted. Was it possible that some coals showing high agglutinating values were not suitable for the manufacture of the best metallurgical coke?

Mr. HAILSTONE asked if dried coal had been used in the experiments. The presence of water was liable to affect the texture of the resulting coke.

Mr. GIBSON said that for strict comparison coals containing the same amount of ash should be used, otherwise a variable was introduced which would be difficult to reconcile with the curves shown.

Mr. M. BARASH asked whether the authors could from their experiments advise as to the best standard conditions for obtaining the most representative agglutinating value of a coal. He had carried out satisfactory experiments with the aid of Lessing's apparatus, provided they were undertaken in a certain way. The furnace was heated to the required temperature, switched off for 5 mins., and then the silica tube containing the coal or the mixture of coal and inert matter was introduced, and the furnace switched on again for 7 mins. At the temperature at which the volatile matter was evolved, the walls of the tube were not hot enough to carbonise the tar etc., yet hot enough to allow the volatile matter to escape without deposition on the inside of the tube. The volatile matter came off completely in the first 2 or 3 mins., and then the contents of the tube caked without any possible interference by gases present during the experiment.

Mr. GROUNDS, answering Mr. Norris, said that research had been made with regard to the agglutinating power and the area of the inert matter exposed, and quoted the curves obtained.

Captain SINNATT, in reply, said that the test had been standardised as far as possible, and in order to eliminate the variations mentioned by Mr. Allan the standard American method was used. As a rule the volatile matter was evolved within about 80 seconds. It was probable that the variation in the results of the sand test was due to the size of the sand particles not being the same in each test. Further, it was not necessary for the ferric oxide of the coals to be reduced during the test, as most of the coals examined contained ferrous iron as a constituent of the white partings (ankerite), which were a common feature of Lancashire coals. The presence of iron in the ferrous state would undoubtedly tend to the formation of silicates which might be fusible. Up to the present it had not been possible to standardise the physical appearance and properties of the coke, but it would be considered during the continuation of the work. The fusibility of the ashes in Lancashire coals varied very greatly, and it was for this reason that electrode carbon was adopted as the inert material, in order to eliminate the influence of the combination of sand with inorganic constituents of the coal. With regard to the remarks of Mr. Norris, an attempt had been made to obtain a mathematical expression for the agglutinating value of coal,

and in the case of certain of the coals there appeared to be a distinct law governing the agglutinating value. It was not, however, considered desirable to elaborate these theories until a greater number of coals had been examined. The difficulty of determining the interspace volume was practically insurmountable, as it was not possible to say whether the particles were normally or abnormally piled. In all the experiments coal which had been air-dried for 24 hours was used, and the ash content was that of the ordinary coal. It was most desirable that the conditions under which the test was carried out should be most carefully standardised, and experiments were projected in which it was proposed to use an apparatus of the type described by Lessing.

ESTIMATION OF THE NITRO GROUP IN AROMATIC ORGANIC COMPOUNDS.

T. CALLAN, M.Sc., Ph.D., J. A. RUSSELL HENDERSON, D.Sc., AND N. STRAFFORD.

Of the many methods proposed for the estimation of the nitro group in organic compounds only a limited number are of general application, although many have a limited application to specific nitro compounds. The general methods available for determination of the nitro group may be classified as follows:—

1. Those involving the reduction of the nitro-group to the amino group by use of excess of a reducing agent, followed by determination of this excess with a suitable reagent.

Of these the better known are those involving:—(a) reduction by titanous chloride according to the methods of Knecht and Hibbert collected together in their monograph ("New Reduction Methods in Volumetric Analysis"); (b) reduction by stannous chloride.

2. Those involving the reduction of the nitro group with a reducing agent, followed by determination of the amount of amino compound produced, e.g., by titration, by means of a standard solution of sodium nitrite.

3. Those involving a determination of nitrogen by various modifications of the Kjeldahl method.

The authors have used the titanous chloride method of Knecht and Hibbert very extensively for a large number of substances, in most cases with extremely satisfactory results. It has been found, however, that certain classes of nitro compounds give very low results when analysed by this method, as will be later shown.

The method has one very serious defect when used in the analysis of substances such as are manufactured as intermediate products in the dyestuff industry. The method as recommended by Knecht and Hibbert requires a very small weight of nitro compound to be taken, and when dealing with moist pastes or damp press-cakes the difficulty of weighing out such a small quantity which shall be a representative sample may introduce a serious error. In such cases less convenient and possibly less intrinsically accurate methods may actually give figures more accurately representing the bulk of material.

Although in so many cases titanous chloride gives excellent results, in certain cases, in particular *a*-mononitronaphthalene, *o*-nitroanisole, nitrocresyl methyl ether, and similarly constituted compounds, quite erroneous results were obtained.

Thus a sample of very carefully purified *o*-nitroanisole, b.pt. 162° C. at 12 mm., when analysed by Knecht and Hibbert's method by boiling with excess of titanous chloride in alcoholic solution, followed by back titration of the excess by iron alum, gave NO₂ equivalent to 61.7%, 64.8%, and 64.0% of *o*-nitroanisole. These results, although of the same order, are obviously incorrect. Similarly, with a

sample of pure nitroresyl methyl ether, the method gave figures corresponding to 68.1%, 70.8%, and 71.9% of nitroresyl methyl ether.

In the case of α -mononitronaphthalene the figures were very variable, ranging from 73.8% to 96.1%. In the case of this substance Knecht specifically states* that reduction with titanous chloride must take place in presence of no larger amount of hydrochloric acid than is necessary to keep the titanium chloride in solution. Even on reducing the quantity of acid to this amount we found that it was still impossible to obtain concordant results, and the same was found to be true with α -nitroanisole and α -nitroresyl methyl ether.

It has long been known that during the reduction of certain nitro compounds in presence of hydrochloric acid chlorination may take place simultaneously with reduction.† For complete reduction to a chlorinated amine, however, only two-thirds of the amount of hydrogen is required, compared to reduction to the non-chlorinated amine, since the hydrogen liberated during chlorination is also available for the reduction. Correspondingly, if a chlorinated amine were produced during the reduction by titanous chloride, in the extreme case, i.e., when the product is entirely the chlorinated amine, only two-thirds of the amount of titanous chloride would be required for complete reduction, so that calculating back from this amount analysis of the substance would only indicate 66.6%.

Normally, however, the reduction of a nitro compound to the corresponding amine takes place quantitatively except when conditions favouring chlorination are chosen, e.g., reduction in presence of alcohol. From the results of our analysis of certain substances such as α -nitroanisole, nitroresyl methyl ether, and nitronaphthalene it would appear that reduction with titanous chloride invariably leads to simultaneous production of chlorinated compounds in these cases, and it is in fact possible to isolate such compounds from the products of reduction.

There is therefore a constant danger that analyses of nitro compounds by the titanous chloride method may be quite erroneous unless it is known whether the reduction product is entirely free from chlorinated derivatives. To obviate this source of error—which is very pronounced in the case of α -nitroanisole—the authors have employed titanous sulphate in all those cases where titanous chloride, even in presence of the smallest feasible amount of hydrochloric acid, gave either low or very discordant results, with the result that it has been found that such substances can be smoothly and quantitatively reduced without simultaneous chlorination.

In using titanous sulphate it was at first found extremely difficult to obtain a constant titre of the solution employed against a standard solution of iron alum. This was found to be entirely due to the great ease with which titanous sulphate solution is oxidised at the boiling temperature by atmospheric oxygen—precautions which are sufficient to prevent oxidation of titanous chloride being quite insufficient for titanous sulphate. On boiling titanous sulphate solution in a flask provided with a two-holed stopper with narrow entrance and exit tubes for carbon dioxide, and working with great care to prevent oxidation, then cooling in a stream of carbon dioxide, it remains of constant strength when titrated against a standard solution of iron alum, no special precautions being necessary with the cold solution.

The following table shows results obtained with certain substances which are found to give anomalous results with titanous chloride. In

addition to these substances, many other nitro compounds, the determination of which with titanous chloride gave no difficulty, have been analysed by means of titanous sulphate, concordant results with the two titanous salts being readily obtained.

	Titanous chloride.	Titanous sulphate.
Nitronaphthalene	73.8% .. 89.0% ..	90.9% .. 90.9% ..
α -Nitroanisole *	61.6% .. 61.7% ..	97.6% .. 97.6% ..
"after previous sulphonation	61.0% ..	97.1% ..
Nitroresyl methyl ether* ..	95.6% .. 95.4% ..	96.7% .. 97.0% ..
"after previous sulphonation	68.1% .. 70.8% ..	97.1% .. 96.7% ..
Nitrobenzene	71.7% ..	96.5% ..
"after previous sulphonation	99.2% ..	97.8% .. 98.2% ..†
		99.2% ..

* These substances could only be purified by fractional distillation under reduced pressure and these figures probably represent the actual amount of nitro body present as these results agree exactly with others obtained by the zinc reduction method.

† Low result probably due to volatilisation of the nitrobenzene during the hot reduction.

From these results it becomes apparent that when the Knecht method of titration with titanous chloride is employed for unknown nitro compounds, the analysis should be confirmed by making a duplicate analysis with titanous sulphate, as only by this means can it be assured that chlorination has not simultaneously taken place. It is, however, just as convenient to substitute titanous sulphate for titanous chloride in all cases, and the possibility of this error is thereby avoided.

The use of titanous sulphate also in many cases avoids the necessity of sulphonating, which in some cases, e.g., nitroresyl methyl ether, readily leads to charring, sulphonation being required only in those cases where the nitro compound is sufficiently volatile in steam to entail possible loss during the reduction in boiling solution.

In using titanous sulphate we find that there is no advantage in using a solution weaker than 5%, as this strength has been found to give satisfactory results and avoids the necessity of having to take such small amounts of the nitro compound for analysis. This solution is conveniently prepared by adding 400 c.c. of commercial titanous sulphate solution (about 12%) to about 500 c.c. of dilute sulphuric acid (1 in 10), boiling for a few minutes, cooling, and making up to 1000 c.c. No further addition of acid is necessary.

Reduction with stannous chloride.

Methods for the estimation of the nitro group by reduction with excess of stannous chloride and subsequent determination of the excess of this substance by means of iodine or permanganate date back several years previous to the introduction of titanous chloride by Knecht and Hibbert, the chief modifications of the original method of Limpricht (Ber., 1878, II, 35) being suggested by Young and Swain (J. Amer. Chem. Soc., 1897, 19, 812); Altmann (J. prakt. Chem., 1901, 63, 370); Colver and Prideaux (this J., 1917, 36, 480); Sachs (*ibid.*, 1915), and more recently by Druce (Chem. News, 1919, 118, 133).

The authors have made numerous determinations of nitro compounds, using the various modified methods involving the use of stannous chloride, and in no case have they found the slightest advantage over titanous chloride, whilst in many cases results were less satisfactory; also the time and manipulative skill required and the chances of possible error were considerably greater.

From a study of the papers already referred to it will be seen that chlorinated amino compounds are at least as readily formed during reduction with stannous chloride as with titanous chloride, and the objections raised to the use of the latter apply to the former with equal force.

* "New Reduction Methods in Volumetric Analysis" 1914 Ed., p. 130.

† Fittig, Ber., 1875, 8, 15. Schöler, Ber., 1878, 11, 1201. Gabriel and Stelzner, Ber., 1896, 29, 306. Blankens, Rec. Trav. Chim., 1907, 25, 365. Ramberger, Ber., 1895, 28, 251. Hurst and Thorpe, Chem. Soc. Trans., 1915, 107, 941.

The authors of papers describing the use of stannous chloride as a reducing agent all state that they get results lower than theoretical in many cases. They incorrectly assume that stannous chloride is not a sufficiently energetic reducing agent, having failed to appreciate the fact that simultaneous chlorination and reduction may take place.

Reduction of the nitro group to the amino group and subsequent determination of the latter.

We have been unable to find any direct references in the literature to this method, which, although only of limited application, is yet of very great value in the case of a considerable number of nitro compounds, i.e., those which can be smoothly reduced to an amino compound which can be quantitatively converted into a diazo compound by means of nitrous acid. About 1/20 grm.-mol. of the nitro compound is either dissolved or suspended in excess of dilute hydrochloric acid and zinc dust gradually added in considerable excess, the reaction being kept vigorous by occasionally warming the solution. After about an hour the solution is filtered, diluted, cooled by addition of ice and titrated with $N/2$ sodium nitrite until a drop of the solution applied to starch-iodide paper used as an external indicator shows excess of nitrous acid to be present.

In this method it is immaterial whether chlorinated amines are formed or not, provided such chlorinated amines, as is usually the case, react with nitrous acid to form diazo compounds. As will be seen, 2000 c.c. of $N/2$ sodium nitrite is required for each grm.-mol. of mononitro-compound reduced to monoamino-compound.

We have found the method extremely useful for a considerable number of substances, e.g., nitroanisole, *p*-nitrochlorobenzene-*o*-sulphonic acid, *p*-nitrotoluenesulphonic acid, etc., and in all cases have obtained excellent agreement with determinations made with titanous sulphate, though not necessarily with titanous chloride. Thus, a sample of nitroanisole gave figures of 97%, 97.6%, and 64.0% when analysed by these three methods respectively. The method has the decided advantage in works' practice that it is free from manipulative difficulties and involves no special form of apparatus, whilst the fact that a comparatively large weight of sample can be taken for analysis means considerably less chance of error due to sampling difficulties.

It is important to insure that both the acid and the zinc dust are free from iron or, alternatively, to make a blank determination to ascertain what amount of standard nitrite solution is taken up by traces of ferrous iron present after reduction.

It is not proposed here to discuss the value of the Kjeldahl method, with its voluminous literature, for the determination of the nitro group, as although the authors have carried out a considerable amount of work on the subject, particularly from the novel point of view of the use of titanous sulphate as both reducing and catalytic agent for the promotion of oxidation, the results are at present not sufficiently advanced to permit of publication.

In conclusion, we desire to express our thanks to The British Dyestuffs Corporation (Blackley), Limited, in whose Central Analytical Laboratory the work was done, for permission to publish the results of this investigation.

DISCUSSION.

Prof. KNECHT said that the fact that the titanous chloride method as originally recommended by him had failed in the case of α -nitronaphthalene, had been communicated to him some years ago. It had been found that by using very much weaker hydrochloric acid apparently reliable results could be obtained. There were several peculiarities in the

reaction of titanium salts which it seemed difficult to explain. For example, when diazobenzene chloride was treated with titanous chloride in concentrated hydrochloric acid solution there was no reduction, whereas in a very dilute solution, in the presence of a small amount of hydrochloric acid, the action was quantitative. Again, when a dilute solution of titanous chloride was boiled with sodium persulphate very little action took place, but if a 15% solution of sulphate were used the reduction was quantitative. Titanous fluoride used in place of the sulphate might give equally reliable results while at the same time being more stable in contact with air. Was the *o*-nitroanisole sulphonated in carrying out the determination or was the product dissolved in alcohol? Alcohol was always liable to introduce certain irregularities into the process, and, personally, he thought that sulphuric acid would, in the majority of cases, even if it only acted as a mechanical solvent, give better results.

Prof. A. LAPWORTH said that in the application of stannous chloride some substances gave results 10% too low, even though they were sulphonated nitro compounds. In such cases it was desirable to standardise the stannous chloride solution with a nitro compound which was nearest to that which was to be estimated. Orton and Jones had shown that the aceto-chloroanilides and hydrochloric acid first reacted to form acetanilides and free chlorine, which then reacted to give the chlorinated acetanilides. If Orton's explanation was general then there was a possibility of effecting a great variation in the results according to the manner of titration.

Mr. L. G. RADCLIFFE said that after some difficulty he had applied titanous chloride to the determination of the nitro musks in admixture with acetanilide.

Dr. CALLAN, in reply, said that they had made practically no differences in the technique of the original method as devised by Prof. Knecht. He did not agree with Prof. Knecht that it was generally advisable to sulphonate. This involved the use of oleum sometimes for some hours, which in many cases, for example, *o*-nitrocresyl methyl ether, caused charring, whilst by dissolving in alcohol and adding to the titanous sulphate solution the analysis required only about 10 minutes. Solution in alcohol certainly increased the likelihood of chlorination, and therefore when using titanous chloride sulphonation was decidedly advantageous, but with titanous sulphate where no chlorination was possible an alcoholic solution was greatly to be preferred. Attempts to use stannous sulphate in place of stannous chloride gave unpromising results, this material being very difficult to work with.

Newcastle Section.

Meeting held at Armstrong College, Newcastle, on November 19, 1919.

PROF. P. P. BEDSON IN THE CHAIR.

A STUDY OF THE FRUIT OF SCHLEICHERA TRIJUGA, WITH SPECIAL REFERENCE TO THE GENERATION OF HYDROCYANIC ACID IN THE SEED.

BY NAGENDRA N. SEN-GUPTA, B.Sc.

Schleichera trijuga is a large deciduous Indian tree belonging to the natural order Sapindaceae. It is of very common occurrence in dry forests all over India—Bengal and Assam being possibly excepted—

and also in Ceylon and the East Indian Archipelago. In virtue of the quality of its timber it is a factor of considerable importance in the forest economy of India, but this study is concerned with the fruit of the tree, the full recognition of the value of which as a source of vegetable fat and cattle food is almost sure to raise its silvicultural status.

The annual production of fruits is enormous, although it is very doubtful whether any serious efforts have so far been made to collect them systematically and to dispose of them in such a way as to obtain the best value of the fat as well as of the residue left after expression.

The cyanogenetic character of the seed of *Schleichera trijuga* was revealed more than a quarter of a century ago in connection with certain investigations into the properties of "Macassar oil," but, as far as I am aware, very little work has been done on the subject. But for the outbreak of the war in 1914 the subject would have been studied before now at the Imperial Institute, through whose generosity the material, which formed the basis of my work, was made available to me.

The work embodied in this paper falls under the following three heads: (1) A general survey of the constituents of the various parts of the fruit from the feeding point of view; (2) A study of the cyanogenesis in the seed; (3) An examination of the fat.

The constituents of the various parts of the fruit.

The fruit is a nut varying considerably in shape and size. The commonest and perhaps the typical shape is ovoid, the diametrical range being from $\frac{1}{2}$ in. to $\frac{3}{4}$ in. Generally the fruit is one-seeded, but twinning is by no means uncommon. The seed is protected by two distinct shells, separated by an intervening dark-brown layer of soft membranous character. Starting from the exterior, these four morphological units may be represented as follows: Outer shell, 50.8% by weight; membranous layer, 12.4%; inner shell, 14.6%; seed, 22.2%. Analysis of the two shells and the membranous layer shows that their inclusion in the composition of any kind of feeding-stuff is entirely out of the question. The shells might, however, be profitably treated for the manufacture of charcoal and volatile products.

The most obvious features of the seeds are their excessively high fat content and their extraordinary liability to the attack of fungi. About 70 per cent. of the fruits placed at my disposal were affected by one or more species of fungi, although the damage to the seed was not a constant consequence. The fact that the sphere of fungoid activity is inside the fruit, with the outer shell intact, indicates that the fungi may be of Indian origin and not the result of inoculation during storage at the Imperial Institute. Fully 40 per cent. of the fruits examined had their interior absolutely destroyed. Another 25 per cent. showed seeds in an advanced stage of disintegration; these chemically altered seeds will be referred to as "diseased seeds," in contradistinction to healthy ones.

The ether-extract from diseased seeds shows a very high degree of decomposition of the fat, the acid value being 109.6 as compared with 13.06 for healthy seeds.

The following figures for the composition of the two kinds of seeds have been obtained by the conventional method of representing the percentage of soluble carbohydrates by the figure obtained by subtracting the combined percentages of water, oil, proteins, fibre, and ash from 100. It is realised that, owing to the fact that nitrogen is present in the seed in at least three forms—true proteins, amides etc., and glucoside—the use of the figure $N \times 6.25$ will give an erroneous idea of the protein and also therefore of the carbohydrates, but since the identity of the glucoside has not been definitely established, it is proposed to adhere to the standard method.

	Healthy seed.	Diseased seed.
Moisture	3.20	3.00
Oil	67.13	63.23
Proteins	18.20	13.30
Carbohydrates	6.14	11.39
Fibre	3.40	5.63
Ash	1.93	2.85
Total nitrogen	2.912	2.128
N as true proteins	2.436	
N as amides etc.	0.448	

The amount of hydrocyanic acid yielded by healthy seeds under the most favourable conditions of hydrolysis hitherto observed has been 0.574%, corresponding to 0.298% of nitrogen. The discrepancy between this figure and the difference between the percentage of total nitrogen and the combined percentages of nitrogen as true proteins (precipitated by copper hydroxide) and amides etc. appears only to be capable of explanation by assuming that copper hydroxide not only prevents the hydrolysis of the glucoside by the water used, but also precipitates the glucoside together with the true proteins. This point remains to be definitely settled. The cyanogenetic hydrolysis of the glucoside during digestion with concentrated sulphuric acid is not a possibility, since when pure amygdalin is digested in a similar way the entire nitrogen-content is recovered as ammonium sulphate.

Probable composition of the feeding stuff derived from healthy seed.

The possible or advisable degree of expression of the seeds can only be definitely established by actual experiments on the manufacturing scale. The inhibitory effect of the common fat-solvents on the action of the enzyme (discussed later) indicates the superiority of the extraction method over that of expression in dealing with the fat in this particular case, unless it is possible to destroy the enzyme by heat (wet or dry) prior to expression without affecting the fat. In the absence of such heat-treatment the expression method will be fraught with the danger of contaminating the fat with hydrocyanic acid and leaving a large proportion of the glucoside together with the active enzyme in the residue, as it has been observed that a certain amount of hydrolysis takes place in crushing the material even in the absence of external moisture. As it is practicable to remove the last trace of fat from the seed by the extraction method, a composition may be suggested for the residual cattle food which will be a fair approximation to what should be the case in actual practice. In view of the striking poverty of carbohydrates in the seed, the fat-content of the residual food must be higher than is the case in the ordinary cattle cakes and meals. Taking all the facts into consideration, it appears that the percentage of fat in the residue should not be less than 15. Calculating on this basis, it is found that if 91.4% of the fat present in the healthy seed is removed, the composition of the residue should include 15 per cent. of fat. Thus, 100 lb. of seed should yield 38.67 lb. of cattle food containing moisture 8.28, oil 14.99, protein 47.07, carbohydrates 15.88, fibre 8.79, ash 4.99 per cent.

A study of the cyanogenesis in the seed.

The literature on the subject is so obscure that it is difficult to say with certainty when and by whom the discovery of the cyanogenetic property of the seed was made, but it is more than probable that the credit belongs to Trümmel,* who, working alone in 1889, and in conjunction with Kwassick,† in 1891, was able to obtain hydrocyanic acid both from the seed and the fat. The fat has since been examined by several workers, most of whom have confirmed the presence of hydrocyanic acid in it.

It is hardly conceivable that hydrocyanic acid can be present in the seed in a pre-formed state, and it

* Apoth. Zeit. 1889.

† Pharm. Zeit. 1891.

may be assumed that its source is a cyanogenetic glucoside. The hydrolysis under different conditions is quite typical of enzyme activity, hydrocyanic acid and a reducing sugar having been detected. Attempts to isolate the glucoside have proved unsuccessful.

The usual method of isolating enzymes has led to the separation of a nitrogenous substance which, when dry, presents the typical appearance of an enzyme, and which has therefore been taken to be the agent responsible for the hydrolysis of the glucoside. The dry white powder resulting from the gelatinous mass precipitated by an excess of alcohol is insoluble in water, but forms an opalescent suspension. Such enzymic solutions will not hydrolyse pure amygdalin or the glucoside in the crushed seed in which the enzyme had previously been destroyed. Further, 1 grm. of ground seed, which, under the same conditions of temperature etc., yielded over 3 mgrms. of hydrocyanic acid in a medium of pure water, was incapable of evolving even a trace of the acid when the hydrolysis-medium consisted of alcohol and water in the same proportions as used for the precipitation of the enzyme. In this respect the enzyme resembles urease.*

Evolution of hydrocyanic acid under different conditions of hydrolysis.

A number of experiments have been carried out with the apparatus devised by Mr. S. H. Collins for his research on linseed.† My best thanks are due to him for permission to use his apparatus. The material is hydrolysed at constant temperature and the hydrocyanic acid evolved is removed by an adjustable current of hydrogen and fixed as sodium cyanide, which is estimated colorimetrically. The hydrocyanic acid has also been estimated volumetrically by means of $N/20$ silver nitrate solution, the end-point being quite sharply defined. The volumetric method has always given somewhat higher results than the colorimetric method, but the latter is a reliable guide to comparing the effects of various conditions on the hydrolytic action of the enzyme.

The results of these tests have proved that cyanogenesis is confined to the seed. The evolution of hydrocyanic acid from diseased seeds is very small. At 25° C. 1 grm. of ground diseased seed evolved only 4% of the amount obtained under similar conditions from healthy seeds, but at 35° C. the amount increased to 19%. The reason for this increase is not apparent, since the enzyme activity is at a maximum at about 25° C. (see later). The figures showing the rate and amount of evolution of hydrocyanic acid in the two cases both at 25° and 35° C. are tabulated below:—

Table I.

Weight of material used=1 grm.

Time.	Mgm. HCN from healthy seed at 25° C.	Mgm. HCN from diseased seed at 25° C.	Mgm. HCN from healthy seed at 35° C.	Mgm. HCN from diseased seed at 35° C.
$\frac{1}{2}$ hour	0.16	0	0.19	0
1 $\frac{1}{2}$..	1.56	0.06	1.03	0.05
3 $\frac{1}{2}$..	2.56	0.15	1.48	0.28
5 $\frac{1}{2}$..	3.22	—	1.62	—
7 $\frac{1}{2}$..	3.50	—	—	—

Effects of thermal conditions.

Several investigators‡ on the subject of enzyme

* H. E. Armstrong and E. Horton, Roy. Soc. Proc., 1912, B. 85.

† Proc. of Univ. Durham Phil. Soc., 4, [3]; J. Chem. Soc. 1912, ii., 586.

‡ Collins—Rate of evolution of HCN from linseed under digestive conditions. Proc. of University of Durham Phil. Soc., Vol. IV., Part 3. Compton—Constancy of the optimum temperature of an enzyme under varying concentrations of substrate and of enzymes, Roy. Soc. Proc., B, Vol. 28, 1914.

activity have found that the optimum temperature for enzymes lies somewhere about 45° C. My experiments with the enzyme of *Schleicheria* seeds indicate that in this case the optimum is 25° C., at which temperature the yield of hydrocyanic acid is, roughly, 120% higher than that at 35°, while at 15° the evolution is less than at 25°, but is still in excess of the amount given off at 35° C. Readings have also been taken at 45°, 65°, 75°, and 95° C., and the results show a progressively inhibitory effect of wet heat.

The following results of volumetric estimations made at 15°, 25°, and 35° C. give an idea of the total amount of hydrocyanic acid evolved. These results confirm the statement made as to the optimum temperature of enzyme activity: At 15° C. 2.499 mgrms. of HCN was evolved per grm. of material; at 25° C., 3.982 mgrms.; at 35° C., 1.890 mgrms.

To observe the effect of steam 3 grms. of the ground material was subjected to a current of steam for half an hour, the distillate being collected in a dilute solution of sodium hydroxide. The steamed material was examined for hydrocyanic acid in the usual way for about four hours at 25° C. The distillate showed the presence of 0.54 mgrm. of HCN (0.18 mgrm. per grm. of material), while the steam-treated material failed to show any trace of the acid. The effect of steam on the whole seed was the same as with the ground material. Steam treatment may thus prove a suitable way of dealing with the enzyme if condensation on a large scale in contact with the material can be avoided.

The power of the enzyme to resist the effect of dry heat is very considerable indeed. Whilst half an hour's steaming is practically fatal to the enzyme, the ground material dried for half an hour at 100° C. evolved in contact with water at 35° C. 1.5 mgrms. of hydrocyanic acid per grm. of the material as compared with 1.62 mgrms. normally. Similar treatment for longer periods showed that dry heat, though slow in its action, eventually destroys the enzyme (see Table II.).

In deciding on the most convenient method of destroying the enzyme on the manufacturing scale the claims of dry heat should be considered on account of its simplicity and practicability. The process is already adopted in connection with the manufacture of linseed cake.

Time.	Temp. of hydrolysis=35° C.				Temp. of hydrolysis=25° C.	
	Mgm. HCN from 1 grm. ground material dried at 100° C. for				Mgm. HCN from 2 grms. ground material dried at 100° C. for 12 hours.	Mgm. HCN from 1 grm. of whole seed* dried at 100° C for 12 hours.
	$\frac{1}{2}$ hour.	1 hour.	2 hours.	7 $\frac{1}{2}$ hours.		
$\frac{1}{2}$ hour	0.11	0.03	0	0	0	0
1 $\frac{1}{2}$..	0.34	0.29	0.16	0.09	0	0.1
3 $\frac{1}{2}$..	0.65	0.59	0.38	0.16	0	0.28
5 $\frac{1}{2}$..	0.86	0.87	0.68	0.28	0	0.44
7 $\frac{1}{2}$..	1.05	1.03	0.81	—	0	—
5 $\frac{1}{2}$..	1.15	1.10	0.89	—	0	—
6 $\frac{1}{2}$..	—	—	—	—	0	—

* Crushed before examination for HCN.

Enzymic response to acidity.

The behaviour of the enzyme in the presence of acid is somewhat remarkable. Whilst $N/1000$ hydrochloric acid at 35° C. liberated practically as much hydrocyanic acid as water, the use of $N/500$ acid resulted in the evolution of at least 73% more hydrocyanic acid than with water at the same temperature. At 25° C. volumetric estimation indicated that 5.74 mgrms. of hydrocyanic acid was liberated from 1 grm. of material, as compared with 3.982 mgrms. when water was the medium of hydrolysis. It thus appears that a certain degree of acidity, probably depending on the normal extent of ionic

dissociation of the acid, is stimulative to the activity of the enzyme. In this connection it may be noted that H. E. Armstrong* found that carbonic acid up to a certain point had an accelerative influence on the activity of urease, which he attributes to the function of the acid neutralising the basic product of hydrolysis (ammonia) which would otherwise tend to combine with the carboxyl of the "agent" radical of the enzyme and thus destroy its power of hydrolysis. This explanation is not applicable to the present case. I am inclined to think that the concentration of hydrogen ions is the factor which governs the behaviour of the enzyme in this series of experiments. The very slight ionic dissociation which characterises water in the normal state does not perhaps supply the concentration of hydrogen ions needed for the maximum activity of the enzyme possible under the other experimental conditions, such as temperature etc. The optimum hydrogen-ion concentration for the enzyme of *Schleichera* seeds is supplied by $N/500$ hydrochloric acid, or thereabouts.

Effect of fat solvents on the enzyme.

The ground material was treated with anhydrous solvents and the extracted material examined in the usual way at 35° and at 25° C. In all cases the evolution of hydrocyanic acid was either nil or so low as to be almost negligible. 3 grms. of material percolated with ether for three hours, when examined at 35° C. evolved no acid in one case and only 0.16 mgrm. in another. In another experiment conducted at 25° C. with the same quantity of material after four hours' ether extraction, 0.29 mgrm. of acid was evolved. Extracted material after exposure to the atmosphere for 12 days failed to evolve any acid. Experiments have indicated that it is the enzyme and not the glucoside that suffers from the action of ether. Petroleum spirit has a similar inhibitory power. The solvent used had a very low b. pt., and it is probable that the vapour of commercial petroleum spirit would be even more inhibitory. In one experiment 2 grms. of material extracted with petroleum spirit for 2½ hours failed to give off any hydrocyanic acid, and in another 3 grms. of material extracted for three hours evolved 0.57 mgrm. (temperature 25° C.). Carbon bisulphide and carbon tetrachloride have a similar effect on the enzyme.

The above observations indicate that by the use of any of the common fat solvents and by allowing the solvent to act on the enzyme for a sufficiently long time, the safety of the residue as a cattle food can be practically guaranteed, provided, of course, no other food taken simultaneously contains an enzyme capable of hydrolysing the glucoside. The cyanogenetic hydrolysis of the glucoside by enzymes secreted in the animal body is highly improbable. Pure amygdalin fed to animals has been found to be without any deleterious effect.

Other antiseptics, including phenol, salicylic acid, hydrogen peroxide, mercuric chloride, and bleaching powder, are all more or less effective in inhibiting the action of the enzyme. Mercuric chloride is the most effective.

A few experiments have been carried out to determine the influence of a number of commonly employed cattle foods, such as grass, hay, roots, and dried silage. A temperature of 39° C. was maintained throughout these experiments. The proportions of the ground seed and of the food were determined with reference to rations in vogue. The evolution of hydrocyanic acid was greatest in presence of silage and least with grass. Silage increased the activity of the enzyme by more than 100% and hay by 33%. In presence of roots the evolution is slightly higher than normally, whilst grass depresses the activity by about 30%. The increased action

with silage and with hay is the result of the acidity of the material.

Estimation of a reducing sugar due to hydrolysis of the glucoside.

The seed is remarkably deficient in reducing sugars, but the water in which the hydrolysis was carried out contained an appreciable amount of a reducing sugar (probably dextrose). Experiments indicated an approximate ratio of 1 HCN:3½ dextrose, showing that the principal glucoside has a high nitrogen content.

The enzyme.

The precipitated enzyme material, dried and dissolved in water, was without activity towards either amygdalin or the glucoside in the seed in which the enzyme had previously been destroyed. The aqueous extract of the seed, without alcohol, is capable of hydrolysing both. The activity of the aqueous extract was vigorous during the first two days, after which it began to decrease and became nil in the course of a week. The fact that amygdalin was decomposed by the enzyme of the seed indicates the presence of an emulsin-like body. Although it has not been possible to discover directly any other products of hydrolysis than hydrocyanic acid and dextrose, it should be mentioned that a faint, but unmistakable, smell of benzaldehyde was developed in the seed when a fairly large quantity of it was submitted to very high pressure, but as the quantity produced is very small it is probable that there are at least two glucosides in the seed, and that the glucoside responsible for benzaldehyde is present in a very small proportion.

Examination of the fat.

Schleichera fat has already been examined by Wijs, Itallie, Glenk, Lewkowitsch, and others. The fat is known by several names, e.g., Kon oil, Kusum oil, Macassar oil, etc.; it has only attained commercial importance for the manufacture of hair oil.

The freshly extracted material is a solid, which begins to liquefy at a comparatively low temperature. It possesses a pleasant smell and is yellowish-white in colour, which changes into dark brown on prolonged heating accompanied by a marked change of odour. The following results were obtained:—Sp. gr. $99^{\circ}/15^{\circ}$ C., 0.86. M. pt., initial 21° C., complete transparency 31° C. Solidifying pt., 29° — 18° C. Saponification value: healthy seeds (a) 229.1, (b) 214.4; mixture of healthy and diseased seeds, 207.6. Iodine values 52.1, 55, 54.6 respectively. Refractive index, at 21° C., 1.46757; at 27° , 1.46655; at 31.5° , 1.4646; at 45° C., 1.4636. Acid value: fat from healthy seeds 13.06; after storing three months 16.28; fat from diseased seeds 109.6.

My best thanks are due to the Director of the Imperial Institute, and to Dr. T. A. Henry, for their kindness in supplying me with the material of the research, and to Mr. S. H. Collins, of the Agricultural Department, of Armstrong College, who has given me the benefit of his experience and allowed me to use his apparatus.

Communications.

A PRELIMINARY ACCOUNT OF THE CHEMISTRY OF THE NILE SUDD.

BY A. F. JOSEPH, D.S.C., F.I.C., AND F. J. MARTIN, M.A.

One of the chief obstacles to navigation in the upper reaches of the Nile and its tributaries is the blockage caused by masses of vegetable matter, locally termed "sudd," an Arabic word meaning blockage or dam. This is composed of fixed and

* Roy. Soc. Proc, 1913., B, 86, 328.

swimming plants, which vary from place to place. Of the many species that have been identified the most important are a papyrus (*Cyperus papyrus*), which grows to a considerable height, often exceeding 15 ft.; a grass (*Panicum pyramidale*), known locally as "Um Soof," a name meaning "Mother of Wool," given on account of the peculiar hairy character of its leaf sheath; and a convolvulus (*Ipomoea aquatica*). "Um Soof" has recently been the subject of an investigation on account of a dermatitis caused when its fine hairs come in contact with the skin, and which appeared to be produced mechanically and not by an actively irritant constituent.

As stated above, the sudd is a great impediment to river transport. From the Zeraf mouth, 525 miles above Khartoum, to Shambe, a distance of 300 miles, a passage through the main channel is at times only kept clear with difficulty, whilst all the tributaries in this region are blocked for many miles.

The vast area of the sudd is, of course, only known very roughly; experienced observers, however, estimate the denser portion at not less than 5000 square miles—150 miles long and 30–40 miles wide—in extent. It is obviously of the greatest importance to consider the possibility of utilising such a gigantic store of material.

Possible utilisation of Sudd material.

Various proposals have been made in recent years to utilise the material of the sudd as fuel. The amount available for this purpose has been estimated at 45 tons of air-dried papyrus per acre, from experiments carried out on the spot some years ago. Thus there is a store of 144 million tons in the area under consideration.

The calorific value of dried papyrus has been found to be about 2000 calories per gm., against 8500 for bituminous coal; the 144 million tons may therefore be put thermally equivalent to 28 million tons of coal.

In terms of energy the 144 million tons should, if burnt, be capable of furnishing 3·6 million horse-power continually, on the assumption that 10 lb. of the material is required for the generation of one horse-power hour against 1½ to 2 lb. of coal. This amount of power is about three-fourths of the theoretical average power of the Niagara Falls, and is an enormous asset.

Fuel is only one of many ways which have been suggested for the use of the sudd material; others, such as paper pulp, rope fibre, fertiliser (from the ash), and distillation products are amongst the possibilities of the future, and a detailed chemical examination of the subject is obviously desirable.

References.

Very few references to the sudd are to be found in chemical literature. Cross and Bevan ("Paper Making," p. 161) refer to papyrus as a possible source of material for fine papers of the esparto type, and an experimental treatment of the same subject by the late Dr. W. Beam forms the subject of Bulletin No. 2, 1915 (Chemical Section) of the Wellcome Tropical Research Laboratories (see this J., 1915, 792). Cresswell (this J., 1915, 389) refers to the sudd as a possible source of potash, and it will be seen that this is fully borne out from the analysis recorded below.

Proximate analysis.

Two samples of papyrus were examined, the first (a) collected some years ago, and therefore subjected to a long period of air drying, and the second (b) in September, 1919, the analysis being carried out in October. The Um Soof was obtained about the same time as (b). Arrangements are being made to obtain other samples collected at

different times of the year, as seasonal variations in the amount and composition of the ash may occur (compare Purvis, Proc. Camb. Phil. Soc., 19, 261; also Chemical Abstracts, 1918, p. 1737).

The analysis was confined to the determination of groups of compounds rather than of individuals (compare Dore, J. Ind. Eng. Chem., 1919, 11, 536). The following were the determinations made:—(a) *Moisture*, determined by heating in the steam oven at about 97°C. for four hours. (b) *Mineral constituents*.—Ash was determined by ignition in a platinum basin over a spirit Bunsen burner for about two hours. The volatility of potassium chloride under these conditions is unfortunately sufficiently great to render the results somewhat uncertain. (c) *Fatty and waxy matter*.—The dried material was extracted for about 10 hours in a Soxhlet extractor with a mixture of alcohol and benzene in equal proportions. (d) *Soluble proteins*.—The dried material was treated with water at room temperature for 24 hours, the mixture filtered, and the filtrate evaporated to dryness on a water bath (soluble protein = N in residue × 6·25). (e) *Insoluble protein* was determined by subtracting (d) from the total protein. (f) *Pentosans*.—The dried material was boiled with 12% hydrochloric acid in a flask connected with a condenser, and the furfural estimated in the distillate by means of phloroglucinol in the usual way. (g) *Soluble carbohydrates*.—The water-soluble portion was assumed to consist of protein, carbohydrates, and mineral matter. The first and last of these being determined, the amount of the soluble carbohydrates was obtained by difference. (h) *Fibre*.—The material was boiled with 1·25% sulphuric acid for half an hour, the mixture filtered and washed, and the residue boiled with 1·25% caustic soda for half an hour. The undissolved material dried at 110° C. was weighed, ignited, and the weight of the ash subtracted from the weight of the residue dried at 110°, was taken as fibre.

Methods (f) and (h) are those of the Association of Official Agricultural Chemists (U.S. Dept. of Agriculture, Bureau of Chemistry, Bul. 107).

Stems of papyrus and Um Soof were used for the proximate analysis. The former consists of long, straight rods of triangular section varying from ½ in. to 1½ in. along the edge. It is exceedingly light, the apparent specific gravity of pieces cut from different stems being 0·174 (mean of six determinations). When reduced to pieces about 1 c.c. in size the "bulk density" of the uncompressed material is only 0·1, i.e., about 300 cub. ft. to the ton.

The analytical results obtained were as follows:—

	Papyrus stems	Um Soof stems
(a) Moisture	5·5	5·0
(b) Mineral constituents	6·0	8·6
(c) Fatty and waxy matter	9·4	5·2
(d) Soluble proteins	0·8	3·6
(e) Insoluble proteins	0·8	1·1
(f) Pentosans	24·2	28·2
(g) Soluble carbohydrates	7·9	6·6
(h) Fibre	40·4	38·8
Total	95·0	97·1

Dore (*loc. cit.*) found a similar deficiency to that shown above in his analysis of five different kinds of American wood. This may be due to neutral volatile substances which are lost during hydrolytic changes.

The mineral matter is dealt with in detail below. The total protein figures are between those for woods and grasses; the papyrus, being more of a ligneous nature than Um Soof, contains, as would be expected, the smaller proportion.

The figures for fibre may be of practical importance if the material is to be used for paper pulp manufacture. In Dr. Beam's experiments on this

subject, previously referred to, it was found that erude pulp or "half stuff" was readily obtained by heating papyrus stems (either green or dry) under pressure to about 150° C. for a few hours with water alone, no caustic soda or other chemicals being required. As Beam pointed out, the advantage of such a method would be very great in a region so far removed from chemical supplies and where recovery of waste products would be an additional difficulty. Beam found that the air-dried papyrus yielded about 40% of dry "half stuff" and about 20% of dry bleached pulp; the former figure is in close agreement with the value for "fibre" recorded by us.

Mineral constituents.

The following are the most important of the determinations made:—

Composition of the entire ash.

Percentage.	Papyrus A Flower head	Papyrus A stems	Papyrus B stems	Um Soof stems
Total ash from air-dried material.	11.6	4.66	6.00	8.56
Calculated on the ash				
K ₂ O ..	10.50	34.50	35.27	29.62
Na ₂ O ..	0.52	N.D.*	8.07	1.90
CaO—MgO ..	3.54	N.D.	3.12	4.85
Fe ₂ O ₃ —MnO ..	3.04	3.80	1.44	1.71
SiO ₂ ..	59.01	36.36	32.58	50.84
CO ₂ ..	1.42	N.D.*	4.27	—
Cl ..	1.70	10.43	16.60	12.05
SO ₂ ..	Trace	Trace	Trace	Trace
P ₂ O ₅ ..	5.04	3.92	2.35	3.64
C ..	9.55	—	—	—
Deduct oxygen equivalent to chlorine	0.39	2.35	3.72	2.72
Total ..	99.93	—	99.98	101.89

*Not determined.

The most important of the above results is the high potash content of the stems; at the same time the amount of chlorine indicates that a considerable proportion is soluble in water. The following table gives the complete analysis of the water-soluble portion of papyrus stems, sample B:—

The water-soluble portion had the following composition (calculated on the total ash): Total water-soluble salts 57.20%, K₂O 29.72, Na₂O 5.59, SiO₂ 8.00, CO₂ 3.78, Cl 11.76%; total, 61.85; deduct oxygen equivalent to chlorine 3.32% = 58.53%. The following composition for the water-soluble portion would agree with the above figures: NaCl 10.53, KCl 17.61, K₂CO₃ 11.88, K₂SiO₃ 17.27%; total, 57.29%. Such a product containing 39% of potassium (or over 50% as K₂O) might well be the basis of a very valuable fertiliser, and it is hoped to carry out some field experiments to test this next season. At the same time the whole ash (containing 35% K₂O) will be examined for manurial value.

Action of water.

The action of water on dried papyrus has been examined at ordinary and elevated temperatures and pressures.

In the first series of experiments the air-dried material was treated with cold water (about 30° C.) for 24 hours, the filtered solution evaporated to dryness, and the extract dried in the steam oven.

The following results were obtained:—

Percentages calculated on the original material.			
	Papyrus B		Um Soof
	%	%	%
Dried extract	9.0	12.0
Mineral matter	2.6	2.0
Ignition	2.3	—
soluble in water after

A 20-grm. portion of the material was heated in the water bath with 500 c.c. of water, and the filtered extract evaporated to dryness. The undissolved material was heated in the autoclave with water for four hours at four atmospheres pressure

(150° C.), and the total extract and the mineral matter again determined.

In one experiment an attempt was made to retain the volatile products of hydrolysis from the autoclave. A small amount of carbon dioxide was evolved. The distillate was titrated against standard alkali and the results expressed in terms of acetic acid. The extract in the autoclave was also examined for acid.

The results given below are for the extraction by hot water at ordinary pressure, followed by four hours in the autoclave. All the results are percentages calculated on the original material.

The figures show that water extracts about one quarter of the material, including about four-fifths of the mineral constituents. Samples A and B gave substantially the same results.

In neither experiment is 100% of the material accounted for, the deficiencies being 16.3 and 7.6% respectively.

	Dried extract		Mineral matter		Mineral matter soluble in water after ignition	
	A %	B %	A %	B %	A %	B %
On the water-bath ..	9.66	9.39	3.21	3.82	2.75	3.25
Further material extracted in the autoclave ..	13.79	16.56	1.52	1.56	0.68	0.63
Total extracts ..	23.65	26.95	4.73	5.58	3.43	3.83
Residue in the autoclave ..	60.00	65.5	1.50	0.79	0.22	0.11
Total mineral matter extracted ..	—	—	6.23	6.35	3.65	3.99

An additional experiment was carried out in which 20 grms. of sample B was heated in the autoclave with water under a pressure of about four atmospheres, the valve being connected to an absorption apparatus. After two hours the heating was interrupted, the contents of the autoclave filtered, and determinations made of the acidity of the extract and of the distillate, and the weight, protein content, and ash of the extract. The residue left after filtration was then returned to the autoclave and the heating with water continued for another two hours, after which the same determinations were made. After a third heating with water, followed by the same analytical determinations, the residue was dried at 100° C., weighed, and the protein and ash determined in it. The collected results, expressed as percentages on the original material, are given below:—

Percentage	First 2 hours.	Second 2 hours.	Third 2 hours.	Total
Volatile matter absorbed in the tube ..	0.185	0.100	0.145	0.33
Acid in distillate as acetic acid ..	0.245	0.220	0.060	0.52
Acid in extract ..	0.765	0.810	0.415	1.99
Protein in extract ..	0.61	0.42	0.18	1.21
Mineral matter in extract ..	4.01	1.11	0.43	5.55
Soluble carbohydrate in extract ..	11.93	10.34	2.68	24.95
Total material removed ..	17.74	13.00	4.53	35.27

The weight of the residue (11.3 grms.) was 56.5% of the original material taken; 35.3% is accounted for as above, so that there is 8.2% not identified. The residue contained 0.81% of protein and 0.07% of mineral matter calculated on the original material; the autoclave treatment therefore removed rather more than half the former and practically the whole of the latter, in three successive heatings. The first four hours extracted four-fifths of the total material dissolved.

Conclusions.

(a) Examination of papyrus and Um Soof—two of the most important constituents of the Nile sudd indicates that these two plants may be of economic importance in paper pulp manufacture and as a source of potash.

(b) The ash of papyrus stems contains more potash than that of Um Soof, but this is more than

balanced by the higher proportion of ash yielded by Um Soof. Calculated on the dry plant, papyrus stems contain 21 and Um Soof stems 23% of potassium oxide.

(c) Papyrus stems are readily acted on by hot water, more especially under a pressure of about four atmospheres. Treated in this way, practically all the mineral matter and most of the protein passes into solution, and the residue constitutes a satisfactory "half stuff" for paper.

It is hoped that the work will be continued in order to complete the above preliminary account of the chemistry of this important material.

Our best thanks are due to Mr. F. J. Tear, of the Sudan Forests Department, who kindly supplied the samples of material, and to Mr. G. W. Grabham, Government geologist, for much valuable information as to the sudd.

Wellcome Tropical Research Laboratories.
Khartoum.

NOTE ON AN EXPLOSION DURING THE SEPARATION OF POTASSIUM BY THE PERCHLORATE METHOD.

BY A. F. JOSEPH, D.SC., F.I.C., AND F. J. MARTIN, M.A.

In the course of the examination of the Nile sudd (*cf. ante*) it was necessary to determine the total amount of sodium in papyrus ash. The sodium was brought into solution by fusion with potassium carbonate and extracting with water, the solution then containing potassium in excess, sodium, phosphates, and silica. After removal of the latter in the usual way, the phosphate was precipitated by ferric chloride and ammonium acetate, the filtrate evaporated to dryness, and the residue ignited to expel ammonium salts. The potassium was then

separated as perchlorate and the sodium estimated directly in the filtrate, as any method of determining it by difference appeared to be subject to too great an error, owing to the relatively large amount of potassium present.

The process was carried out as follows: After the removal of the silica the solution was evaporated two or three times with a slight excess of perchloric acid and the potassium perchlorate separated in the usual way. The filtrate and the alcohol washings, containing the sodium and excess of perchloric acid, were evaporated in a beaker nearly to dryness, and then transferred to a porcelain crucible, the evaporation being finished on an electrically heated hot-plate. The residue was treated with sulphuric acid and the sodium finally weighed as sulphate. A number of estimations in this manner were made without mishap. On the last occasion, however, a violent explosion occurred during the evaporation on the hot plate, when only 1 or 2 c.c. of liquid remained in the crucible. The force of the explosion produced a shallow cavity in the heavy top of the iron hot-plate over $\frac{1}{4}$ in. deep, the crucible being completely shattered and splinters driven with considerable force to a distance of 30 or 40 ft. Some of these splinters actually punctured bottles, flasks, and windows several yards away with holes about 1/10 in. diameter. Vessels close to the explosion were of course completely shattered.

The only cause we can suggest for this explosion is that some organic matter derived from the alcohol was not removed in the first part of the evaporation, and remained until the perchloric acid was concentrated. We have for the time being discontinued the use of this method.

Wellcome Tropical Research Laboratories,
Khartoum.

Birmingham Section.

Meeting held at Birmingham University on Thursday, February 12, 1920.

DR. H. W. BROWNSDON IS THE CHAIR.

CATALYSIS FROM AN INDUSTRIAL STAND-POINT.

BY E. B. MAXTED, PH.D., D.S.C.

THE discovery of new catalytic processes and of suitable catalysts has necessarily been made by the purely empirical method of trial and observation, except in so far as the choice of a probable catalyst was guided by analogy with known reactions. And the same is true regarding the study of catalyst poisoning, which, from an industrial standpoint, is almost of equal importance to that of catalysis itself.

While, however, the fruits of this purely experimental study are indisputably great, the systematic and intensified development of catalysis in the future depends on a better understanding of the theoretical principles which underlie the mechanism of the various types of catalytic processes.

In some cases, where catalysis appears to proceed by the purely chemical participation of the catalyst in the reaction, it is possible to carry out the process in stages, in such a way as to obtain a clear insight into what takes place under conditions such that the intermediate products are incapable of stable existence and the catalysed reaction consequently proceeds to completion, as, for instance, in the case of oxides of nitrogen in the chamber process for the manufacture of sulphuric acid, or the catalytic production of hydrogen by the interaction of water-gas with steam in the presence of iron. Whether this latter reaction takes place catalytically in one stage or by alternate oxidation and reduction, the contact mass emerges from the reaction in the same condition chemically as in the commencement, but fundamentally altered in physical structure.

In other cases, such as where the above reaction is catalysed by means of platinum, the rôle played by the catalyst is not so easily investigated by resolution into a series of changes and the effect observed may possibly be attributed to effects of a physical nature, especially since the reaction may also be catalysed to a certain degree by the mere presence of porous bodies, such as unglazed porcelain.

Thus a general theory of catalysis, which shall apply universally to every type of reaction is probably impossible, and each reaction, and, indeed, each catalyst employed for a given reaction, must receive special consideration with the object of setting up certain types of accelerating mechanism, for each of which separate theoretical principles will be involved. This is the necessary preliminary to the prediction of the degree of activity of a catalyst for a required reaction at a given temperature. A welcome step towards this goal has recently been made by Lewis in the application of the quantum theory of radiation to catalysis in general.

Catalysis in heterogeneous systems can only proceed provided that the required preliminary association of the catalyst with the reacting system can take place freely. Suspension of such free association will result in an effect similar to that observed in the presence of a catalyst poison, e.g., in the hydrogenation of an unsaturated glyceride with palladium at a temperature sufficiently low for the stearine produced to remain in a solid form on the surface of the palladium and thus gradually to

obstruct the free participation of the catalyst in the chemical change required, a "poisoned" curve being obtained.

A slightly more complicated example of the same type of inhibition is to be found in cases where the preliminary occlusion of one or more of the reacting substances is necessary before catalysis can proceed. Thus, in catalytic hydrogenation all the catalysts usually employed occlude hydrogen to a greater or smaller extent, and, with any given catalyst, catalytic activity and occlusive power appear to run more or less hand in hand, for catalyst poisons, such as hydrogen sulphide, not only destroy the activity of palladium but also its power of occluding hydrogen.

While the catalytic activity of the various metals does not vary directly as their occlusive power for hydrogen, what is ordinarily understood as occlusive power refers to the power of absorption at room temperature. As far as can be seen, no work has yet been carried out on the quantitative relation between the true activity of metals and the content of occluded hydrogen. If the function of the occluding metal is merely to render hydrogen available in the occluded condition, the catalytic activity of the preparation should, *ceteris paribus*, be proportional to the hydrogen content. If this is not so, the occluding metal itself, in addition to reducing the hydrogen to an active state, must itself take a part in the series of changes involved. Suspension of the occluding power should thus entail a suspension of catalytic activity and *vice versa*.

Paal and his collaborators have shown that palladium or platinum precipitated on metals such as lead, aluminium, silver, etc., is inactive. Mercury, which also acts as a catalyst poison, actually caused the evolution of hydrogen when brought into contact with palladium charged with this gas.

The poisoning of a catalyst for a desired reaction may be also caused by the presence of impurities such that the possibility exists of a second reaction, which may then take place in preference to that required. An example of this is the inhibitive effect of the presence of sulphur vapour in sulphur dioxide undergoing oxidation to trioxide in presence of platinum, oxidation of the sulphur taking place with partial inhibition of the reaction between sulphur dioxide and oxygen. It is, in general, typical of poisoning effects due to preferential reactions that the catalyst regains its normal activity on being subsequently used for pure reactants.

On thermodynamical grounds it may be shown that if the equilibrium point of a reverse reaction is independent of the presence or absence of catalyst, the activity of the catalyst for a given reaction must be the same as for the reaction when carried out in the reversed sense. Thus iron, which is active for the synthesis of ammonia, is also active for its decomposition, while nickel or palladium may be employed both for the synthesis of hexahydrobenzene from benzene and hydrogen or for the decomposition of hexahydrobenzene into its constituents. In the same way the principle may be extended to promoters and to poisons. For example, when ammonia was passed through an iron tube activated with potash the decomposition was four times as great as with the same tube unactivated. Similarly, on passing ammonia containing hydrogen sulphide through a similar unactivated tube, only about one-fifth as much decomposition was obtained as with pure gas. It does not follow, where more than one reaction can proceed simultaneously from the same reactants, that each of these reactions shall be equally accelerated by the presence of a given catalyst; indeed, one of the most important uses of catalysis in industry is for the preferential acceleration of what may be under

non-catalysed conditions the less important of two parallel reactions.

A typical example of this is given by Sabatier and Mailhe's work on the decomposition of formic acid in the presence of various contact bodies, these being grouped as dehydrogenation or dehydration catalysts respectively, according as the decomposition is represented by $\text{H.COOH} = \text{H}_2 + \text{CO}_2$, or by $\text{H.COOH} = \text{CO} + \text{H}_2\text{O}$. Copper or nickel, and the oxides of metals like zinc or tin, were found to give rise to hydrogen and carbon dioxide, while with, for instance, titanium dioxide decomposition into water and carbon monoxide occurred.

The catalysts may be grouped in such a way as to summarise their activating properties and the conditions under which these are exhibited. Such a tabulation is the only way in which it is at present possible to predict to some degree the most suitable catalyst for a required reaction.

The first great group comprises those substances which possess the power of activating hydrogen and are consequently of service both for catalytic hydrogenation and dehydrogenation. The temperature and the nature of the reaction will determine which catalyst is likely to be most suitable.

The platinum metals, generally, activate hydrogen readily at the ordinary temperature, and may be used for reactions in solvents where the temperature cannot be raised to that required for the base metal hydrogenation catalysts, this low temperature being also conducive to the absence of by-products. Thus, while the hydrogenation of benzene according to the original Sabatier method is always accompanied by considerable decomposition, the same reaction proceeds in presence of palladium or platinum at lower temperatures without any undesired secondary effects. Nickel does not usually begin to exhibit activity until well above 100°C ., but between about 150° and 350° it is a cheap and active catalyst for many reactions, e.g., the hardening of oils. Cobalt requires a higher temperature than nickel and is also less active, while with iron these two factors are even more pronounced than with cobalt. Iron is indeed specially useful for reactions at a comparatively high temperature, for instance 500° – 600°C ., at which it is often more efficacious than nickel, the activity of which is to a large degree destroyed by sintering when exposed to temperatures much over 400° . The successful use of iron for reactions such as the synthesis of ammonia is probably due to the fact that it activates nitrogen as well as hydrogen. Copper is a somewhat less violent hydrogenation catalyst than nickel. For example, Sabatier found that in hydrogenating styrolene with copper as the contact body the side chain only was reduced with production of ethylbenzene, whilst with nickel the benzene nucleus also underwent hydrogenation, ethylhexahydrobenzene resulting. Again, normal amylacetylene $\text{CH}_3(\text{CH}_2)_4\text{C}:\text{CH}$ with copper gave normal amylethylene, while with nickel heptane was produced.

With all the above base metal catalysts the temperature of preparation (usually by the reduction of their oxides by hydrogen) plays a great part in determining their activity, it being found that, above a certain minimum temperature, a low reduction temperature is conducive to high activity. All these catalysts probably depend on initial occlusion of hydrogen for their action, possibly followed by the chemical participation of the catalyst in the reaction, poisoning taking place usually either by the inhibition of this occlusive power or by reason of a preferred reaction.

The oxygen-activating catalysts are concerned with many important industrial operations, such as the oxidation of sulphur dioxide to trioxide and of ammonia to nitric oxide, the use of siccatives for drying oils, and the induction of surface combustion. Two distinct classes appear to exist, the first,

typified by porous porcelain and possibly by platinum, operating probably by physical means, at any rate as a preliminary; the second, catalysts which, as far as can be seen, operate by cyclic changes.

Of the oxidation catalysts of the first class platinum is probably the most generally applicable, being employed *inter alia* for the oxidation of sulphur dioxide to trioxide, of ammonia to nitric oxide, and, generally, for the catalytic oxidation of organic compounds of the most varied nature in the Dennstedt method of elementary analysis. It has been shown that the heat of occlusion of oxygen by platinum is of about the same magnitude as the heat of formation of platinum monoxide, but no proof exists that this oxide is an intermediate product during catalytic oxidation.

The second class of oxidation catalysts consists almost exclusively of substances which possess the property of forming higher oxides, which, in order that the catalysed reaction may proceed to completion, must, if they are formed as intermediate products, be unstable under the conditions employed for the reaction, these conditions including the presence of the substance to be oxidised. An example of this class of activator is to be found in nickel or cobalt oxide when used to evolve oxygen from bleaching powder. Cobalt forms an oxide CoO_2 , which may be prepared by the action of iodine and an alkali on a cobaltous salt, but which is unstable under the conditions employed for the evolution of oxygen by the reaction in question. Other examples of this class are to be found in manganese, cobalt, nickel, lead, or cerium salts when used for catalytically accelerating the oxidation of linseed and similar oils.

Nitrogen-activating catalysts have increased rapidly in technical importance during the past decade. Iron appears to be the most generally useful catalyst of this nature for reactions between 500° and 1000°C .; for instance, for the synthesis of ammonia and in the Bucher process for the synthesis of alkali cyanides. Iron does not form a nitride directly, although this can readily be obtained by interaction with ammonia. Other examples of nitrogen catalysis, the mechanism of which is not easily understood, are to be found in the acceleration of calcium cyanamide formation by calcium chloride, and the remarkable activity of osmium in causing the union of nitrogen and hydrogen.

The catalytic acceleration of hydrolysis and esterification by means of hydrogen ions is of considerable technical importance. The most probable explanation of the mechanism involved in this case seems to be that recently advanced by Lewis on the basis of the quantum theory of radiation. A case of considerable interest in connection with the hydrolysis of substances insoluble in water is the commercial hydrolysis of glycerides by means of the Twitchell reagent, which is soluble in oils and in water, and at the same time supplies the hydrogen ions necessary for hydrolysis.

A point of considerable interest arises in connection with catalysis generally, namely, the possibility of setting up a standard scale of activities on which it should be possible to express as a number the observed activity of a catalyst for a given reaction. Catalytic reactions in heterogeneous systems, especially gas reactions in the presence of solid catalysts, do not usually conform to the simple kinetic laws which obtain in homogeneous systems, and hence it is not always possible to take, as the "activity number" of the catalyst in question, the quotient formed by the reaction constant over the concentration of the catalyst, or some similar relation under standard conditions.

Similarly, the space-time-yield in heterogeneous gas reactions varies enormously not only with the

activity of the catalyst, but also with the velocity of passage of the reacting gases. If, however, for those heterogeneous gas reactions in which the gas passes over a solid catalyst, we take as the activity number at a given temperature and pressure the hourly output of product corresponding to unit weight of catalyst, with a standard space-velocity of gas flow, chosen in such a way that the time of contact with the catalyst is a convenient unit, a scale can be constructed which should be of considerable utility for the purposes required.

As an instance of the method of calculation the following examples may be cited. In the synthesis of ammonia, published data record a yield of 3.3 kilos. of ammonia per hour per litre of catalyst space, containing about 1.5 kilo. of iron/potash catalyst, the temperature being 580° C., the pressure 150 atm., and the time of contact one second, which for a slow process of the above nature may be taken as a convenient unit.

The activity number of the catalyst under the given conditions is thus: Hourly yield of product ÷ weight of catalyst = $3.3 \div 1.5 = 2.2$.

Where reactions take place at a comparatively high velocity a smaller unit of time than one second must be chosen for the standard time of contact, and there exist also numerous cases where more than one resultant is produced. In the latter case the activity number may be referred to the rate of production of any one of the products as required. For example, the production of hydrogen by the interaction of carbon monoxide and steam takes place with considerably greater velocity than the synthesis of ammonia, and for unit time of contact we may conveniently take 0.1 second. In an experiment to determine the activity number of an iron/soda catalyst at 500° C., carbon monoxide and steam in stoichiometric proportions were led through a 5 c.c. catalyst space, containing about 10 grms. of the iron/soda catalyst, at a rate calculated to give unit time of contact at 500° C. Hydrogen was found to be produced at the rate of 3.8 litres per hour, corresponding to 0.34 gm. The activity number of the catalyst, referred to hydrogen, under the conditions employed is thus:—

$A_{t=50} = \text{Hourly production} \div \text{weight of catalyst} = 0.34 \div 10 = 0.034$.

Finally, for reactions which proceed at extremely high velocities, such as the oxidation of sulphur dioxide to trioxide in presence of platinum, the standard time of contact may conveniently be reduced, for instance, to 0.01 second. Thus, on placing 0.48 c.c. of platinised asbestos, containing about 0.005 gm. of platinum, in the measuring apparatus employed for the last determination and passing sulphur dioxide and oxygen, in stoichiometric proportions, at 425° C., at a rate calculated to give a time of contact of 0.01 second, the rate of production of sulphur trioxide was found to be 27 grms. per hour. Calculating as before,

$A_{t=425} = 27 \div 0.005 = 5400$.

It is obvious that the activity number refers to the actual effect produced, and will vary considerably with the physical form in which the catalyst is employed. Further, for reactions of types other than those considered above, the same ratio between the weight of product formed per hour and that of the catalyst inducing the change, all under suitable standard conditions, may be taken as the activity number of the catalyst in question for the given reaction at the temperature employed.

DISCUSSION.

The CHAIRMAN said that the reduction of spelter from zinc oxide was a difficult reaction requiring much energy in the form of heat, and it was possible that the reaction might be cheapened by the use of a suitable catalyst.

Mr. L. P. WILSON said that catalysts had, as a rule, been employed in the production of cellulose

acetate for dope. Numerous catalysts had been suggested, including sulphuric acid, zinc chloride, bromine, phosphorus oxychloride, hydrazine, and sulphuryl chloride.

Prof. G. T. MORGAN agreed with the author that there was at present no possibility of any general theory of catalysis. The subject was much too complicated for a general theory to eventuate in the immediate future. The hypothesis advocated by van't Hoff and others that a catalyst was an agent which accelerated the velocity of the inverse change to the same extent as that of the direct change was, he thought, far from being generally proven. There were many cases in which this principle could hardly operate. Sabatier had stated that the hypothesis which had inspired him in his classical researches was that catalysis involved the formation of some intermediate compound. The speaker thought that this opinion still held the field in the majority of cases. It might be argued that it was improbable that intermediate compounds would be formed if, under the conditions of the reaction, they would be unstable; but that could hardly be postulated generally with regard to the transient existence of these substances.

The alkali metals with a large atomic volume were not so effective in certain catalytic operations as metals like platinum and copper having a small atomic volume, a circumstance which seemed to imply the intervention of residual affinity. A dilemma, which arose in regard to the intermediate compound when one considered the catalytic effect of porous materials, was the difficulty of determining where chemical action began and physical action left off. Strenuous efforts had been made recently to obtain a continuous process for making aniline catalytically from nitrobenzene and hydrogen, and extremely complex catalysts had been described. These facts tended to show that chemists in search of industrial catalysts still proceeded by a method of trial and error and not on any general hypothesis of catalysis.

Dr. T. SLATER PRICE said that many catalysts had been discovered purely by accident, as in the case of oxidation of naphthalene to phthalic acid; but in recent years undoubtedly there had been a tendency towards the opinion that catalysts in certain reactions should first be sought among metals which exerted different valencies. In connection with catalyses in homogeneous systems he thought there had been very definite progress in connection with the theory of the formation of an intermediate compound. It had been shown in quite a number of cases that this intermediate compound did exist and played the part which was assigned to it. Very often, because equations could be written assigning a definite rôle to an intermediate compound, it was supposed that an explanation of the catalytic effect had been given. But it was pointed out that such explanations were not valid until it could be shown that the velocity of the complete reaction was equal to the sum of velocities of the intermediate reactions postulated. That had been shown to be the case in a few instances and led to the belief in the general explanation. The question was raised as to the effect of a catalyst on the equilibrium attained, and Dr. Maxted referred to the usual thermodynamical proof that the equilibrium should be the same in the presence as in the absence of a catalyst. That had been proved experimentally in a number of cases where homogeneous systems had been investigated. The question, however, had been brought to the front again by Prof. Lewis, who had recently shown theoretically, by the application of the quantum theory, that it was possible for the equilibrium to be disturbed by the presence of the catalyst. Prof. Morgan had mentioned the probable connection between small atomic volumes and occlusive power. That did not agree, however, with the effect of sodium as a promoter in the

catalytic action of iron. The catalytic action of light had played an important part in the production of war chemicals, for example, in the manufacture of diphosgene (trichloromethyl chloroformate).

Dr. D. F. TWISS remarked that although there was a possibly natural tendency to propound various general theories as to the seat of activity in all catalytic substances, such generalisations would not bear close examination, and, at the present stage of investigation of the subject, were not particularly helpful.

Mr. F. R. O'SHAUGHNESSY endorsed the view expressed by Dr. Twiss, and pointed out that whereas the catalysts under discussion were inorganic bodies, active mostly at very high temperatures, the organic catalysts known as enzymes acted at low temperatures, and transformed masses of material under these conditions.

Mr. F. H. ALCOCK mentioned the case of the conversion of yellow phosphorus into amorphous phosphorus in presence of a trace of iodine.

Dr. MAXTED, in reply, said with regard to the point raised by the chairman, it was possible in some cases to lower reduction temperatures catalytically. For instance, it was possible to reduce copper oxide to copper at the ordinary temperature by the action of palladium; further, mixed oxides of nickel and copper were reduced by hydrogen at a lower temperature than nickel oxide alone. The production of cellulose acetate might not be a purely catalytic reaction, but to a certain extent an absorptive one. If only a little of the accelerating body were used it was catalytic. The evidence as to the atomic condition of hydrogen occluded by metals was based principally on the measurements of Sieverts and others on the relation between the pressure and the solubility of hydrogen. Regarding the time required for the penetration of hydrogen into the catalyst, and into the substance which was undergoing hydrogenation, the absorption was usually not so great quite at the beginning as subsequently. It took a short but appreciable time for the hydrogen to enter the liquid and reach the surface of the catalyst. A falling-off in the activity of platinum following disintegration would not be expected if no poisoning took place; in fact, the disintegration itself might cause an increase in activity by increasing the catalysing surface. It was found that with pure gases under certain conditions the activity of the catalyst might increase after it had been used for a certain time, but that, of course, had nothing to do with disintegration.

THE PREPARATION OF CYANOGEN CHLORIDE ON A LARGE LABORATORY SCALE.

BY T. SLATER PRICE, O.B.E., D.SC., AND STANLEY J. GREEN, M.A., A.I.C.

Examination of the literature on the subject* indicated that three methods might be feasible for preparing large quantities of cyanogen chloride, namely, the action of chlorine upon (a) hydrocyanic acid, (b) sodium cyanide, or (c) the complex cyanide of sodium and zinc.

Action of chlorine on a solution of sodium cyanide.

In this method, first suggested by Langlois,† considerable heat is evolved, and unless adequate cooling is employed the solution soon darkens in colour.

Experiments were also made with a modification proposed by Jennings and Scott (*loc. cit.*), using carbon tetrachloride as diluent, with 5000 grms. of sodium cyanide*; chlorine was introduced at the rate of about 2 litres per minute, and refrigeration approximately kept pace with the heat development of the reaction. A higher rate of passage of chlorine could not be used without a rapid rise in temperature taking place. The time necessary for the chlorination of the 5000 grms. of sodium cyanide would thus be from 19–20 hours, unless the refrigeration could be greatly improved. On a large scale such an improvement would be very costly, especially since the reaction mixture is not homogeneous.

A further point is that the reaction consists partly in the conversion of one solid (sodium cyanide) into another (sodium chloride), so that for quick reaction the sodium cyanide would have to be finely ground and vigorous agitation during chlorination would be required.† Calculations from thermochemical data show that in the production and condensation of one ton of cyanogen chloride the refrigerating plant would have to account for the absorption of at least 2,160,000 C.H.U.‡

Action of chlorine on the complex cyanide of zinc and sodium.

This method was first suggested by Held§ in 1897, the idea being that the addition of zinc sulphate to a solution of potassium cyanide made the solution more stable to the action of chlorine.

In our experiments sodium cyanide was used in place of potassium cyanide, the quantities of sodium cyanide and zinc sulphate taken being such that the compound $2\text{NaCN} \cdot \text{Zn}(\text{CN})_2$ was formed. In some cases to ensure a non-alkaline reaction mixture the mixture was such that a slight excess of zinc cyanide remained in suspension. The method gave fairly good results (compare Mauguin and Simon, *Comptes rend.*, 1919, 169, 383), but in view of the fact that it seemed to be much less economical and convenient than the one about to be described, it was abandoned. Jennings and Scott (*loc. cit.* p. 1244) also report adversely on the method.

Action of chlorine on a dilute solution of hydrocyanic acid.

Cyanogen chloride was first prepared in 1787 by Berthollet|| by the interaction of chlorine and hydrocyanic acid, and this method has since been the subject of many investigations (for literature references, see Jennings and Scott, *loc. cit.*). In all cases, however, the experiments were on a comparatively small scale, so that for our purpose it was necessary to carry out an extended series of trials before a satisfactory method of working was obtained.

The use of anhydrous hydrocyanic acid is impracticable for various reasons. For example, during the reaction hydrogen chloride is formed and polymerises any cyanogen chloride formed to cyanuric chloride. Also, the heat of reaction is so great that the temperature rises rapidly, in spite of vigorous cooling, and unchanged hydrocyanic acid, together with hydrogen chloride and any cyanogen chloride formed, distil over.

Tests showed that comparatively weak hydrochloric acid (about 2N–5N) had very little polymerising

* Jennings and Scott used only 50 grms.

† We are indebted to Prof. N. T. M. Willsmore for some of these criticisms and also for the thermochemical data which are given in other parts of the paper.

‡ 1 C.H.U. (Centigrade Heat Unit) is the quantity of heat necessary to raise 1 lb. through 1° C.

§ Bull. Soc. Chim., 1897, [3], 47, 287.

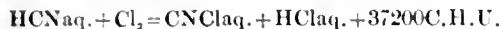
|| Ann. Chim. Phys., 1789 [1], 4, 35.

* Full literature references are given in a paper by Jennings and Scott, J. Amer. Chem. Soc., 1910, 41, 1241.

† Ann. Chim. Phys., 1861, [3], 41, 481.

effect on cyanogen chloride. Experiments were, however, first made with a 20% solution of hydrocyanic acid, the reaction mixture being cooled during the chlorination. It was intended then to separate the cyanogen chloride layer, but it was found that the aqueous layer increases in density, whilst the cyanogen chloride dissolves hydrocyanic acid and water, so that its density is lowered. Consequently the crude cyanogen chloride separates, sometimes above and sometimes below the aqueous layer. If chlorination were carried to completion cyanogen chloride should always separate at the bottom, but it is difficult to chlorinate completely without using excess of chlorine, which would carry much cyanogen chloride with it. All experiments in which a current of gas passed through the apparatus gave very bad yields. It was finally decided to employ a strength of hydrocyanic acid solution, such that at the end of the reaction the concentration of the hydrochloric acid formed is sufficiently below the saturation point to prevent much hydrogen chloride gas passing off: 12–15% hydrocyanic acid was found to be fairly satisfactory.

The reaction vessel was not cooled, the heat of reaction being utilised to distil off the cyanogen chloride as it was formed, so that it was quickly removed from the action of the hydrochloric acid set free at the same time. The heat of reaction is:



Porous pot water filters, suitably bunged and strengthened, and connected by glass tubing with the source of chlorine, were placed in the hydrocyanic acid solution and chlorine forced through under the pressure from a cylinder of liquid chlorine. The chlorine escaped in very fine bubbles and chlorination was extremely rapid.

Cyanogen chloride soon distilled and was condensed in two vessels containing water or dilute hydrocyanic acid (2%) and well cooled by brine. Absorption of chlorine takes place very rapidly, and the current of chlorine is so adjusted that no gas escapes at the end of the apparatus. The speed of chlorination in all experiments is so great that the rate of production of cyanogen chloride is determined by the rate of its condensation, and not by the rate of absorption of gas by the acid. In a typical experiment 7.35 litres of 12.2% hydrocyanic acid was chlorinated, the reaction vessel not being cooled. The cyanogen chloride was collected in water. The temperature rose quickly to about 30° C. and then ceased to rise. Chlorination was continued for 1 hour 50 mins., by which time much chlorine passed unabsorbed through the apparatus. It was assumed that this indicated the end of the reaction, but analysis showed that this was not really the case. The contents of the two receivers were added together and well mixed, after which the two layers, which separated on standing, were analysed. The total weight of cyanogen chloride formed from the 897 grms. of HCN used was 698 grms., corresponding with 79% of the theory. The cyanogen chloride layer formed in the receivers still contained more than 10% of hydrocyanic acid, although chlorine was passing all the time. By maintaining a low temperature in the reaction vessel the amount of hydrogen cyanide carried over into the receivers could be diminished, but this would also hold back a considerable quantity of cyanogen chloride and thereby cause it to be acted upon by the hydrochloric acid formed in the reaction.

In another experiment 7460 c.c. of 14.7% HCN was chlorinated, without cooling. The first receiver contained 970 c.c. of 2.21% HCN and the second contained water. Hence the total weight of hydrocyanic acid used initially was 1119 grms. After chlorination for 3–4 hours, the contents of the reaction vessel and the two layers obtained by

mixing the contents of the receivers were examined analytically, with the following results:—

	HCN		CNCl		HCl	
	Vol. c.c.	%	Wt. (grms.)	%	Wt. (grms.)	%
Reaction vessel	6300	0.76	48	2.0	126	13.9
Receivers—						
CNCl layer	1510	22.4	338	78.4	1184	Trace
Aqueous layer	1750	5.09	89	5.3	93	0.27
Total weight ..	—	—	475	—	1403	—

Correcting these figures for samples withdrawn for analysis, it appears that at the end of the experiment there were 495 grms. HCN, 1423 grms. CNCl, and 896 grms. HCl. The amount of HCN used is therefore 1119–495=624 grms., and the amount of CNCl equivalent to this is 1421 grms.

In another experiment carried out under similar conditions, and in which the temperature rose to 40° C., the total weight of cyanogen chloride was 97% of the theoretical.

The results of these and other experiments indicate that during the chlorination of 12–15% HCN cyanogen chloride is formed quantitatively according to the equation already given. A rise in temperature up to 40° C. does not diminish the amount produced; it does, however, lead to the distillation of much larger quantities of hydrogen cyanide, with a consequently higher content of this acid in the cyanogen chloride obtained. The hydrochloric acid formed during the reaction is retained almost completely in the reaction vessel.

The hydrogen cyanide contained in the cyanogen chloride can be approximately completely changed into the latter compound by bubbling the crude gas through a series of vessels containing water through which chlorine is also passed.

Further experiments were carried out in which the hydrocyanic acid was formed *in situ* from a mixture of sodium cyanide and mineral acid. In such a method the solutions must be dilute and the temperature not too low, and the mineral acid must be kept in excess. In the manufacture of hydrocyanic acid we have found that it is necessary to add the sodium cyanide solution to the acid, stirring vigorously meanwhile, otherwise pockets of alkaline liquid may be formed and give rise to polymer.

A method was also considered in which sodium cyanide solution is added to a dilute acid, say about 5%, until nearly the equivalent of sodium cyanide has been introduced. Chlorination then leads to the regeneration of the acid, so that sodium cyanide solution can be run in continuously during chlorination if the rate of running in is so regulated that the acid remains in excess. One difficulty with such a method was found to lie in adequate cooling of the reaction mixture, since both the reactions occurring are strongly exothermic, whilst on a large scale it is also difficult so to regulate the rates of addition of sodium cyanide solution and chlorine that the reaction mixture always remains acid. In the experiments where hydrocyanic acid was first made *in situ* and then chlorinated the solution of hydrocyanic acid was made in a carboy of 40–50 litres capacity, containing a cooling coil. The chlorine was introduced through a porous pot, as in previous experiments. Two Winchester bottles containing 2% HCN and water respectively were not cooled, but served to remove excess of chlorine and also some of the hydrocyanic and hydrochloric acids. The cyanogen chloride passed through these Winchester bottles and was condensed in a long, brine-cooled coil, being then col-

lected under water containing a little zinc oxide in suspension in order to neutralise any acid.

Chlorination was maintained at such a rate that the contents of the carboy kept below 20° C. Under these circumstances, once the air had been displaced from the apparatus, no gas or vapour passed from the exit tube of the apparatus. Chlorination proceeded very rapidly, and cyanogen chloride condensed in a continuous stream.

In one experiment 15 litres of 20% sodium cyanide solution was added to 15 litres of 21% sulphuric acid contained in a carboy, under continuous stirring and cooling. The strength of the hydrocyanic acid solution produced was 5.82%. After chlorinating for two hours the cyanogen chloride layer was collected, the various layers mixed, and analyses made. The results showed that the total weight of cyanogen chloride formed was 3022 grms., or 80% of theory. The presence of leaks probably explains the low percentage total production of cyanogen chloride. It is difficult adequately to seal a carboy so that it will stand a slight pressure over some hours without showing signs of leakage. There was only a very small quantity of ammonium chloride formed, which was found as a deposit in the condenser coils.

In a similar experiment where 30% solutions of sodium cyanide and sulphuric acid were mixed, giving a 9% solution of hydrocyanic acid, the yield was only 70% of the theory, and larger quantities of ammonium chloride were formed. Moreover, the cyanogen chloride collected contained 12% of hydrocyanic acid, as against only 1.8% in the above experiment. These results indicate that it is best to use dilute solutions, of about 5% strength, and not allow the temperature in the reaction vessel to rise above 20° C.

Purification of cyanogen chloride.

The cyanogen chloride obtained in the above experiments always contained water and hydrogen cyanide and also traces of hydrochloric acid. When it was not necessary to remove the hydrogen cyanide it was purified by distilling through columns of marble and calcium chloride, the former removing traces of acid and the latter drying the vapour. Shaking with water containing zinc oxide in suspension enables the column of marble to be dispensed with; the cyanogen chloride is simply separated, dried over calcium chloride, and distilled.

Three methods were actually used for purifying the cyanogen chloride, according to the purpose for which it was to be used:—

(1) Chemically pure cyanogen chloride was made on a small scale by cooling the crude substance, saturating with chlorine, shaking the product with (a) mercury to remove traces of chlorine, (b) sodium carbonate or sodium hydrogen carbonate* to remove hydrogen chloride, (c) drying over calcium chloride and distilling.†

(2) A product containing 96–99% CNCl was obtained by passing chlorine through a series of Winchester bottles containing 80–90% CNCl and a good excess of water, the cyanogen chloride thereby distilling in a current of chlorine. The condensed product sometimes contained chlorine in excess, and sometimes hydrogen cyanide, according to the purity of the original cyanogen chloride, rate of passage of chlorine, etc. This process can be applied in conjunction with the process of preparation, although in making large quantities of cyanogen chloride it was found more convenient to carry out the two processes separately.

(3) Shaking with zinc oxide was the practice when 99–100% CNCl was required. Prolonged shaking

with zinc oxide and water reduces the hydrogen cyanide content to 0.05%, and the product so obtained is then dried and distilled. Yellow mercuric oxide is also very convenient for removing even larger percentages of hydrogen cyanide.

The question may arise as to whether in the large-scale preparation (or manufacture) of cyanogen chloride it would be better to make the hydrocyanic acid *in situ*, or first prepare the pure aqueous solution. In the first method the solutions of sodium cyanide and acid used have to be well cooled before mixing, and extensive refrigeration must also take place during the reaction in order to prevent the hydrogen cyanide escaping. With the second method a brine-cooled condenser is only necessary to condense the last traces of vapour, the greater part being condensed in a water-cooled condenser. The demands on a refrigerating plant are therefore very small, and this second method would seem to be preferable. If an aqueous solution of hydrocyanic acid is all that is necessary, there will probably be no need for a brine condenser, so that no special refrigerating plant will be required.

Properties of cyanogen chloride.*

The pure product was a colourless liquid, boiling sharply at 13° C. (uncorr.). Jennings and Scott (*loc. cit.*) give the b. pt. as 13° at 748mm. The freezing point was -8° (Jennings and Scott give -5° to -6°; Regnault gives -7.4°). On pouring the liquid from one vessel to another it froze readily to a snow-like solid. Sp. gr. at 9°/4° = 1.207 and at 4°/4° = 1.218. Analysis gave N 22.7% and Cl 57.8%, theory requiring 22.8% and 57.7% respectively.

Cyanogen chloride is soluble in water to the extent of 7–8% at 8° C. Water is also soluble in cyanogen chloride; the presence of hydrogen cyanide markedly increases these mutual solubilities.

Investigation of the action of hydrogen chloride on cyanogen chloride shows that the following effects are produced:—(a) The cyanogen chloride is polymerised to cyanuric chloride, (CNCl)₃, a reaction which takes place slowly, and, apparently, only to any extent in the absence of water; (b) hydroxylation of the cyanogen chloride to cyanic acid and carbon dioxide, and hence to ammonium chloride and carbon dioxide. This is a much more rapid reaction than (a), and naturally requires the presence of water. Hydroxylation increases in rapidity with the strength of the acid, the action being complete in a few hours with 9N hydrochloric acid. With 2N acid the reaction is very slow, and even with 5N acid takes several days.

Pure cyanogen chloride has been kept for months without showing any signs of polymerisation; even exposure to sunlight for several days has no apparent effect. If traces of water are present a few minute crystals of ammonium chloride are soon deposited on the sides of the containing vessel, but if then poured into a clean dry vessel it can apparently be kept indefinitely.

In agreement with Jennings and Scott (*loc. cit.*) we have not found that dry chlorine has any polymerising effect on cyanogen chloride. With moist chlorine there is a slow action, but no polymer is formed, the action being simply due to the water present.

We have, furthermore, found that pure anhydrous phosgene and cyanogen chloride do not react. In the presence of moisture, however, both ammonium chloride and cyanuric chloride are formed.

ADDENDUM.

Some experiments with a small scale continuous-running apparatus were devised and carried out by Mr. S. B. Phillips. The apparatus was so arranged

* The use of sodium hydrogen carbonate is to be preferred, since it is not alkaline, and the cyanogen chloride is therefore more stable in its presence if water is present.

† This method was worked out by Mr. H. D. K. Drew, M.Sc.

* This work was mainly carried out by Mr. H. D. K. Drew.

that chlorine and a solution of hydrocyanic acid were brought together continuously, in approximately the correct proportions, and the cyanogen chloride formed was separated quickly from the reaction product.

The chlorinating part of the apparatus consists of a brine-cooled tower up which a steady stream of 10–12% HCN gradually flows. Chlorine is forced in through a porous pot fitting loosely into the lower end of the tower, the hydrocyanic acid solution passing through the annular space separating the porous pot from the walls of the tower. The remainder of the tower is filled with beads. As hydrocyanic acid solution flows into the bottom of the tower, the products of reaction overflow at the top, and are there impinged on by a good jet of steam. Cyanogen chloride is thereby vaporised, the vapours passing to a condenser and receiver. The exhaust reaction mixture is collected separately.

In one experiment with 15% hydrocyanic acid solution the product contained 99% of cyanogen chloride and less than 0.2% of hydrogen chloride. In a second experiment 4 litres of 8.9% hydrocyanic acid was run through the apparatus in two and a half hours. The temperature in the tower rose to 15° and remained constant. The product collected contained 95.7% cyanogen chloride and amounted to 70% of the theoretical.

It was not possible to work this method out in further detail, but we are of the opinion that it could be made to give very good results.

ACKNOWLEDGMENTS.

We are indebted to the Admiralty for permission to publish this paper, which is a record* of work on cyanogen chloride carried out under our direction at the Royal Naval Experimental Station, Stratford, E., during the war. We have also to express our indebtedness to a number of service subordinates, who carried out much of the experimental work involved, especially to Mr. W. Auger, B.Sc., A.R.C.S., who was Chief Petty Officer of the laboratory staff.

London Section.

*Meeting held at Burlington House on
March 1st, 1920.*

PROF. A. W. PORTER IN THE CHAIR.

THE PROPERTIES OF POWDERS.

PART I.—THE CAKING OF SALTS.

BY T. MARTIN LOWRY AND FREDERICK C. HEMMINGS.

1.—THE STUDY OF POWDERS.

Although much attention has been paid to the general properties of solids, liquids, and gases, and also more recently to the study of matter in the disperse or colloidal condition, the study of powdered solids has received less attention than its importance would appear to demand.

The properties of powders which merit consideration include:—

(1) The production of powders by *mechanical disintegration* or milling, and especially the process of *fine grinding*, which has been studied much less fully than that of disintegration or rough grinding.

(2) *Disintegration by physical processes*, e.g., the decrepitation of barytes by heat, which has been applied commercially to separate it from blende and other minerals, and the change of crystalline state which makes roll-sulphur brittle, and which certainly assists in the disintegration of ammonium nitrate.

(3) *Disintegration by chemical processes* such as the hydration of quicklime and the dehydration of certain hydrated salts.

(4) *Methods of testing* the fineness of powders produced by these three methods, and especially of those which will pass through the finest sieves on which trustworthy measurements can readily be made.

(5) *Incorporation* of mixed powders as in the milling of gunpowder.

(6) *Bulk density* of a powder, as influenced by its method of preparation and subsequent treatment, and the choice of suitable grades to produce a maximum or a minimum density.

(7) The *compressibility* of powders both towards static loads and under impact.

(8) The *flow* of powders, including the special case in which a powder, such as dried milk, is packed into a bottle or some similar receptacle by the action of a worm-feed.

(9) The *shrinkage* or contraction which takes place in some powders, such as soap powders, after they have been packed into a container; and finally,

(10) The *caking* of a powder to form either a loose agglomerate or a hard stony mass as discussed in the present paper.

2.—GENERAL CAUSES OF CAKING.

A preliminary account of the work which we had carried out on the caking of salts between September, 1915, and December, 1917, was communicated to a meeting of the Faraday Society on Jan. 14, 1918, as part of a general discussion on the "Setting of Cements and Plasters." At the same time some of the principal causes of caking were set out by F. G. Donnan in a paper on the "Agglomeration of Granular Masses," the conclusions arrived at being substantially identical in the two cases.

The agreed causes of caking in soluble salts may be summarised as follows:—

(i.) *Evaporation of moisture* from a wet salt, or alternate absorption and evaporation of water as the pressure of water-vapour in the atmosphere rises above and falls below the vapour-pressure of a saturated solution of the salt. The further lowering of vapour-pressure due to saturation with a second salt or impurity (e.g., $MgCl_2$ in $NaCl$) may be an important factor in bringing the vapour-pressure of the solution within the normal range of variations of the moisture-content of the atmosphere.

(ii.) *Mechanical stress* or pressure resulting in a local increase of solubility in the strained material followed by recrystallisation elsewhere.

(iii.) *Recrystallisation of fine particles* to form larger crystals, as in the prolonged annealing of a metal.

(iv.) *Recrystallisation of metastable crystalline forms* after passing through a transition-point; this may be related very closely to (iii.), since the pseudomorphs may consist initially of particles even more minute than those produced by grinding.

(v.) *Recrystallisation of amorphous material* produced by cold-working of the material.

In each case the presence of a trace of solvent may be postulated. Consolidation by pressing may depend on (ii.), where the solvent plays a predominant part, or on (v.) where shearing and flow may be fol-

* Owing to want of space this record has been considerably condensed for the purposes of publication.

lowed by cohesion even when no solvent can be detected.

3. THE CAKING OF AMMONIUM NITRATE.

Our attention was first directed to the caking of powdered solids by observations made in the case of ammonium nitrate. This salt crystallises from aqueous solutions in long needle-shaped or prismatic crystals, which may run to a length of several feet when the salt is crystallising slowly in a large vat. These crystals carry with them a considerable proportion of moisture, not only on the surface, but in the interior, since the crystals are often tubular in character and contain long threads of mother liquor. When drained either by gravity or in a centrifugal machine a coarsely crystalline product is obtained in which the long needles are broken down into short lengths but otherwise remain intact.

(a) *Caking after drying.*—These crystals, if allowed to dry spontaneously in the air, bind together slightly as the mother liquor on the surface evaporates; but no very great cohesion is observed in the mass, which can easily be broken down again. A coarse, granular product can also be obtained by drying the crystals in a current of warm air at a temperature only slightly above that of the atmosphere. If, however, the wet crystals are dried in any of the ordinary commercial dryers, either by hot air or by contact with a steam-heated surface, the product when packed into barrels cakes together into a hard mass resembling artificial stone, which must be broken up with steel wedges. The contrast between these samples was most readily explained by the fact that the crystals dried at a low temperature retain their original crystalline form, whilst the samples dried by heat undergo a change of state by heating and cooling through 32°C .

(b) *Caking after grinding.*—Caking of an equally marked character occurred in ammonium nitrate which had been poured out in a molten condition, with a small percentage of water, at a temperature a little below the melting-point of the dry salt. On breaking up the rough slabs formed by casting, a product was obtained which did not bind together on storage; but when the slabs were disintegrated and packed whilst still warm, a very hard cake was produced. Some caking was also noticed when the fused product was disintegrated and packed cold. From these experiments it appeared likely that disintegration might also be a factor in promoting the caking of ammonium nitrate.

(c) *Caking after milling.*—Experiments were made to determine the effect of milling cold ammonium nitrate under the heavy rolls of an edge-runner mill. After milling for an hour and leaving under gentle pressure overnight, distinct caking was observed, although the temperature of the nitrate had not risen during milling to the transition-temperature at 32°C .

4. CAUSES OF CAKING OF AMMONIUM NITRATE.

(a) *Change of state.*—When ammonium nitrate is dried by heat, changes of state occur at 32° and at 84°C . The change at 32° is accompanied by an abrupt change in the texture of the nitrate, and even in a commercial dryer a slight arrest of temperature can sometimes be observed (Figs. 1 and 2). At this point the coarse crystals break down, either into brittle pseudomorphs, retaining the form of the parent crystal, or (when the drying is accompanied by agitation) into a snow-like mass which dries to a fine powder. The change of state at 84° is accompanied by a very marked arrest of temperature (see Figs. 1 and 2), but by no visible alteration of texture, as the snow-like mass has usually fallen to a dry powder before this temperature is reached.

Heating of 80 lb. of ammonium nitrate in a small Hind and Lund machine.

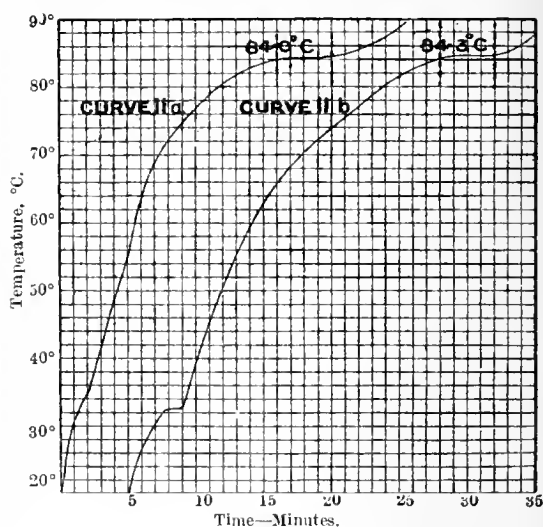


Fig. 1.

Heating of 600 lb. of ammonium nitrate in a large Hind and Lund machine.

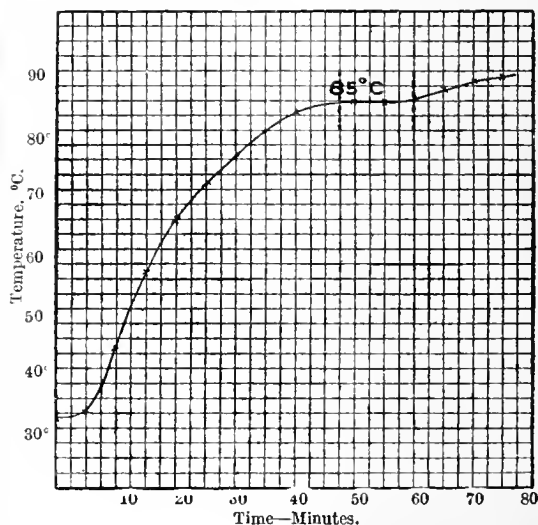


Fig. 2.

It was thought at one time that disintegration rather than caking followed the expansion which accompanies the change of state on heating through 32° (or on cooling through 84°), and that caking as a result of change of state took place on cooling through 32°C . (and possibly on heating through 84°C), following upon contraction of volume. This view could not be maintained, however, in view of the fact that crystals which had been disintegrated by heating to a temperature between 32° and 84°C . formed an extremely hard cake within an hour or two after packing and probably a whole day in advance of passing through the transition-temperature of 32°C . From this observation it was clear that change of state might result in disintegration, but that it was likely to lead to caking only as a result of secondary changes.

(b) *Recrystallisation of fine particles.*—Whilst the view that change of state was a direct cause of caking could not be maintained, it appeared likely that the minute particles of the pseudomorph might tend to grow into larger crystals, just as in the case of a metal submitted to prolonged annealing, and that the growth of the fine particles might be accompanied by caking as a result of the obliteration of the original boundaries between them. This view was supported by the caking after disintegration of the slabs of cast ammonium nitrate referred to under 2 (b) above, since strong caking took place when the hot powder was allowed to cool through 32°, whilst a much less pronounced caking was produced by the mechanical disintegration of the cold slabs. The view that the production of fine particles, either by thermal treatment or by mechanical disintegration, was the principal cause of caking was, however, abandoned in its turn in view of the experiments described under (c) below.

(c) *Mechanical working.*—Mechanical working, leading to the formation of amorphous material, could not be regarded as a potent factor in promoting the caking of ammonium nitrate, since only slight caking was noticed after the disintegration of cold ammonium nitrate, whilst the agglomeration after milling under edge-runners was scarcely enough to enable the product to support its own weight.

(d) *Compression.*—Ammonium nitrate can be consolidated into very hard blocks by pressing. In this process the mechanical working is so much more severe than in disintegration or milling that the production and recrystallisation of amorphous material might play an important part in producing cohesion but even here any greater readiness on the part of the nitrate to consolidate in comparison with an insoluble compound such as calcium carbonate or barium sulphate, could be interpreted most readily as due to the increased solvent action of moisture on the particles supporting the stress of the plunger.

(e) *Liberation of moisture.*—The conclusion arrived at finally was that the key to all the observations that had been made on the caking of ammonium nitrate was to be found in the effect of drying, grinding, milling, etc., on the moisture in the salt. Conclusive proof of this was obtained when it was found that whilst commercial drying at a high temperature was always followed by caking, it was possible in the laboratory to carry the drying of the salt to a point at which it could be kept indefinitely as a loose powder under precisely those conditions which had been found to provoke the hardest caking. Indeed, during the latter part of the time when these experiments were in progress the absence of caking was found to be the most suitable test of the effective drying of samples required for use in other investigations.

(f) *Conclusions.*—The conclusions finally arrived at in reference to the causes of caking of ammonium nitrate were as follows:—

(1) Caking is not caused directly by change of state, but this gives rise directly only to disintegration and to liberation of moisture from the partially-dried crystals; the immediate cause of caking is then the cementing effect on the tiny particles of the disintegrated salt of moisture released during the change of crystalline form.

(2) Liberation of enclosed moisture can also be effected by mechanical disintegration, and this may give rise to caking in precisely the same way as the disintegration and liberation of moisture consequent upon change of state, although the effects are much less pronounced. In the absence of moisture mechanical disintegration would probably not give rise to caking.

(3) In an edge-runner mill the moisture is able to evaporate as fast as it is released by milling, a

fact that is frequently made use of as a convenient method of drying, e.g., in the manufacture of gun-powder; under these conditions the effect of the released moisture is only slight, and the mechanical working does not suffice to produce any serious caking of the salt.

5. EXPERIMENTS ON THE INFLUENCE OF MOISTURE.

The correctness of the views set out above was established by experiments which showed that caking after change of state could be stopped by drying. Supporting evidence was found (i.) in experiments which showed that disintegration of ammonium nitrate gave rise to liberation of moisture; (ii.) in the intimate relationship between solubility and caking which was established by experiments described in the later portions of the present paper; and (iii.) in the fact that, in other cases, mechanical working was proved to have little influence in causing caking except when a solvent was present.

(a) *Caking of ammonium nitrate prevented by drying.*—In order to obtain ammonium nitrate quite free from moisture, 30 grms. of the recrystallised salt was heated in a steam oven at 90° C. for ten hours, the hot salt being ground every hour; it was then left in a vacuum-desiccator overnight. On the following day it was heated again for two hours, during which it was ground every half hour. The salt was then sealed under a lead weight of about 70 grms. in a hard glass tube containing a little phosphoric anhydride kept in position by means of a plug of glass wool. Before sealing, the tube was heated in water at 80° C. and exhausted with a Gaede pump during eight hours, the weight being moved up and down to disintegrate the salt from time to time. After a month this sample of ammonium nitrate showed no tendency to "cake," and even at the end of a year the particles were still free; but so long as there was even a trace of water present agglomeration of the particles occurred.

(b) *Caking by evaporation of moisture.*—The loss of moisture from wet samples of ammonium nitrate is always accompanied by the caking of the salt; and when slight pressure was brought to bear upon the moist substance after packing, hard solid blocks were sometimes obtained. The hardness of the masses formed in this way was found to correspond roughly with the amount of water that had evaporated.

As indicating the importance of solubility in determining caking, reference may be made to the behaviour of ammonium nitrate that has been crystallised from alcohol. When a sample of this product containing about 3% of mother liquor was packed in a glass dish and allowed to stand, the alcohol evaporated, but the salt showed very much less tendency to "cake" than when crystallised from water, the difference being due presumably to the difference in the solubilities of ammonium nitrate in water and in alcohol.

(c) *Liberation of moisture after grinding.*—A sample of coarsely powdered ammonium nitrate that was packed in a mortar after having been exposed to a dry atmosphere was found to show very little tendency to "cake"; when, however, the fragments were ground for about 10 minutes and then repacked distinct "caking" took place. This process of grinding and packing was continued until there was very little evidence of further caking. The original crystals contained 0.24% of moisture; after the first grinding and period of quiescence the moisture-content was reduced to 0.18%, and finally to 0.09%. It thus appeared that one effect of grinding was to break down the crystal structure and to release the moisture that had been retained in the crystal; as this moisture was present as saturated solution, its evaporation would be likely to cause the salt to cake.

In a confirmatory experiment a single crystal weighing 19.963 grms. lost 0.151 gm., or 0.76% moisture by grinding and exposure to a dry atmosphere. In a further experiment some small air-dried crystals lost 0.084 gm., or 0.34% of moisture in a vacuum desiccator, and a further 0.54% after grinding; the amount of moisture trapped in the interior of the crystal and released during grinding (0.54%) was therefore half as much again as the amount of moisture (0.34%) originally present on the surface of the crystal, and released by mere exposure to a dry atmosphere.

(d) *Influence of moisture on caking of amorphous material.*—From the preliminary observations re-recorded in 2 (b) and (c) above, it seemed possible that the formation of amorphous material might play an important part in caking, as is almost certainly the case when blocks of ammonium nitrate are prepared by strong compression, e.g., with a hydraulic press. As it was not practicable to do any effective "cold-working" without introducing moisture to the dry salt, it was not possible to determine finally whether drying the salt would inhibit entirely the caking due to the formation and crystallisation of amorphous material; two simple comparative experiments on ammonium nitrate dried with and without phosphoric anhydride were carried out which, though not very conclusive, suggested that when the salt is not completely dried the effect of work in causing adherence is more pronounced than when drying is as complete as possible.

6. COMPARISON WITH OTHER NITRATES.

Assuming the caking of ammonium nitrate to be due to liberation of included mother-liquor as a result of disintegration by grinding or by change of state, it was to be expected that sodium and potassium nitrates would exhibit some similar phenomena, but in a lesser degree owing to their much smaller solubility in water, namely, ammonium nitrate 185.0, sodium nitrate 87.5, potassium nitrate 31.2 grms. per 100 grms. of water.

(a) These salts do, in fact, cake under much the same conditions as ammonium nitrate, e.g., after grinding, although change of state cannot be utilised as a method of disintegration. The amount of caking is considerably less in sodium nitrate than in the case of ammonium nitrate, and very much less in potassium nitrate. Sodium nitrate has usually to be quarried from a store in which it has been kept for a few weeks, and the lumps which have formed there are so hard that they have been known to break the axle of the machine used to crush them; caking during drying is also very severe, and under some conditions of working a rotary dryer may become so choked that sodium nitrate has to be removed with a pickaxe. At Messrs. Curtis's and Harvey's factories it is stated that potassium nitrate, crude or refined, also has a great tendency to cake, especially when moist or under the influence of heat or pressure. In each case it was found that caking was entirely prevented by drying the salt completely, as in par. 5 (a), and in manufacturing practice drying to about 0.1 to 0.2% of moisture is said to be sufficient to prevent caking on storage.

(b) An experiment was made to compare the amounts of mother-liquor retained by the three nitrates. Crystals which passed through a 4-mesh sieve but were retained on a 10-mesh sieve were weighed out into aluminium boxes and left in vacuum desiccators containing concentrated sulphuric acid, until the weights were constant. The crystals were then broken up from time to time until no further loss of weight could be detected. The final losses in weight were as follows:—Ammonium nitrate, 0.31%; sodium nitrate, 0.68%; potassium nitrate, 0.32%. These results show that the amount of caking that takes place must be deter-

mined by solubility rather than by the proportion of included moisture. It is also interesting to notice that ammonium and potassium nitrates, which both crystallise in the orthorhombic system, contain equal proportions of included moisture, whilst sodium nitrate, crystallising in the rhombohedral system, contains twice as much.

7. OTHER EXAMPLES OF CAKING IN COMMERCIAL PRODUCTS.

Although the caking of the nitrates described in the preceding paragraphs is of a particularly striking character, the same phenomenon is encountered amongst other compounds and is a frequent source of annoyance. In view of the widespread character of this trouble, an inquiry was made, with the financial assistance of the Department of Scientific and Industrial Research, into a number of cases in which difficulties due to caking had been experienced by manufacturers. The substances named as giving rise to trouble as the result of caking were ammonium, sodium, potassium, and copper nitrates, potassium chlorate and perchlorate, potash alum, soda ash, common salt, borax, sodium sulphite, hydrosulphite, and phosphate, Rochelle salt, citric acid and tartaric acid. The majority of these compounds were therefore examined in the laboratory, and in certain cases it was possible to make useful suggestions with a view to reducing the tendency of the salt to a cake.

8. EXPERIMENTS ON THE INFLUENCE OF SOLUBILITY ON CAKING.

Samples of various substances were taken from the laboratory store, ground to pass through a 60-mesh sieve, packed into stoppered bottles, and allowed to remain at rest. The substances examined were grouped as follows:—

(a) *Insoluble salts:* Barium sulphate, calcium carbonate (chalk, marble, calcite).

(b) *Soluble substances* selected to cover a considerable range of solubility: Ammonium, sodium, and potassium nitrates, common salt, sugar, potassium chlorate and perchlorate, potassium bichromate, calcium acetate (less soluble in hot water than in cold).

(c) *Hydrated salts* containing different amounts of combined water: Alum (24 mols.), borax, washing soda, and Glauber's salts (10 mols.), Epsom salts (7 mols.), Rochelle salt (4 mols.), citric acid (1 mol.).

On examining these specimens after six weeks it was found

(a) That the insoluble substances showed no tendency to cake;

(b) That all the soluble salts showed a tendency to cake, and that this tendency appeared to be related in some way to their solubilities;

(c) That the hydrated salts showed a distinct tendency to cake, but that the amount of caking did not appear to be related to the amount of combined water.

The view that water plays an important and perhaps a vital part in the caking of powdered salts was confirmed by experiments in which we attempted to produce caking in an insoluble compound. The substance selected for this purpose was calcite.

(i.) Crystals of this were ground to pass a 100-mesh sieve. The powder was packed in a crucible and allowed to stand in the air, but no signs of caking were visible at the end of three months.

(ii.) 2% of freshly-boiled distilled water was added during the grinding. At the end of a week all the water had evaporated, but there was no sign of caking.

(iii.) When water containing carbon dioxide in solution was used, a definite tendency to cake was observed, which increased as the amount of carbon dioxide in the water was increased.

(iv.) Smooth cleavage-surfaces of calcite, when rubbed together and allowed to rest in contact under a load of 500 grms. showed no tendency to adhere either when the faces were dry or when they were moistened with a few drops of water during the rubbing. When, however, water saturated with carbon dioxide was used, the crystals adhered appreciably.

9. CAKING OF POTASSIUM PERCHLORATE.

Potassium perchlorate for use in explosives requires to be in a very fine state of subdivision and is generally specified to pass through a 120-mesh sieve. It separates from solution in coarse crystals, which when ground in a ball-mill caked into a solid mass on which the balls run with very little grinding action. This caking was traced to the liberation of moisture during grinding, and by eliminating this the caking was prevented. The presence of moisture in samples that have not been dried after grinding may also be responsible for the growth of the grains of this salt during storage which has been reported frequently.

10. CAKING OF CITRIC AND TARTARIC ACIDS.

These two acids in the form of powder are liable to very serious caking. By grinding in a current of warm air and packing in oiled paper this trouble can be eliminated almost entirely.

II. THE CAKING OF CRYSTALLINE HYDRATES. ROCHELLE SALT.

Many hydrated salts give a great deal of trouble through caking, especially when they are ground before being packed. Complete removal of water from the powder before packing is usually impracticable on account of the difficulty of removing the last traces of solvent water without driving off also some of the combined water.

A typical case is that of Rochelle salt,



of which two samples were received. One of these was a fine powder, but the other, originally in the same condition, was now composed of hard solid lumps. It was stated by the manufacturers that after a long and careful series of titration tests they had been able to discover no difference in composition between the caked and uncaked samples. Experiments showed:—

(1) That there is practically no tendency for the salt to absorb or lose water under ordinary atmospheric conditions; but slight setting of a coarsely powdered sample was noticed after about 2 months' exposure to the air of the laboratory.

(2) That setting takes place in the usual way when excess of water is present. The water evaporates and crystalline material is deposited.

(3) That the crystalline salt disintegrates to a very fine powder when dehydrated slowly in a desiccator over sulphuric acid.

(4) That when water is absorbed by the powdered partially-dehydrated salt from air saturated with water-vapour there is distinct caking, accompanied by contraction.

(5) That the caking and contraction continue for some time after the amount of water needed for complete hydration has been absorbed.

From these observations it appeared that the setting might be attributed either (i.) to the escape of water from an imperfectly dried sample of the salt, or (ii.) to the absorption of water by the salt after being partially dehydrated. The commercial samples of the salt are, however, packed in tins provided with tightly fitting lids, and lined with water-proof paper, so that there is very little opportunity for either absorption or escape of water to take place; it is, moreover, difficult to reconcile either of these theories with the statement of the manufac-

turers that they have found no difference in hydration between caked and uncaked specimens of the same original salt. Some theory based upon redistribution, rather than actual loss or gain of moisture, is therefore required to provide a satisfactory explanation of the caking of the salt in the circumstances in which it actually occurs.

12. LIBERATION OF MOISTURE FROM HYDRATED SALTS DURING GRINDING.

In order to clear up this question experiments were made to determine the effects produced by grinding Rochelle salt. A minutely-crystalline sample of the salt was prepared, dried on a porous plate and exposed to the air until the weight was constant. The crystals were then well ground and weighed again immediately after grinding; the loss of weight was 5 mgrms., but after an interval of a quarter of an hour, and again on the following day the weight was the same as before grinding. The experiment was repeated with the same result. After grinding for another five minutes the total loss of weight was increased to 9 mgrms., and after further grinding 15 mgrms., or 0.07% of the weight of the salt; but on allowing the salt to stand for an hour the original weight was regained.

Similar results were obtained with potash alum. The loss was slightly greater than with Rochelle salt, and amounted to 0.09% of the weight of the salt, but the original weight was regained on standing. In the case of salts without combined water grinding releases included moisture, and the solvent that evaporates does not return, provided atmospheric conditions are constant; with the hydrated salts, however, the water that is lost during grinding appears to return during quiescence.

These experiments suggest that the effect of grinding Rochelle salt is to break down the molecule, so that some of the water that was previously in combination becomes free. Of the free water thus produced a small amount is given off as vapour to the air, but the part that remains in contact with the salt will become supersaturated with the lower hydrate, and from this solution crystals of the fully hydrated salt will be deposited on standing. In this way it is possible to explain why there is no difference in the amount of water present in the caked and uncaked samples of Rochelle salt; the very small amount of water that evaporates during grinding returns during packing and need not be specially taken into account in the process of caking. When the salt is packed after grinding the water is present partly in the combined state and partly as saturated solution; during the period of quiescence the water of the saturated solution is taken up to form crystals of the fully hydrated salt, and setting must necessarily accompany such a definite process of crystallisation. The actual amount of water present will, however, be exactly the same after as before caking. The principles here given no doubt hold good for other crystalline hydrates.

13. CONTRACTION DURING CAKING OF HYDRATED SALTS.

The contraction observed during the caking of Rochelle salt was noticed on several occasions when dealing with other salts, including potash alum, and was particularly noticeable in the case of copper sulphate. As this phenomenon has an important bearing on many problems connected with chemical industry, it was considered that the mechanism of the process was well worth investigation, especially as the contraction appeared to be related very closely to the caking. Copper sulphate was chosen for the investigation, and a careful study was made of the changes that take place during the hydration of this substance.

A quantity of the anhydrous salt, prepared by heating the pentahydrate to 240° C. in a crucible until the weight was constant, and kept in a desiccator over phosphoric anhydride, was weighed into a thick-walled crystallising dish, transferred to an atmosphere saturated with water-vapour, and weighed at intervals of 24 hours. The following observations were recorded:—

(1) The rate of absorption of water was greater in the system $\text{CuSO}_4\text{—CuSO}_4\cdot\text{H}_2\text{O}$ than in the system $\text{CuSO}_4\cdot\text{H}_2\text{O—CuSO}_4\cdot 3\text{H}_2\text{O}$; and greater in the latter than in the system $\text{CuSO}_4\cdot 3\text{H}_2\text{O—CuSO}_4\cdot 5\text{H}_2\text{O}$.

(2) There was very little change in the volume of the powder until after sufficient water for the formation of the monohydrate had been absorbed. After this point had been reached the mass began to swell up in a remarkable manner. The swollen mass was broken and levelled down periodically, but the swelling continued until an amount of water equivalent to nearly 4 molecules had been absorbed.

(3) When the hydrate had reached a point corresponding to the absorption of 4.95 molecules of water, setting and contraction commenced, the salt shrinking right away from the sides of the containing vessel. This point was marked by a great slowing down in the rate of absorption of water. The contraction continued until the amount of water present was equivalent to 5.1 molecules.

(4) On exposing these fully hydrated blocks to the atmosphere, water was given off, rapidly at first, but subsequently very slowly, until the amount present was almost exactly the same as when contraction commenced. The density of two of the blocks was found to be 2.02 and 2.05 respectively, the absolute density of $\text{CuSO}_4\cdot 5\text{H}_2\text{O}$ at 15° C. being 2.284.

(5) A sample of fully hydrated salt was ground and exposed again to a saturated atmosphere. Setting and contraction were apparent, but were much less intense than those observed previously.

(6) A sample of the salt was partially dehydrated and ground. Setting and contraction were observed at approximately the same point as before (4.95 molecules of water), but here, again the changes were not so marked as when the anhydrous salt was under consideration.

14. CAUSES OF CAKING AND CONTRACTION IN COPPER SULPHATE.

It is noteworthy that whereas anhydrous copper sulphate swells during hydration, up to about $4\text{H}_2\text{O}$, contraction and caking only occur when hydration is almost complete, namely, 4.95 molecules. At this stage the salt will consist almost entirely of the pentahydrate with a few particles of trihydrate. The latter will no doubt be found near the centre of the mass, and the water absorbed on the surface will therefore be present temporarily as a solution saturated with respect to the pentahydrate. When this comes into contact with the trihydrate this will dissolve to form a supersaturated solution, and crystallisation as pentahydrate will at once set in. Only when partially dehydrated salt and free water (as saturated solution) are present in the same mass does marked contraction or caking occur. Thus, when the partially dehydrated salt is in large excess there is no contraction because the water is directly absorbed by it; and, again, caking and contraction are only slight when the fully hydrated salt is exposed to a saturated atmosphere, because there is none of the trihydrate present to produce a supersaturated solution.

After caking in a saturated atmosphere is complete, the fully hydrated blocks of copper sulphate give up all their solvent water when transferred to an unsaturated atmosphere, and return practically to the composition at which caking began, namely, almost the theoretical composition of the pentahydrate.

As regards the mechanism by which contraction is produced during caking, it may be suggested that when the saturated solution of the pentahydrate comes into contact with the particles of trihydrate, the smallest particles will dissolve first and equilibrium will be restored by the growth of the larger crystals. This virtually resolves itself into a process whereby several small crystals are formed into one large one, and this may be a direct cause of the contraction which takes place. The amount of contraction that is possible will be determined in part by the amount of air space or "voids" originally present. The high density (approaching that of the crystal itself) obtained in the hydrated blocks of copper sulphate affords excellent evidence of the efficiency of the forces that are at work to produce contraction.

An incidental factor in determining the amount of caking and contraction is the original size of the particles. Attention may be directed specifically to the beautifully disintegrated products which are formed by dehydration, since these compare not unfavourably with the powdering of lime by slaking or the disintegration of ammonium nitrate by change of state. It is, indeed, very difficult to grind the fully hydrated salt to anything like the same degree of fineness as that to which the anhydrous salt was reduced. The same difficulty was experienced, though not nearly to so large an extent, in grinding a sample that had been partially dehydrated. It is therefore possible that the dehydration of the copper sulphate (like the change of state in ammonium nitrate) may owe much of its importance as a factor in causing caking to its efficiency in producing a more minute subdivision than can be reached by the mechanical grinding of the salt.

15. CAKING OF COPPER NITRATE.

This salt gives very great difficulties, due to caking. Below 24.5° C. it exists as a hexahydrate, $\text{Cu}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$, but when this temperature is exceeded the salt begins to melt; crystals of the trihydrate separate at the bottom of the containing vessel, and the upper part is filled with solution; on cooling, crystals of the hexahydrate again separate, and a solid block is formed. Now, this temperature, 24.5° C, is often exceeded in the laboratory and factory. Direct sunlight easily brings about the decomposition of the hexahydrate, and so does grinding. It appears, too, that even the pressure of the substance, when packed in bulk, may be sufficient to bring about this change.

16. CAKING OF SODIUM BISULPHITE.

Sodium bisulphite cakes under rather peculiar circumstances, and (with the hydrosulphite described in the following paragraph) may be regarded as an illustration of a third type of caking, which is accompanied by and may indeed be caused by a chemical decomposition other than the mere gain or loss of water.

When sodium bisulphite is exposed to the air of the laboratory, it loses sulphur dioxide gradually, but there is little tendency to cake. When, however, it is placed in a confined atmosphere saturated with water-vapour, the loss of weight is more than thirty times as rapid, and under these circumstances the loss of sulphur dioxide is accompanied by hard caking of the salt. After a time gas ceases to be given off, the weight of the salt begins to increase as water is absorbed, and there is a slight softening of the mass.

The explanation of these changes is that the sulphur dioxide, being more than twice as dense as ordinary air, can only free itself from the bisulphite very slowly. Equilibrium is soon reached between the dissociation-pressure of the sulphur dioxide in the salt and the vapour-pressure of that which has been set free. When, however, the surrounding air

is saturated with water-vapour, the conditions of equilibrium are disturbed; the solubility of the gas (5000 volumes in 100) is enormous, and any sulphur dioxide set free from the salt is at once taken up by the water in the surrounding atmosphere, thereby opening the way for the release of more gas from the solid. That this decomposition of the salt actually takes place during caking was confirmed by the strongly acid reaction of the water in the vessel in which the experiment was conducted.

17. CAKING OF SODIUM HYDROSULPHITE.

Two samples of this substance of the same manufacture, and packed in 1 lb. bottles, were investigated. One bottle had been opened on several occasions, and in this the salt was in the form of a hard, rock-like mass. The other was as originally supplied, i.e., tightly corked and sealed with wax, and in this the particles were perfectly free.

Now the hydrosulphite can form a hydrate containing 2 molecules of water, and the formation of this from the anhydrous compound necessarily produces caking. The hydrate is, however, very unstable, and decomposes rapidly, giving off sulphur dioxide; this decomposition serves to increase the caking that has already commenced.

The granular commercial product is obtained by boiling the dihydrate with absolute alcohol to remove the combined water, the resulting anhydrous compound being comparatively stable. It is evident, therefore, that the caking of this substance is due to the formation of a crystalline hydrate and to subsequent decomposition of the salt.

18. METHODS OF PREVENTING CAKING.

The experiments on hydrated salts, together with those on dry ammonium nitrate, prove clearly that both caking and contraction can be avoided if all traces of free solvent are removed. The results obtained during the study of copper sulphate suggest that this can be accomplished very easily in the case of crystalline hydrates, since agglomeration would not be expected to take place in a dry atmosphere so long as there is a small amount of a lower hydrate present. Experiments were therefore made in which 5% of the trihydrate, $\text{CuSO}_4 \cdot 3\text{H}_2\text{O}$, was ground up with the pentahydrate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, and sealed up in a glass tube. A sample of Rochelle salt was treated in a similar manner after adding 5% of the anhydrous compound. Only slight agglomeration occurred in both cases, and this was attributed to the changes produced by grinding, and to unequal distribution of the lower hydrate or anhydrous salt. In order to eliminate these secondary causes of caking fully hydrated samples of both salts were prepared in the form of tiny crystals, which were then subjected to 2% dehydration with stirring, and packed as before. The effect of this treatment was to disintegrate the surfaces of the crystals and to coat them with a film of lower hydrate; as a result, there was not the slightest tendency to agglomeration after a period of several months.

These results suggested the possibility of preventing the caking and contraction of other substances, crystallising without water of crystallisation, by the addition of a small percentage of the anhydrous form of some salt capable of forming a definite crystalline hydrate. A series of experiments was made in each of which 40 grms. of dry ammonium nitrate was taken from the same sample, 1% of water added, and then 1% of a dehydrated salt. Each mixture was stirred, transferred to a hard glass tube, closed by a tightly fitting rubber stopper, and sealed with collodion. A sample of ammonium nitrate with 1% water but no anhydrous salt was prepared at the same time and packed under the

same conditions. The tubes were set aside for a month, when the following observations were made:

SUBSTANCE USED.	RESULT.
Water only	Decided caking, large contraction
Anhydrous calcium chloride.	Caking with contraction.
Anhydrous magnesium sulphate.	Do. Do.
Dehydrated alum.	Slight caking, no contraction.
Anhydrous copper sulphate.	Do. Do.

The different effects produced by the various anhydrous compounds are probably due to differences in the stability of the hydrates to which they give rise. All the samples were, of course, subject to variations of temperature. In order to test the behaviour of samples containing a smaller proportion of water, 2% of anhydrous copper sulphate and 2% of fused calcium chloride were added in two experiments to ammonium nitrate containing only 0.1% moisture. After two months the particles were still perfectly free.

These experiments show that it is possible to prevent the caking of crystalline salts by this method, but the relatively large amount of foreign material that is required makes the method impracticable for industrial purposes.

Two insoluble salts, barium sulphate and calcium carbonate (2½%), when added to ammonium nitrate (1% moisture), at first appeared to prevent caking, but subsequently a gradual hardening took place, which was attributed to chemical interaction.

19. EFFECT OF COLLOIDS.

It was thought that, since the changes under consideration were produced by crystallisation, it might be possible to inhibit them by the addition of a colloid. Agar-agar, powdered gum arabic, gelatin and glue were the colloids used, but here again the amount necessary to produce a change worthy of consideration makes the adoption of such a method of theoretical interest only. Agar-agar seemed to have the most marked effect; a sample of ammonium nitrate containing 0.2% of water and 2% of agar showed no tendency to agglomerate. A sample of ammonium nitrate crystallised from a solution containing 1% of gum arabic produced crystals which showed none of the needle-like structure so characteristic of this salt.

20. RECOMMENDATIONS.

From the experiments and observations that have been described in this paper, and the conclusions that have been drawn therefrom, it will be seen that caking is most likely to take place:—

- (a) In crystalline salts that are very soluble.
- (b) In hygroscopic and deliquescent substances.
- (c) In crystalline hydrates.
- (d) In substances that are liable to decompose in the presence of moisture.

In order to minimise the troubles that are caused by caking the following measures are suggested:—

(1) Care should be taken to reduce to a minimum the number and amount of soluble impurities.

(2) When substances are handled in bulk and are not required in a very pure condition, the most convenient method of supply is in the form of large crystals, the surfaces of which have been dried, e.g., in a centrifugal machine, without breaking up the crystalline structure. The larger the crystals the fewer will be the points of contact between them and the smaller will be the tendency for the material to bind together.

(3) When a finer product is required, the most satisfactory way of supplying it is in minute unground crystals, e.g., as prepared by the rapid cooling of a saturated solution with constant stirring whilst crystallisation is in progress. The surfaces of these crystals should be dried before packing. The method of drying will depend on the nature of the substance, but agitation during drying is a very

important factor. In the case of hydrated salts the surfaces of the crystal should be slightly dehydrated.

(4) When a substance is specially required in a finely-powdered form, there should be careful drying, with agitation, after grinding. This is a very important point which has often been neglected. Great care has been taken to dry the crystals before grinding, but the fact that moisture is released by grinding and needs to be driven off subsequently does not appear to be generally recognised in commercial practice. This measure applies particularly to hydrated salts.

(5) As far as possible the drying should be carried out at low temperatures; or, if the first part of the drying is conducted at higher temperatures, the finishing process should be at atmospheric temperatures.

(6) Substances should not be packed hot unless all traces of moisture have been removed.

(7) Steps should be taken to maintain a "dry" atmosphere during packing. In order to prevent absorption of moisture, the pressure of aqueous vapour in the atmosphere should be less than the vapour-pressure of a saturated solution of the salt. This can be checked by taking care that the difference between the readings of the wet and dry bulb thermometer does not fall below a fixed minimum depending on the hygroscopicity of the salt. This difference can readily be maintained in winter by working in a warm room, but this method of drying the air is open to objection in summer.

(8) Care should be taken to prevent access of moisture to the substance after packing, e.g., by taking care that it is packed in a dry vessel; this applies particularly to wooden boxes, casks, etc.

(9) When substances that are particularly liable to cake in the presence of moisture are packed in glass bottles, the corks should be covered with paraffin wax or some similar waterproofing material. When tins are used the edges of the lids, if not soldered, should be bound by adhesive tape. Casks may be lined with paraffined paper to check the access of moisture to the contents.

(10) Caking may be minimised by keeping the temperature as constant as possible during storage.

21. SUMMARY.

The caking of salts is in general dependent on the presence of a solvent, usually water. The following cases have been studied:—

(1) *Nitrates*.—In the case of ammonium nitrate very hard caking follows the change of state which takes place at 32° C., but this caking can be prevented by very thorough drying. Hard caking also follows upon mechanical disintegration of the warm salt, but this is also attributed to the influence of moisture released during disintegration. Ammonium nitrate crystallised from alcohol shows much less tendency to cake than when crystallised from water. Caking of ammonium nitrate due to mechanical working is only slight when the conditions are such that moisture can escape from the powder.

Sodium nitrate forms a very hard cake when stored in bulk, but this can be prevented by commercial drying, whereas it is precisely this process which gives rise to the most serious caking in ammonium nitrate. This difference is attributed to the polymorphism of ammonium nitrate, which has no analogy in the case of sodium nitrate.

Potassium nitrate, which is less soluble than either of the two preceding salts, may cake during storage, but apparently to a much smaller extent.

(2) *Other anhydrous compounds*.—Serious caking is reported to occur, not only in common salt and in soda ash, but also in potassium chlorate, potassium perchlorate, citric acid, and tartaric acid. In the last four cases caking frequently accompanies or follows the grinding of the dried crystals. This is

probably due to the liberation of more moisture during grinding, and can be prevented by drying the compound during or after grinding, and packing in a dry condition.

(3) *Hydrated salts*.—Caking of hydrated salts frequently gives rise to trouble, e.g., in the case of Rochelle salt, borax, sodium phosphate, and alum. These hydrates cannot be dealt with in the same way as anhydrous salts, since it is difficult to secure complete drying of the hydrate without partial decomposition. The view is advanced that mechanical working of a hydrated salt not merely releases included moisture, but may result in a breaking up of the hydrate similar to that which takes place when the salt is heated, just as the phenomena which accompany the formation of amorphous materials during cold-working may be compared with the effects produced by fusion. Grinding is found to be accompanied by a slight loss in weight, which is recovered very quickly from the air. It is not thought that this is sufficient to account for caking of the salt, which is attributed rather to recrystallisation following on the disturbance of the normal distribution of water which results from the grinding of the salt, just as sodium sulphate may recrystallise in the normal form after being melted in its water of crystallisation.

(4) *Loss of sulphur dioxide during caking*.—Sodium bisulphite and sodium hydrosulphite cake on exposure to air, but this is a more complex change, since not only is water absorbed, but sulphur dioxide is also liberated.

(5) *Contraction during caking of copper sulphate*.—When powdered anhydrous copper sulphate is exposed to moist air it swells considerably during hydration, but the absorption of the final 1 per cent. of water is accompanied both by caking and by strong contraction. The slight excess of water taken up by the blocks is quickly lost in a dry atmosphere when hydration and caking are complete. Caking and contraction in this case are believed to be due to the simultaneous presence of particles of the trihydrate and of water saturated with the pentahydrate, in which the trihydrate dissolves and crystallises out again in the ordinary form. The caking of hydrated salts can be prevented by the presence of a small amount of a lower hydrate, which absorbs the traces of moisture which are so effective in producing caking in a salt which is almost fully hydrated.

Attention is directed to the disintegration which accompanies the change of state of ammonium nitrate when dried by heat and which is also observed in the dehydration of many hydrated salts. This disintegration may be used as a substitute for grinding, just as in the converse process of slaking lime by the addition of water.

DISCUSSION.

Dr. J. A. HARKER said that three years ago, after many preliminary experiments in conjunction with some members of the staff of the Ministry of Munitions Research Laboratory, it had been found that under proper conditions it was possible to produce solid ammonium nitrate, free from nitrite, directly by interaction of ammonia, steam and air, with excess of oxides of nitrogen obtained from the oxidation of ammonia. That promised to be an important process, possibly of commercial value. Ammonium nitrate made in that way was nothing like so deliquescent as ordinary ammonium nitrate. It took up water, at all events initially, at a very different rate. If a bottle full of fresh crystals were left to stand for 24 hours, the crystals would be found to have sunk down by about 20 per cent. in volume, but nothing more happened, and if kept dry it remained in the form of fine microscopic crystals, without any visible tendency to cake, even after long keeping.

Mr. A. E. BERRY asked the author if he had ascertained what methods were used by the manufacturers of table salt who had, he believed, solved the problem of caking. He concurred in the view of the author that caking was entirely due to moisture. If an anhydrous salt were ground and re-dried, the problem of caking was solved. If the drying of chemicals was hastened, or if the temperature was too high, caking was bound to occur. It was possible to dry sodium phosphate and grind it into powder containing as much as 30 per cent. of moisture and it would not cake, yet if dried under different conditions it might cake with only 10—15 per cent. of moisture.

Sir ROBERT ROBERTSON thought the author had made out a strong case for the dominant influence of moisture in regard to caking, particularly in connection with ammonium nitrate. The question of the formation of pellets was more or less analogous to the phenomenon of caking. While, on the one hand, ammonium nitrate when perfectly dry, or with a moisture content of about 0.020%, would form into pellets having a definite cohesion with a certain pressure, less pressure was required to get the same cohesion with ammonium nitrate containing about 2 per cent. of moisture. But while ammonium nitrate was thus in line, there were other substances which did not appear to be so. Calcium carbonate gave coherent pellets with varying proportions of moisture, but calcite, under similar circumstances, gave a very friable pellet, the particles in this case seeming to retain their original crystalline form and prevent cohesion. Barium sulphate was like calcite in giving friable pellets, and the hardness of the substance appeared to be of importance. There was undoubtedly much work to be done yet on the conditions favouring the formation of coherent pellets.

Dr. S. RIDEAL recalled that during the Boer war pellets of sodium bisulphate had been required to sterilise water from typhoid; the machine had to be lubricated with boric acid to prevent caking of the salt. The pellets became so hard on keeping that they could not be dissolved in water; this difficulty was overcome by adding a little sodium carbonate to the bisulphate.

Dr. H. BORNS asked if copper sulphate changed colour at any definite stage of hydration.

CAPTAIN C. J. GOODWIN suggested that the impurities in the material might be of some importance in regard to caking. He believed that one of the chief impurities which promoted caking of table salt was calcium sulphate, whereas to prevent deliquescence of sodium nitrate it was necessary to remove the sodium chloride.

Mr. J. G. A. RHODIN did not think that hydration had so much to do with caking of salts as the author seemed to think. The principal factor, it seemed to him, was that adhesion turned into cohesion between the crystals. During the past fortnight he had had the experience of using ordinary Portland cement as the hearth for smelting metal at high temperature. Cement was believed to hold together on account of hydration by adhesion of water, but he had found recently that Portland cement when used as the hearth for smelting metal at 1200° C. held together after a fortnight. He mentioned the well-known case of the adhesion of metal surfaces when placed in contact. The difficulties with ammonium nitrate could be overcome by proper drying.

Dr. R. LESSING asked if the ammonium nitrate used and also the products at the various temperatures of transition were perfectly neutral. It had been found practically impossible to dry ammonium sulphate unless it was at the same time neutralised;

to obtain a salt which would remain perfectly dry and in a fine condition, it was necessary to grind it at the time of drying. This agreed with Dr. Lowry's suggestion that more moisture could be extracted from the inside of the crystals, after the crystals had been once broken up, than from the surface. Whilst agreeing with Dr. Lowry's explanation, he suggested that at the high temperature of 70° C. a certain amount of dissociation might take place, and that nitric acid was formed as well as free ammonia. If that were so, it would be possible that on re-combining the fragments of the dissociated molecules did not re-combine exactly with each other, and that this facilitated caking in a measure that water alone would not justify. This would also explain why caking took place so suddenly after drying. In all the cases described the drying was not very perfect, and that again would account for the rearrangement of acid and ammonia in some of the water. The solubility of ammonia gas in solid ammonium nitrate, investigated by Divers, proved that this salt stood in a class by itself. The acidity of ammonium sulphate was almost invariably due to pyridine in the form of bisulphate, which could never be got entirely neutral without loss of pyridine; if pyridine were present in the original gas liquor from which the sulphate had been made, the neutralisation would never be complete until the whole of the pyridine had been driven off and the acid set free by that method neutralised again.

Mr. R. G. EARLY gave some figures to illustrate the pressure necessary to produce coherent pellets with certain salts. The figures were obtained by pressing the salt at known pressures until a coherent block was obtained as tested by the "Autopunch" on the principle of the Brinell hardness test. In the case of ordinary soluble salts the pressure necessary to give coherence was:—Ammonium chloride, 0.4 ton per sq. in.; potassium nitrate, 0.75 ton; ammonium sulphate and common salt, 1 ton; sodium nitrate, 1.3 ton; potash alum, 2 tons. In the case of insoluble salts, precipitated calcium carbonate required 0.8 ton; the same material after six hours in a steam oven at 100° C. required the same pressure; calcite in the form of small crystals, 20- to 30-mesh, did not very easily go into the form of pellets, and after pressing at 4 tons it could be easily broken down; calcite ground down to 60-mesh showed the same characteristics, as it also did after six days' exposure to the air. Barium sulphate did not produce a coherent block with 8 tons pressure, but broke down into laminae with a gentle pressure of the fingers. Analysing these results, it was seen that in the case of soluble salts, solubilities and pressures did not run parallel, though there was an indication that the less soluble were the more easily pressed into coherent blocks. Ammonium nitrate, however, took a place among the more insoluble salts, an average sample requiring a very small pressure, comparable with that for ammonium chloride. Considering the behaviour of insoluble salts, barium sulphate showed extreme reluctance to bind together. Fineness of material did not appear to have any great effect, there being, perhaps, a little greater difficulty in pressing the finer material. With regard to the amount of mother liquid required for pressing to coherence, ammonium nitrate under a pressure of 0.5 ton went into coherent pellets with 2% of water present; small crystals containing 0.15% required 0.4 ton. When drying was resorted to the results were as follows:—0.16% moisture, 0.45 ton; 0.10%, 0.5 ton; 0.07%, 0.6 ton; and very fine powder containing less than 0.05% moisture, 0.8 ton. Although the effect of drying was not very marked, there was a distinct tendency for the pressure to increase as the salt became more completely dried. The time of pressing was very short, and practically no

time was allowed for maturing, such as was the case when ordinary setting took place. Whilst the actual effect of pressure was to increase the solubility in most cases, ammonium chloride was the well-known exception, and would be deposited from solution under pressure.

PROF. PORTER said that the direct effect of pressure could only be ascertained definitely if pellet formation were conducted at various temperatures. If, for example, some chemical change took place under pressure, he thought it would be found that there was rather a great rate of change with temperature. On the other hand, if it was purely a physical change that was brought about—the mere effect of bringing particles into closer proximity and bringing molecular forces into play—then the pressure required would vary very little with temperature, and would probably diminish as the temperature rose. The influence of pressure upon solubility could be of two kinds. In the first place, supposing a material system which was throughout under pressure, there would be a slight variation of solubility applying to that case. On the other hand, a second case could arise, and judging from Dr. Lowry's remarks he fancied that this was probably the one which came into practice, and that was the case in which the whole of the system was not throughout under the same pressure; in which the liquid into which the solid was dissolving was not under pressure, but might be escaping at the edge, and therefore the pressure was removed. In that case there were precisely analogous phenomena to the solidification of water into ice. With ice and water both under pressure, the variation of the melting point with pressure was $1/140^\circ$ for each atmosphere increase, assuming that the solid ice and the water were both sustaining a pressure of 140 atm., but when the water was escaping freely from the region under pressure the lowering of the melting point was 10 or 11 times as great. That might have a very important bearing upon the problem of the alteration of solubility with pressure which Dr. Lowry brought in to account for the solution taking place which was followed by crystallisation at a subsequent stage. In such a case the actual variation of solubility with pressure might be expected to be very much larger than it would otherwise be. There were many other problems which had to be brought under consideration, and particularly the problem of the effect of complete dryness.

DR. LOWRY, in replying, said that he assumed that pure sodium chloride, in saturated solution, did not give a sufficiently low vapour pressure to absorb moisture from an atmosphere containing an average amount of moisture. The presence of impurities lowered the vapour pressure to a point at which moisture was either absorbed or lost from day to day, and this was really the dominant factor there. With regard to copper sulphate, he did not think there was any change of colour during the caking and shrinkage, as the salt was already almost fully hydrated. The question had been raised as to whether the caking of ammonium nitrate might not be due to the dissociation into ammonia and nitric acid, and whether the ammonium nitrate used was neutral. As a matter of fact, it was; the English ammonium nitrate was always boiled down in alkaline solutions containing free ammonia, and that was why it was always free from pyridine; the American ammonium nitrate was evaporated in acid solution, and they had the pyridine as an impurity whilst we had thiocyanates. He was convinced that moisture was not the universal cause of caking, and that when dealing with substances other than salts other causes operated, e.g., he was an entire believer in the theory of amorphous materials, and in the consolidation of metals by cold welding or by flow through an orifice

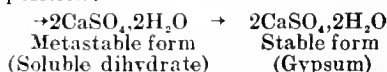
it was pretty certain that these were the operative causes. He had wanted to attribute the caking of salts to causes which might be universally operative, but when he had been shown ammonium nitrate which did not cake merely because it had been dried, he felt bound to admit that moisture was the fundamental cause of caking in this salt.

Dr. T. MARTIN LOWRY and Mr. S. WILDING presented a note on the setting of dental cements, as follows:—

The most important cases of caking or setting may be classified as follows:—

1. *Recrystallisation of an anhydrous or hydrated salt without change of chemical composition, as discussed in the preceding paper.*

2. *Formation of hydrates, as in the setting of plaster of Paris, although in this case the hydration is followed by a recrystallisation without change of composition:*



3. *Hydrolysis of complex salts by water, as in the setting of barium silicate as formulated by Desch, $\text{Ba}_2\text{SiO}_4 + 15\text{H}_2\text{O} = \text{BaSiO}_3 \cdot 6\text{H}_2\text{O} + \text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$, and of Portland cement.*

4. *Formation of new salts, e.g.:—*

(a) *Magnesium oxy-chloride cements, in which a basic chloride is formed by the addition of aqueous magnesium chloride to powdered magnesium oxide,*



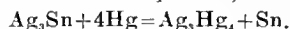
(b) *Zinc oxy-phosphate cements, used very largely in dentistry, in which zinc oxide is converted into a basic phosphate by a solution of phosphoric acid which may already contain some zinc phosphate.*

(c) *"Silicate" cements, in which various silicates, and various salts of aluminium, are converted into phosphates by the action of an aqueous solution of phosphoric acid, sometimes saturated with aluminium or zinc phosphate.*

(d) *Caking, practically equivalent to the formation of a cement, accompanies the conversion of calcium phosphate by the action of sulphuric acid into a mixture of sulphate and superphosphate.*

5. *Amalgams.*—In these cements mercury takes the place of water. Unfortunately no information appears to be available in reference to the setting of the amalgams of copper and palladium, which have both been used effectively in dentistry, but cadmium amalgam appears to set because the solid solution of mercury and cadmium, which is isomorphous with mercury, is less soluble at atmospheric temperatures than a corresponding solid solution isomorphous with cadmium.

The best of the amalgam-cements is, however, made by the action of mercury on an alloy of silver and tin. Experiments carried out in the laboratories of Professor McBain at Bristol have proved that the setting of this amalgam is due to the chemical change shown in the equation,



The more complex cases of setting, which are set out under the last two headings, are used very extensively in dentistry, where the conditions are particularly exacting and the cost is of little importance, in view of the small quantities used. They are well worthy of detailed study in any scheme for the elucidation of the general phenomena of caking or setting. Thus, one point demanding investigation is the part played in all these changes by amorphous or colloidal material, which appears to be important even in the case of the amalgams.

Bristol and S. Wales Section.

Meeting held at Cardiff on March 3, 1920.

MR. W. S. HIRD IN THE CHAIR.

RESEARCHES ON COAL.

Part I.—The thermal decomposition of coal at low temperatures.

BY S. ROY ILLINGWORTH, B.Sc., F.I.C., A.R.C.S.

The thermal decomposition of coal at low temperatures has been investigated by a number of independent workers, both as regards the gaseous products evolved at definite temperatures, and as to the liquid products. Important results have been published by Burgess and Wheeler (Trans. Chem. Soc., 1910—1914), Porter and Ovitiz (U.S. Bureau of Mines), Porter and Taylor (U.S. Bureau of Mines), Jones and Wheeler (Trans. Chem. Soc., 1915), and others, and a study of certain Scotch coking coals was carried out by Anderson in 1895. An excellent and critical survey of the subject is contained in the monograph on the "Constitution of Coal" by Stopes and Wheeler, pp. 7-12 (H.M. Stationery Office).

The present communication deals with the results of an investigation planned to elucidate the relative stability of certain known components of coal when subjected to carbonisation under standard conditions. The relative behaviour of certain coals when carbonised at various temperatures up to 450° C. has been studied. The coals chosen are coals which when carbonised at 950° C. yield cokes of different types. The samples used were "mine" samples, cut from roof to floor of the seam, and they comprised about 80—100 lb. of coal, from which a 14 lb. laboratory sample was obtained by the usual process of quartering down the bulk; this smaller sample furnished the coal actually used in the investigation.

The analyses of the coals were as under:—

TABLE I.

	No. 2 Llantwit.	No. 3 Rhondda, S. crop.	No. 2 Rhondda,	Two-foot nine, S. crop.
Volatile	37.06	31.50	21.16	26.24
Fixed carbon	57.53	66.93	71.20	71.26
Ash	5.41	1.57	7.74	2.50
On dry ash-free coal—				
C	82.87	86.70	87.96	87.70
H	5.80	5.00	4.35	4.94
O	7.76	6.21	4.32	5.39
N	1.49	1.45	1.42	1.34
S	2.08	0.64	1.95	0.63
Ratio C/H	14.29	17.34	20.22	17.75

The No. 2 Llantwit is used for gas making and yields a very porous coke. On the basis of the classification of coals as revised by Professor Bone (see "Coal and Its Uses," p. 64), it must be classed as a "caking long-flame coal of the bituminous genus."

The No. 3 Rhondda is carbonised to produce metallurgical coke and is also used in the gas industry. The coal yields a dense coke which tends to be brittle. This coal is classified as a "hard coking, bituminous coal," and is intermediate between the gas coals and the typical coking coals. The Two-foot-nine is a typical "hard coking coal," whilst the No. 2 Rhondda is a "hard coking (short-flame) coal" and yields a dense coke.

A known weight of the coal ground to the same degree of fineness was submitted to the influence of definite fixed temperatures for varying periods of time and the resultant loss of weight determined. Since the liquid products were not completely volatile at the temperatures maintained in the work, the residue from each heating operation was washed with cold carbon tetrachloride to remove the oily substances, dried *in vacuo* at 105° C., and then weighed. The loss of weight was calculated on the washed and dried residue. A known weight of the final residue was extracted with pyridine in a Soxhlet apparatus and the pyridine extract dried and extracted with chloroform. This cycle of operations furnished the data necessary for calculating the amounts of the pyridine-soluble compounds, the γ - or resinic bodies, and the β -cellulosic substances in the residues at different stages. The terms β - and γ -compound are used in conformity with the terminology proposed by Wheeler. The β -compound is that portion of the coal soluble in pyridine but insoluble in chloroform, whilst the γ -compound is that portion soluble in both pyridine and chloroform. The processes outlined above were carried out as follows:—

Drying.—All coals, extracts, or residues were dried in a vacuum oven at 105° C., under a pressure of 60 mm. of mercury.

Heating the coals.—The samples of coal were ground to pass through a 30-mesh sieve and to be retained on a 60-mesh; 25 grms. of dry coal was used for each charge, and was heated in an electric muffle 2 in. \times 4 in. \times 10 in. The heating tubes were made of 1-inch Mannesmann tubing so arranged that 10 in. of the tube was in the oven. The tubes were charged with coal, exhausted, filled with dry nitrogen, and the open end of the delivery tube closed by a rubber cap. They were inserted in the oven when a steady condition of temperature in the muffle had been attained, as indicated by no fluctuation greater than 5° C. being recorded in an hour, and the delivery tube was immediately connected to a vessel containing nitrogen. The time requisite for the whole charge to reach the temperature of the oven was found to be 30 mins. when the oven was stabilised at 450° C., 40 mins. at 400° C., and 45 mins. at 350° C. The tubes were heated one by one, and throughout the period the variation of the temperature of the oven did not exceed $\pm 5^\circ$ C. At the end of the experiment the exit tube of the charged tube was attached to a nitrogen supply, the heating tube was withdrawn from the furnace, and cooled by gradually lowering it into water. The residue was rapidly weighed, placed in a flask, and covered with carbon tetrachloride. The flask was shaken for three or four minutes, allowed to stand for one hour, the mixture filtered, the residue washed with carbon tetrachloride, dried *in vacuo* for two hours, and weighed. Repeat determinations of the loss of weight of a coal under definite conditions agreed within 0.2%.

Pyridine extractions.—The portion of the coal or residue which passed an 80-mesh sieve but was retained on a 90-mesh sieve was dried *in vacuo* for one hour; 5 grms. was mixed with 5 grms. of ignited sodium chloride, and the mixture extracted in a dry double-walled extraction thimble in an atmosphere of nitrogen, in a Soxhlet apparatus, the end of the condenser being connected with a manometer tube. The temperature was so regulated that the siphon functioned every 12 minutes. The pyridine used distilled entirely between the limits 116° C. and 122° C., and was prepared from the commercial product by fractional distillation and drying over caustic potash. The extraction was conducted with 250 c.c. of pyridine as follows:—The apparatus was kept continuously at work for 120 hours, then allowed to cool, and a fresh charge of pyridine

placed in the flask. The charge in the thimble was well stirred and the apparatus re-charged with nitrogen, after which the extraction was continued for a further 72 hours. The charge in the thimble was again mixed, and after re-filling with nitrogen the apparatus was kept at work for a final period of 48 hours. The united pyridine extracts were concentrated to a convenient bulk in an atmosphere of carbon dioxide, and the cold residue was slowly poured into an excess of 1:1 hydrochloric acid made with air-free water, the precipitate filtered, washed until it was free from acid, dried for eight hours *in vacuo*, and weighed.

A definite weight of the dried extract was extracted on the water-bath with pure dry chloroform in a similar apparatus; in this case the extractions were continued for at least one day from the time the chloroform in the extraction chamber had become colourless—a stage generally reached about 48 hours from the commencement of the process. The chloroform solution was filtered through a tared paper and the weight of any β -cellulose mechanically carried over during the extraction was added to that determined by weighing the contents of the thimble after it had been dried *in vacuo* for 2 hours. The amount of β -cellulose present in any coal or residue is based on the direct weight obtained by the above method. The resinic matter was determined by distilling off the chloroform in a tared flask and weighing the residue, but the results for resin are taken as the difference between the amounts of pyridine-soluble and the amount of β -cellulose. The investigation necessitated the preparation of comparatively large amounts of the β -cellulosic and resinic substances. These were prepared by extracting in an atmosphere of nitrogen a definite amount of coal contained in a metal flask which was heated on an air bath and the pyridine extract was subsequently extracted with chloroform in a Soxhlet apparatus.

Volatile matter was determined by heating 1 grm. of coal etc. in a platinum crucible for seven minutes in an electric muffle maintained at the temperature specified.

Sulphur was determined by the Eschka method.

Nitrogen was determined by the modified Gronig Kjeldhal method. The ammonia was distilled into 10 c.c. of standard acid, the excess of which was determined by titration with *N*/10 ammonia solution, using cochineal as indicator.

Method of calculation.—All results, unless otherwise specified, are calculated to an ash-free and dry basis. Such figures are subsequently referred to as based on the "coal substance."

Pyridine extraction of the original coal.—In the course of the study of the behaviour of the coals

vals of heating. In nearly every case an enhanced amount of the coal substance was soluble in pyridine, a result in agreement with the observations of Harger (this J., 1914, 389, 393). Since it was found necessary to maintain the charge of coal for half an hour at a temperature of 450° C. before the charge was in thermal equilibrium with its surroundings, the rate of the decomposition of the coal substance was based upon the residue resulting from placing the heating tube for half an hour in the muffle regulated to maintain a constant temperature of 450° C. During this period the temperature of the muffle fell to 370° and then gradually rose to 450° C., which temperature was reached 25 mins. after the insertion of the tube. The amounts of the various substances present in the residues thus obtained are given in Table II.

TABLE II.

Constituents in residue from half-hour treatment of the coals at 370°—450° C. % Constituent in residue from ash-free, dry coal.

Coal.	Pyridine-soluble.	α -Cellulosic.	β -Cellulosic.	Resinic.
No. 2 Llantwit ..	31.97	68.03	24.26	7.71
No. 3 Rhondda ..	30.98	69.02	19.77	11.21
No. 2 Rhondda ..	28.41	71.59	17.56	10.85
2 ft. 9 in. ..	31.99	68.01	20.07	11.92

The amounts of the constituents determined by direct extraction of the virgin coal are given in Table III., from which it is evident that the enhanced values of the pyridine-soluble figure is greater in the case of the true coking coals.

TABLE III.

Percentage of constituents in the coal substance determined by direct extraction of the coal.

Coal.	Pyridine-soluble.	α -Cellulosic.	β -Cellulosic.	Resinic.
No. 2 Llantwit ..	28.06	71.94	18.41	9.65
No. 3 Rhondda ..	33.37	66.63	19.30	14.07
No. 2 Rhondda ..	7.62	92.38	2.69	4.93
2 ft. 9 in. ..	21.08	78.92	16.74	4.34

The different properties of these coals are due rather to the nature of their various constituents than to the quantity present. Further work on the nature of the constituents is proceeding. Meanwhile the following characteristics reveal marked differences in the constituents from the different coals.

TABLE IV.

Analysis of β -cellulosic constituents.

	Analysis of β -cellulosic constituents.				Analysis of resinic constituents.			
	No. 2 Llantwit.	No. 3 Rhondda.	No. 2 Rhondda ex $\frac{1}{2}$ hour residue, 450° C.	2 ft. 9 in. ex $\frac{1}{2}$ hour residue, 450° C.	No. 2 Llantwit.	No. 3 Rhondda.	No. 2 Rhondda ex $\frac{1}{2}$ hour residue, 450° C.	2 ft. 9 in. ex $\frac{1}{2}$ hour residue, 450° C.
C	77.56	80.50	83.77	83.08	83.37	82.80	86.16	85.76
H	4.34	4.06	4.95	4.72	7.71	6.97	6.31	6.53
O	14.91	12.92	8.58	12.20	4.99	8.54	6.53	7.71
S	2.19	0.57	0.83	—	2.69	0.32	—	—
N	1.00	2.01	1.87	—	1.24	1.37	1.00	—
Ratio, C/H ..	17.86	20.12	16.92	17.60	10.81	11.89	13.66	13.12
Volatile 900° C. ..	37.50	27.01	28.69	—	80.98	69.45	64.21	—

at 450° C. anomalous results were obtained for the amount of substance extracted by pyridine from the residues obtained after comparatively short inter-

The above analyses reveal the same gradation in nature of the constituents as exists amongst the coals, and again indicate that No. 3 Rhondda is

intermediate in nature between the gas-making coals and the true coking coals, but no generalisations as to the various factors determining the coking qualities are evident from a survey of these figures.

An endeavour was made to elucidate the causes of the variation of the nature of the coke produced by

particles comprising the cokes had very rounded edges. The whole of the various charges of the No. 2 Rhondda were easily poured from the tubes and the particles of the residue were possessed of sharp unaltered edges—a fact which leads one to believe that no softening of the coal had taken place at this temperature.

TABLE VII.
Percentage loss of weight of coal substance, 450° C.

Coal.	Hours of heating.							
	1	1½	2½	3½	5½	9½	12½	24½
No. 2 Llantwit	3.85	11.09	—	12.47	13.68	14.17	—	15.14
No. 3 Rhondda	3.36	8.13	8.82	11.42	12.43	13.53	—	15.81
No. 2 Rhondda	1.86	5.07	8.87	—	9.81	—	10.06	10.16
2 ft. 9 in.	2.38	8.28	—	11.07	11.76	—	11.98	12.01

these coals by investigating the thermal stability of the components. Considerable differences in the stability of the coals were shown by the behaviour at 350°, 400°, and 450° C. for varying intervals of time. The results for the percentage loss of weight under varying conditions are given in tables V., VI., and VII.

TABLE V.
Loss of weight of coal substance at 350° C.

Coal.	Hours of heating.*			
	1½	2½	4½	10½
No. 2 Llantwit	0%	0%	0%	0.94
No. 3 Rhondda	2.14	2.21	3.66	5.94
No. 2 Rhondda	0.85	1.25	—	2.27
No. 2 Rhondda	0.91	1.34	2.00	2.43

* From insertion of tube into oven stabilised at 350° C.

In every case the whole of the residue was readily emptied out of the tube, it exhibited no sign of cohesion, and the edges of the individual particles were as sharp as those of the original charge. These facts preclude the idea that the residue had melted or softened. Anderson gave 300° C. as the softening point of the Scotch coking coals which he examined, whereas the above coals do not soften or coke at 350° C. The above figures show that the No. 2 Llantwit is the least stable of the coals examined, and that the other two are of the same order of stability at this temperature.

TABLE VI.
Loss of weight of coal substance at 400° C.

Coal.	Hours of heating				Remarks.
	1½	2½	4½	10½	
No. 2 Llantwit	5.93	7.73	9.52	12.77	Residue had all coked.
No. 3 Rhondda	2.6	3.73	4.26	8.43	
No. 2 Rhondda	2.15	2.54	3.61	4.77	No residue had coked.

No. 2 Llantwit thus decomposes progressively at 400° C. to a very considerable extent, No. 3 Rhondda more slowly (by comparison), and No. 2 Rhondda only very slowly. This may represent the decomposition of certain constituents unstable at 400° C. which are present in different amounts in the three coals. The cokes from the first two coals were very brittle and possessed a very bright appearance—one might term them "Coal bright." The individual

These figures reveal the rapid initial decomposition of the coals at 450° C. and the greater volatility of the first two coals compared with the coking coals. The dense coking coals approach their maximum volatile figure at this temperature in a comparatively short time, whilst the other two coals approach it more gradually. The residues were coked in every case with the exception of that residue resulting from the half-hour treatment of the No. 2 Rhondda; this had cohered into lumps in various places, and there were definite signs that an incipient softening of the coal had taken place.

From the above results around 400° C. may be taken as the temperature of coke formation for the porous coking coals, and 430°—450° C. for the dense coking coals. In view of these results the thermal behaviour of the β -cellulosic and resinic constituents in the coals was investigated at 450° C.

Relative stability of β -cellulosic and resinic constituents.—No. 2 Rhondda.

TABLE VIII.
Constituents in residues from coal at 450° C.

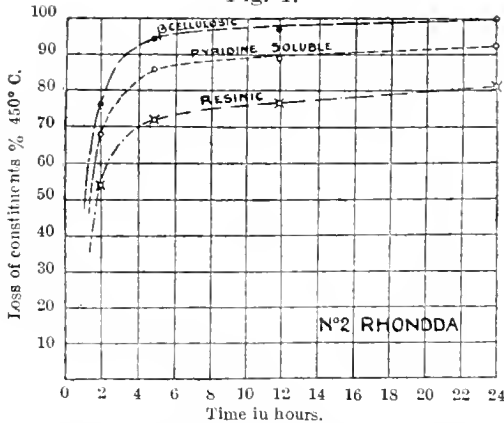
Period, hrs.	In residue.			In residue ex 100 coal substance.		
	Pyridine soluble.	β -Cellu-losic.	Resinic.	Pyridine soluble.	β -Cellu-losic.	Resinic.
1	26.66	16.48	10.18	28.41	17.56	10.85
1½	21.33	13.34	7.99	22.04	13.79	8.25
2	9.23	4.22	5.01	9.18	4.19	4.99
5½	4.10	0.98	3.12	4.04	0.96	3.08
12½	3.2	0.6	2.6	3.14	0.58	2.56
24½	1.9	nil	1.90	2.10	nil	2.10

The rapid decrease in the amount of substance soluble in pyridine is thus due in a greater degree to the destruction of the β -cellulosic than to the destruction of resinic substance. The relative ease of decomposition of the several constituents is more clearly indicated in the graphs in Fig. 1, which have been arrived at by subtracting from the half-hour values the amounts of any constituent present at the end of a particular period of heating, and expressing this difference as a percentage of the amount present in the half-hour residue.

The curves for both the β -cellulose and resinic constituents tend to exhibit a point of flexure at the end of the 5-hour period, no doubt due to the presence of two types of compound in each of these constituents—one readily decomposed at 450° C., and the other more stable. The fact that the point of flexure in the curve for the β -cellulose occurs when more than 90% of this constituent has been decomposed suggests that this

type of constituent in the No. 2 Rhondda approximates to a homogeneous substance. An alternative explanation of the point of flexure is that the slower rate of change of these compounds in the later periods is due to the decomposition of secondary products still soluble in pyridine which arise from the destruction of the primary substance.

Fig. 1.



Analyses of several of the β -cellulosic and resinic components isolated from various residues are given in Table IX.

TABLE IX.

Residue.	β -Cellulosic.		Resinic.	
	$\frac{1}{2}$ hour.	2 hours.	$\frac{1}{2}$ hour.	2 hours.
C	83.77	83.58	86.16	88.65
H	4.95	4.89	6.31	5.95
Ratio, C/H ..	16.92	17.09	13.66	14.91

These figures confirm the homogeneity of the β -cellulosic constituents in so much as no great difference is produced by partial destruction. The resinic substances appear to be less homogeneous than the β -cellulosic constituents; the more stable resinic has a greater C/H ratio than the resinic less stable at this temperature.

In so much as the β -cellulosic substances decompose more rapidly than the resinic bodies, the ratio of the resinic to β -cellulosic substance must increase as decomposition of the coal proceeds.

Nitrogen and sulphur.

The nitrogen and sulphur were estimated in the various residues, and the amounts present in the residues from one hundred parts of the coal substance were calculated from the figures thus obtained. Details of the results are given in Table X.

TABLE X.

Nitrogen and sulphur in residues ex 100 grms. coal substance.—No. 2 Rhondda, 450° C.

Hours of heating.				N. %	S. %
Original coal	1.42	1.95
1½	1.38	1.99
2½	1.25	1.90
5½	1.21	1.79
12½	1.23	1.57
24½	1.25	1.61

The decomposition at 450° C. of the nitrogenous substance in the coal thus results in the elimination of approximately 15% of the total nitrogen in the

coal. The nitrogenous substance decomposed is markedly unstable at this temperature, the decomposition being completed in the first two hours. The subsequent figures agree within the limits of experimental error. The continuous elimination of sulphur over the first period of twelve hours indicates the gradual decomposition of sulphur compounds moderately stable at 450° C.

TABLE XI.

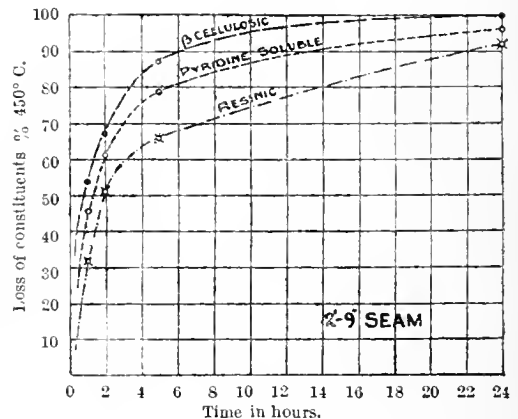
Two Foot Nine seam. Constituents in residues at 450° C.

Period, hrs.	Percentage to residue.			Amount in residues ex 100 coal substance.		
	Pyridine-soluble.	β -Cellulosic.	Resinic.	Pyridine-soluble.	β -Cellulosic.	Resinic.
$\frac{1}{2}$	31.94	20.04	11.90	31.99	20.07	11.92
1½	18.40	9.78	8.62	17.36	9.22	8.14
2½	13.68	7.32	6.36	12.47	6.51	5.79
5½	7.43	2.92	4.51	6.72	2.64	4.08
24½	1.12	nil	1.12	1.01	nil	1.01

The curves drawn from these figures are generally similar to those obtained from the figures of Table VIII.

The percentage loss of the various constituents with lapse of time have been calculated as for fig. 1, and are plotted in fig. 2.

Fig. 2.



These curves emphasise the slower initial decomposition of the various constituents of this coal compared to the No. 2 Rhondda. The apparent homogeneity of the β -cellulosic constituent is indicated, but is not quite so pronounced as in the case of the No. 2 Rhondda. These coals exhibit a close similarity in their behaviour and mode of decomposition at 450° C. Analyses of various constituents isolated from several of the residues are given in Table XII.

TABLE XII.

	β -Cellulosic.		Resinic.	
	1½ hours residue 450° C.	5½ hours residue 450° C.	1½ hours residue 450° C.	$\frac{1}{2}$ hour residue.
C	83.08	82.85	87.80	85.76
H	4.72	4.19	6.43	6.53
Ratio, C/H ..	17.60	19.77	13.67	13.13

The homogeneity of the β -cellulosic constituent is not so clearly indicated by these results.

Nitrogen and sulphur.

The amounts of nitrogen and sulphur in the residues calculated on the basis of the residue arising from 100 parts of coal substance are detailed in Table XIII.

TABLE XIII.

Nitrogen and sulphur in residues ex 100 grms. coal substance.

Two Foot Nine. Temperature 450° C.

Hours of heating.				Nitrogen in residue.	Sulphur in residue.
Original coal	1.34	0.63
1½ hours	1.34	0.63
3½	1.26	0.63
5½	1.29	0.69
12½	1.31	0.67
24½	1.34	0.60

It is evident that in the case of this coal the early stages of decomposition are not associated with the destruction of molecules containing nitrogen or sulphur to any appreciable extent.

No. 3 Rhondda S. crop.

TABLE XIV.

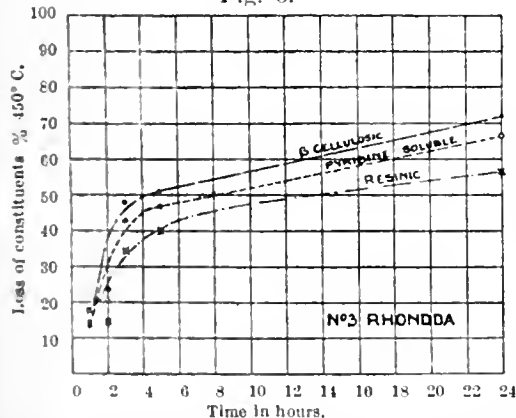
Percentage of constituents in residues obtained at 450° C.

Hours of heating	Percentage in residue.			Amount in residue ex 100 coal substance.		
	Pyridine-soluble.	β -Cellulose.	Residue.	Pyridine-soluble.	β -Cellulose.	Residue.
1½	31.22	19.03	11.20	30.98	19.77	11.21
2½	28.20	18.34	9.86	26.34	17.14	9.20
3½	26.62	16.29	10.32	24.60	15.03	9.57
5½	19.66	11.43	7.58	17.66	10.27	7.39
9½	18.36	10.78	5.30	16.33	9.61	6.72
24½	12.14	6.41	5.73	10.37	5.48	4.89

These results show that β -cellulose is associated with the resinic substances throughout the period of heating, and that the decrease in the amount of these substances present with the variation in the time of heating is far less rapid than is the case in the other two coals considered.

The percentage losses of the constituents with variation in the duration of heating, calculated as before, are plotted in Fig. 3.

Fig. 3.



It is evident that the pyridine-soluble constituents of this coal are more stable than the corresponding substances in the Two Foot Nine and No. 2 Rhondda, for in the latter coals approximately 95% of the

pyridine-soluble constituent is decomposed in 24 hours against only 66% in the case of this coal. A point of inflexure in the curves for the β -cellulose substances occurs when 50% of this substance has been decomposed; the curve for the resinic bodies tends to exhibit no definite change of direction. These facts can be interpreted on the basis that the β -cellulose comprises nearly equal amounts of substances of different stability at 450° C., whilst the resinic bodies approach homogeneity. Contrasting the behaviour of these substances with the corresponding constituents of the Two Foot Nine and No. 2 Rhondda, the marked stability of the β -cellulose in this coal is very evident; moreover, the ratio of the resinic bodies to the β -cellulose substances in the residues increases much more slowly than in the case of the two first-named coals; this is due not only to the greater stability of the β -cellulose but also to a corresponding greater stability of the resins in this coal as compared with the resinic constituents contained in the other coals—a fact readily evident by comparison of the resinic curves for each coal, and emphasised by the fact that the extent of decomposition of the resins in the No. 3 Rhondda only reaches in 24 hours the same value as is reached in the case of the other coals in 2½ hours. By prolongation of the curves for the β -cellulose and resinic bodies of this coal it becomes evident that the whole of the β -cellulose will be decomposed at the end of 48 hours' treatment at 450° C., and that when this has taken place the resins will have undergone decomposition to an extent of 66% of the amount originally present in the coal.

Analyses of β -cellulose and resinic substances isolated from various residues are given in Table XV.

TABLE XV.

Residue.	β -Cellulose.		Resinic.	
	Original.	2 hours 450° C.	Original.	24 hours 450° C.
C	80.50	83.03	82.80	83.16
H	4.00	3.76	6.96	6.16
Ratio, C:H ..	20.12	17.43	11.88	12.47

The approach of the resins to homogeneity is evident from the above figures.

Nitrogen and sulphur.

TABLE XVI.

Amounts of N and S present in the residues from 100 parts of coal substance 450° C.

Hours of heating.				Nitrogen.	Sulphur.
Original coal substance	1.45	0.64
1½	1.20	0.50
3½	1.28	0.50
5½	1.27	0.51
9½	1.28	0.54
24½	1.22	0.56

It is evident from these results that approximately 14% of the nitrogen present is eliminated from this coal at 450° C., and since this occurs within the first 1½ hrs. of treatment the nitrogenous constituent decomposed is relatively unstable at this temperature. Associated with the decomposition of this nitrogenous substance is the elimination of 20% of the total sulphur present in the coal, no doubt due to the destruction of an organic substance containing sulphur of the same order of stability as the nitrogenous constituent. The elimination of pyritic sulphur at this temperature is not very probable.

No. 2 Llantwit.—Temperature 450° C.

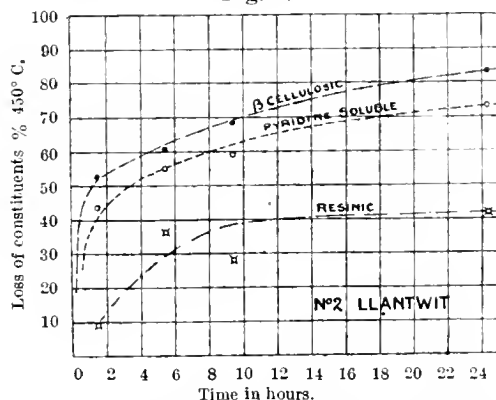
TABLE XVII.

Percentage of constituents in residues obtained at 450° C.

Hours of heating.	Percentage in residue.			Amount in residues ex 100 coal substance.		
	Pyridine-soluble.	β -Cellulosic.	Resinic.	Pyridine-soluble.	β -Cellulosic.	Resinic.
1	31.38	23.80	7.56	31.97	24.26	7.71
1½	19.04	11.67	7.37	18.02	11.04	6.98
5	15.67	10.34	5.33	14.42	9.50	4.92
9	14.38	8.35	5.63	13.16	7.63	5.13
24	9.49	4.50	4.99	8.58	4.07	4.51

The general similarity of behaviour during the 24 hours' heating of this coal and the No. 3 Rhondda is thus evident. Residues arising from these two coals contained β -cellulosic constituents associated with the resinic substances throughout the 24 hours' heating; the ratios of resinic to β -cellulosic constituents in the residues are of the same order, and indicate a greater stability of the resinic as compared to the β -cellulosic substances. The stability of these latter constituents are of the same order and markedly distinct from that of the corresponding portions of the Two-Foot-Nine Seam and the No. 2 Rhondda. The No. 2 Llantwit undoubtedly decomposes faster in the earlier stages than does the No. 3 Rhondda. The percentage loss of constituents is represented graphically in Fig. 4.

Fig. 4.



from which definite points of flexure are evident in the curves for both the resinic and β -cellulosic substances. The β -cellulose appears to consist of two types of substance in equal amounts, and compared with that of the No. 3 Rhondda this constituent is less stable since it is decomposed to a 10% greater extent for the same duration of heating than in the case of the latter coal. The more stable portion of the resinic substances is more stable than the stable resin of the No. 3 Rhondda, and it represents a resinic practically stable at 450° C. Subsequent results of this investigation reveals a marked difference in properties of these two types of resin.

TABLE XVIII.

Residue.	Original.	5 hours 450° C.
C	83.37	84.53
H	7.71	7.15
Ratio, C/H	10.81	11.82

The analyses of resinic substances isolated from various residues are given in Table XVIII.

Nitrogen and sulphur.

TABLE XIX.

Amounts of N and S present in the residues from 100 parts of coal substance. No. 2 Llantwit, 450° C.

Hours of heating.	Nitrogen.		Sulphur.	
	%		%	
Original	1.49	2.08	1.40	1.72
1	1.40	1.72	1.39	1.56
5	1.42	1.40	1.42	1.40
24	1.38	1.22	1.38	1.22

These results show that a nitrogenous substance is decomposed in the early stages of the decomposition of the coal substance. Constituents containing "organic" sulphur are undergoing decomposition throughout the whole period of decomposition.

Examination of stability of β -cellulose and resinic at 350° C., No. 2 Llantwit.

The examination of the residues resulting by heating charges of the No. 2 Llantwit at 350° C. and the No. 3 Rhondda at 400° C. for various times was next undertaken with a view to determine with greater exactitude the difference in stability of the β -cellulose relative to the resinic constituents in each of these coals. The following results were obtained, from which were calculated the amounts of the constituents present in the residues resulting from 100 parts of coal substance.

TABLE XX.

Constituents in residues from No. 2 Llantwit at 350° C.

Hours of heating.	Percentage in residue.			Amount in residue ex 100 coal substance.		
	Pyridine-soluble.	β -Cellulosic.	Resinic.	Pyridine-soluble.	β -Cellulosic.	Resinic.
1½	23.74					
2½	20.90*					
4½	21.79	12.47	9.32	22.55	12.90	9.65
10½(a)	29.36	19.98	9.38	28.80	19.60	9.20
(b)	27.21					
	27.00	17.70	9.30	26.87	17.62	9.25

* On repeating heating operation loss of wt. = 2.11%.

The anomalous results in the case of the residues resulting in the two early periods of heating are rather remarkable. The total repetition of the determination for a period of 2½ hours gave practically the same results. A similar anomaly is evident in the case of the No. 3 Rhondda at 450° C. The effect of various temperatures below the temperature of decomposition of the coal upon the amount of substance extractable from a coal is under investigation; until this work is completed the anomalous behaviour may be explained as arising from the following causes: The amount of substance soluble in pyridine obtained from any coal that has been heated above its "decomposition point" is the resultant of two opposing factors; one, due to the "unbinding" of the coal structure, results in an increase of soluble constituents; the other produces a decrease of pyridine-soluble constituents by virtue of the destruction of substances which in the unheated coal are soluble in pyridine. The net result will depend upon the relative velocity with which these opposing factors operate. It is apparent from the above results that the β -cellulosic constituent is decomposed more rapidly than the

resinic substances, which appear to be practically stable at this temperature. No doubt the portion of the β -cellulose here destroyed is that portion rapidly decomposed at 450°C .

No. 3 Rhondda, 400°C .

TABLE XXI.

Constituents in residue from No. 3 Rhondda,
Temperature 400°C .

Hours of heating.	Percentage in residue.			Amount in residue ex 100 coal substance.		
	Pyridine- soluble.	β -Cellu- lose.	Resinic.	Pyridine- soluble.	β -Cellu- lose.	Resinic.
1	32.53	21.23	11.30	32.86	21.46	11.66
2	38.48	25.70	12.76	37.63	25.14	12.14
4	36.60	23.60	13.00	35.60	22.96	12.64
10	22.60	11.85	10.75	21.02	11.03	10.00

The β -cellulose is thus more readily decomposed than the resinic substances. The amount of resinic substance found in the coal substance by direct extraction was equal to 14.07%; the half-hour residue at 450°C . contained only 11.21%. At least 2 or 3% of the resinic substance is therefore rapidly decomposed. The increase of the values for the β -cellulose in the residues resulting from heating the coal for periods up to 4½ hours at 400°C . indicates that the "unbinding" of the coal substance renders an enhanced amount of β -cellulose soluble in pyridine. This is very unstable at 450°C ., since the half-hour residue at this temperature only contains 19.93%. It is hoped that work now proceeding on the extraction of the various coals by Harger's method (heating with pyridine at about 150°C .) will give more reliable data as to the amounts of the various substances present in the coals. The values obtained for the various half-hour residues are shown by the experiments now to be described to constitute a definite basis for the discussion of the various series of results at 450°C . and their bearing upon the coking qualities of the coals.

TABLE XXII.

Pyridine and volatile matter evolved at 450°C .
from the residues from the pyridine extraction of
"cokes."

Residue.	1 hour loss at 120°C .	Total volatile 450°C .	Total pyridine evolved, 450°C .	Equivalent volatile in original coal.
½ hour 450°C .				
No. 2 Llantwit ..	0.50	10.21	7.56	1.41
2 hours 350°C .				
No. 2 Llantwit ..	0.50	18.26	7.75	8.21
½ hour 450°C .				
No. 3 Rhondda ..	0.58	9.35	3.14	4.14
½ hour 450°C .				
No. 2 Rhondda ..	0.31	1.64	1.41	0.12

To determine if the decomposition of these coals was in conformity with the results obtained by Wheeler with other coals, namely, that decomposition below 500°C . was concerned with the destruction of the pyridine-soluble substances, that portion of the half-hour residue (450°C) insoluble in pyridine was washed repeatedly with dilute hydrochloric acid and then with water until it was free from acid, and the washed residue dried for eight hours at 105°C . in *vacuo*. One gram. of the residue was then heated in a porcelain combustion boat at 120°C . in nitrogen for one hour, and the loss of weight determined. The boat and its contents were next heated at 450°C . in a current of nitrogen for three hours, the products evolved were collected in standard acid, and the boat and its contents cooled in nitrogen.

Other workers have suggested that pyridine combines with the coal substance. A very pronounced smell of pyridine was observed in blank experiments at 450°C ., moreover a solution in water of the vapours evolved gave a distinct precipitate with picric acid; consequently the equivalent of the acid neutralised was calculated in terms of pyridine. The results obtained with the various residues are given in Table XXII.

Fig. 5.

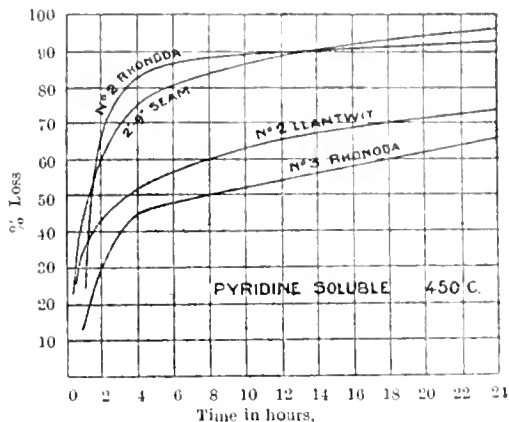


Fig. 6.

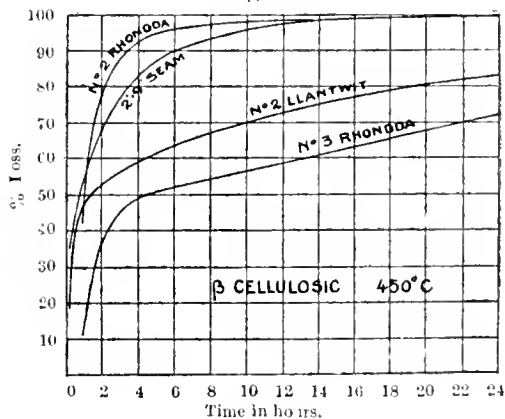
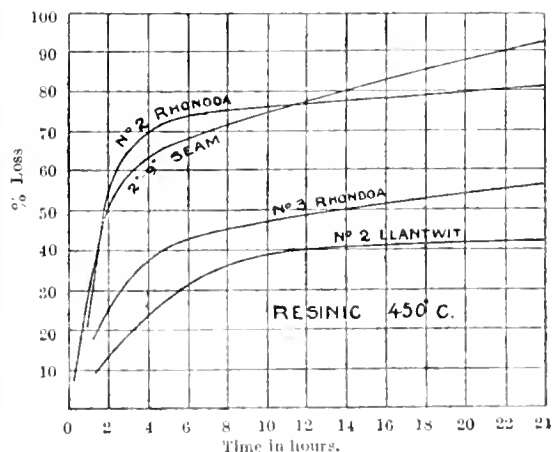


Fig. 7.



The individual coals yielded no products soluble in 10% aqueous caustic potash, and hence no ulmin

compounds are present in these coals. The above results show that after removal of that portion of the residue soluble in pyridine the resulting residue of the No. 2 Llantwit heated to 450° C. contains only a small amount of products decomposable at 450° C. The higher volatile figure for the two-hour residue at 350° C. is due to the β -cellulosic constituents not being rendered soluble, or not being produced as a secondary product by preheating. The close agreement between the pyridine content of the two residues from this coal indicates a similarity of chemical nature of that portion of these residues stable at 450° C., and constitutes direct evidence of the union of pyridine with the coal substance. Moreover, the decrease in "acidity" of the corresponding portions of the other coals is evident from the decrease in the amounts of pyridine in the residues. The high volatile figure of the half-hour residue of the No. 3 Rhondda may be due to the same causes as is the higher volatile figure of the 350° C. residue of the No. 2 Llantwit compared with the 450° C. residue from the same coal. The No. 2 Rhondda, from the above results, yields its maximum pyridine-soluble after the half-hour heating at 450° C. and contains no other constituents decomposable below 450° C. It is evident that in the study of the decomposition of the coals concerned at temperatures below 450° C. the major portions, if not all the products undergoing change, are those soluble in pyridine, and further that the volatile matter expelled from any coal during a given period at 450° C. may be regarded as arising from the destruction of the pyridine-soluble substances.

The individual curves representing the decomposition at 450° C. of the pyridine-soluble, β -cellulosic, and resinic constituents of the several coals are grouped in Figs. 5, 6, and 7 respectively, from which the general relative ease of decomposition of these constituents becomes evident.

The main results of this investigation are as follows:—

- (1) The decomposition of the coals at temperatures up to 450° C. results in the destruction of the pyridine-soluble constituents of the coal substance.
- (2) The stability of the pyridine-soluble constituents in the coals examined is in the following order of decreasing stability:—No. 3 Rhondda, No. 2 Llantwit, Two-foot-Nine, No. 2 Rhondda (see Fig. 5).
- (3) The decomposition of the pyridine-soluble constituents results in a more rapid decomposition of the β -cellulosic substances than of the resinic compounds. In all probability the β -cellulosic constituents are the first to decompose.
- (4) Two types of β -cellulosic and resinic compounds occur in each of the coals examined; (a) one type is readily decomposed at 450° C., (b) the other type is comparatively stable at this temperature.
- (5) The typical coking coals contain an approximately homogeneous β -cellulosic class of substance readily decomposed at 450° C., whilst the other coals examined contain nearly equal amounts of the two types enumerated under (4) (see Fig. 6). These facts are generally characteristic of the resinic substances in these coals, although the homogeneity of the resins is not so well marked in the true coking coals (see Fig. 7).
- (6) The stability of the β -cellulosic relative to the resinic compounds is more pronounced in the No. 3 Rhondda and No. 2 Llantwit than in the typical coking coals.
- (7) The progressive decomposition of the β -cellulosic and resinic substances in the case of the coking coals results in a rapid increase of the

ratio of resinic to β -cellulosic constituents. In the other coals the increase of this ratio is very gradual.

- (8) Progressive decomposition of the coal substance at temperatures not exceeding 450° C. will ultimately result in a residue devoid of β -cellulosic substance but one which still contains resinic substances.
- (9) Nitrogenous compounds in the coal are destroyed; at 450° C. the nitrogen eliminated represents approximately 10% of the total nitrogen present in the coal.
- (10) Sulphur compounds are more readily decomposed in the highly bituminous than in the less bituminous coals, certain of which do not evolve volatile sulphur compounds at 450° C.

Edinburgh Section.

Meeting held at Edinburgh, on February 27, 1920.

DR. D. S. JERDAN IN THE CHAIR.

RATE OF CORROSION OF ALUMINIUM.

BY G. H. BAILEY, D.SC., PH.D.

The author introduced his subject by pointing out that the large bulk of the work done in this direction gave entirely fallacious results owing to the fact that alteration in weight during corrosion afforded in itself no trustworthy information. He then dealt with methods of procedure based upon methods formulated and published (Bailey, J. Inst. Metals, 1913, 9, 79). Having recounted the steps to be taken and the precautions to be observed, he showed that a definite measure of the extent to which a sample of aluminium had been corroded, in terms of the metal so corroded per day per 100 sq. cm. of surface, was given by

$$CR = \frac{3}{8} (S + w - W)$$

where W was the weight of the sheet employed, w its weight after exposure, and S the weight of the precipitate obtained from the corrosive liquid after ignition.

It was pointed out that a prolonged and varied series of experiments had been carried out, extending over a period of nearly ten years, which had fully confirmed the value of this method of estimation.

Typical results were then quoted as to the effect of temperature, time of exposure, concentration of the corrosive medium, deposits on the metal and other variables. For most of the details relating to the method his original paper (*loc. cit.*) may be consulted.*

Diagram 1 summarises the results of experiments carried out with a view to ascertain the effects of concentration of the liquid reagent. It is plotted in logarithmic terms to show more clearly the behaviour of the lower degrees of concentration, and some remarkable effects of higher concentration are also apparent.

Some observations were then made upon the effects observed when the liquids used were at high and even boiling temperatures, under which conditions the phenomena exhibited were by no means comparable with those shown at ordinary temperature, though in many respects considerable analogy was apparent.

[The remainder of the paper follows in *extenso*.]

* A correction has been made in the formula there given for which the author is indebted to the timely criticism of Mr. G. Cecil Jones.

In practice, at ordinary temperatures, the amount of reagent taken is always largely in excess of that which can be reacted upon during the period

imposed by the accepted equations for their reaction with aluminium. But if the residual liquid from this reaction be raised to say 50° C. its activity is restored and a very distinct corrosion takes place in both cases.

Aluminium placed in contact with 200 c.c. of N/1 (3.6%) hydrochloric acid at 95° C. dissolved rapidly and continued to do so over a prolonged period. The liquid became more viscous but remained translucent, until the relationship of the chlorine to aluminium reacted upon, approximated to that required by the formula $Al_2(OH)_2Cl$. It was, however, materially changed in character and had become not only viscous but strictly colloidal. On further continuing the experiment (for the rate of corrosion was still very pronounced) turbidity appeared, and this rapidly grew to a bulky gelatinous precipitate containing very little chlorine and corresponding to the formula $Al_2(OH)_4Cl$. The corrosive activity was still manifest, and apparently it is possible to carry on the reaction indefinitely with a quantity of hydrochloric acid which, at ordinary temperatures, would react only upon a very small fraction of the aluminium exposed.

Similar effects were found with other acids and have been observed by Seligman and Williams (this J., 1917, 409) in the case of acetic acid. The solid product ultimately obtained is hydroxide of aluminium, whilst the free acetic acid (200 c.c. of a 2% solution was used in my experiments) remains practically unchanged in amount and is still active.

Such effects cannot be, of course, of the same order as experiments conducted with a large excess of the acid at ordinary temperatures, and appear to be mainly the results of oxidation through the intermediary of basic salt production and afford a prelude to the last section of my paper.

The protective coating formed during corrosion.

We have seen already that when the exposure of metal to a liquid has been somewhat prolonged there is a very distinct decrease in the rate of corrosion resembling the parallel case of metallic lead. Independent experiments have shown that this cannot be attributed to a loss of concentration in the reagent. Nor is it attributable, otherwise than exceptionally, to the diluent or inhibitive action of the soluble by-products which may be

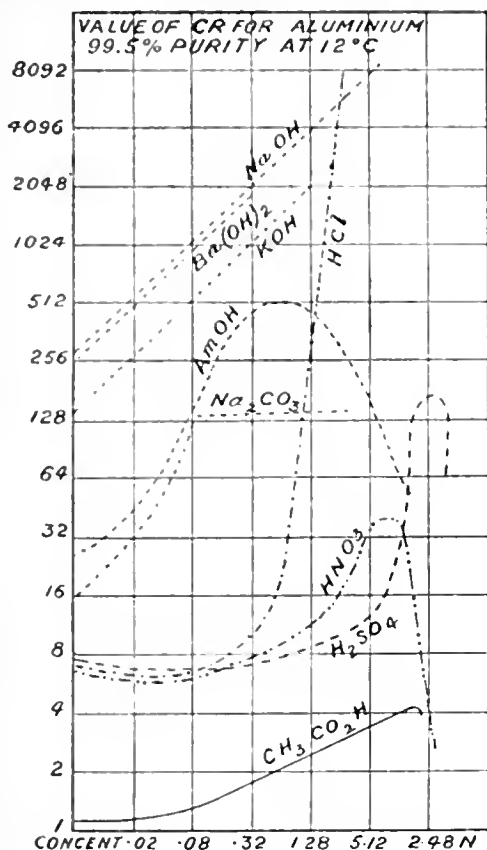


Diagram 1.

of exposure. The question as to when the limit of activity will be reached by reason of the exhaustion of the reagent or the effect of its reduced concen-

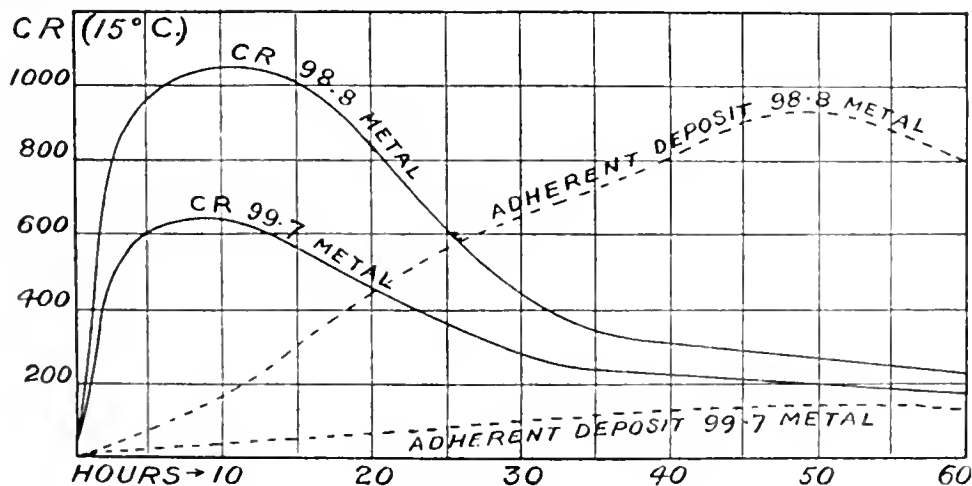


Diagram 2.

tration upon the rate of corrosion is of some interest. For instance, to take two quite different reagents, caustic soda and hydrochloric acid become exhausted and cease to show corrosive action at ordinary atmospheric temperatures at the limit

formed. In mineral acids of medium concentration only soluble products are formed, but in the majority of cases solid products consisting chiefly of alumina and small amounts of the oxides of iron and silicon separate as the result of oxidation and

remain either in suspension or attached to the metallic surface. Thus is formed the protective coating which, of course, interferes with the direct action of the reagent and reduces the rate of corrosion.

Such reduction is much more marked with impure metal than with that of high purity because in the initial stages the rate of corrosion is greater in metal of low purity, so that not only is there more alumina formed but still more of the other oxides.

The result is manifest and I append, merely as one example out of many, that of the course of corrosion when two samples of aluminium, the one of the highest purity, 99.7%, and the other metal of 98.8% purity, containing nearly 0.9% of iron. These were exposed over a prolonged period (nearly four days) to a solution of ammonia of N/1 concentration (1.8% NH_3).

Experimental means were devised for estimating the amount of corrosion and the amount of the adherent deposit at frequent intervals. The accompanying diagram shows the results of the investigation in a typical case. The amount of the adherent deposit is shown by the broken curves which are on a vertical scale such that the maxima are, for 98.8% metal (0.9% Fe) 240 mgrms. per 100 sq. cm. of surface, and for the high purity metal 50 mgrms. The larger of these deposits is a mere film about 0.01 mm. in thickness.

It is evident that if the deposit be allowed to remain upon the metal the rate of corrosion at the end of the period is reduced about five-fold owing to the protection afforded. This opens up possibilities in the way of applications to the arts and manufactures by which the effects of corrosion may be minimised. Already, by the use of special means important developments have arisen from the facility with which a thin oxidised film on the surface of aluminium has afforded perfect insulation under tolerably high tension electric currents, and the same principle may find application in the protection of chemical plant against corrosion. Aluminium alloys containing a small percentage of copper show this property of self-protection against corrosion and open up the possibility of advantage coupled with the application of metal having a much higher tensile strength than that of aluminium itself. Proceeding on the basis laid down, it will be manifest that by modifying the course of procedure and introducing corrections in the expressions made use of in the calculations, metals such as zinc, copper, iron, etc., and even alloys such as brass may be subjected to similar methods of investigation with considerable precision.

Additional.

The following observations at atmospheric temperatures are from various sources and have been nearly all confirmed or corrected by the author of this paper. The results which appear in the foregoing diagram are not included. They have been classified in such a manner as to condense the statement as far as possible, and where values for CR need to be given these are placed in parenthesis and are for 15° C.

I. Substances whose CR is practically negligible.

(a) *Gases*.—Air or oxygen, carbon monoxide and dioxide, chlorine, hydrogen sulphide, sulphur dioxide: all unsaturated with water.

(b) *Organic compounds*.—Acetone, alcohol and alcoholic beverages, antiseptics, benzol, citric acid, ether, oils, fats and fatty acids generally (if free from mineral acid), formaldehyde, glycerin, gelatin, salicylic acid, saliva, tannic and gallic acids, tartaric acid, phenol (even in solid state or alcoholic solution), lactic acid.

(c) *Aqueous solutions*.—Boric acid, salts of alkalis and alkaline-earths, except when alkaline or acid in reaction; also salts of many other metals.

II. Substances whose aqueous solutions show

higher corrosive properties.—Salts of copper, iron, mercury, lead, silver, etc.; hydroxide of calcium and other hydroxides of alkalis and alkaline-earths; sodium aluminate, sp. gr. 1.13, highly alkaline; oxalic acid (4.5% solution, CR=5.9); hydriodic acid; hydrofluoric acid; ammonium chloride (5.35%, CR=5.6); sodium fluoride (saturated solution, CR=9.3); sodium chloride (saturated solution, CR=12.0); calcium chloride and potassium chloride, also iodide, have, however, very slight action.

Liverpool Section.

Meeting held on April 22, 1920.

DR. E. F. ARMSTRONG IN THE CHAIR.

CATALYTIC ACTION AT SOLID SURFACES: SOME FACTORS CONTROLLING THE RATE OF HYDROGENATION OF ETHYLENIC COMPOUNDS IN PRESENCE OF NICKEL.

BY E. F. ARMSTRONG, D.SC., AND T. P. HILDITCH, D.SC., F.I.C.

The technique of the catalytic hydrogenation of liquid organic compounds is sufficiently well understood to permit investigations of the chemical kinetics of the action to be carried out with considerable precision. Of all surface actions it is probably that best adapted to treatment of this kind, since the progress of hydrogenation may be followed directly by measurements of the absorption of hydrogen, whilst the action is suitably rapid and may be studied over a range of temperature at which constancy of the latter factor is comparatively easily maintained.

With the exception of a few measurements of rate of hydrogenation of organic compounds in presence of colloidal palladium or platinum, however, work of this type has hitherto been concerned with the saturation of the mixture of glycerides present in natural oils, where it has been difficult to study the hydrogenation of a single chemical individual.

In most of the studies of the rate of hydrogenation of unsaturated fatty oils which have appeared within the past few years the main action is referred to the unimolecular or "logarithmic" type, in which the same fraction of the substance present at a given moment is changed in successive equal intervals of time, and which is expressed mathematically as

$$dx/dt = k(a-x) \text{ or } k = 1/t \log a/(a-x)$$

where a = total volume of hydrogen required to saturate the glyceride present. For example Fokin (*Z. angew. Chem.*, 1908, 22, 1451, 1492) states that the reduction is to be included in the category of unimolecular reactions, but that the curves often show a straightening out towards the abscissa axis.

Moore, Richter, and van Arsdel (*J. Ind. Eng. Chem.*, 1917, 9, 451), who studied the incomplete hydrogenation of cotton-seed oil, found that the linolein present hydrogenated very much more rapidly than olein, and that the relative speeds of hydrogenation of the two glycerides could be varied within limits by alteration of temperature or pressure. The relation between fall in iodine number and time is stated to take the form of curves showing a general similarity to a logarithmic curve, but "flattening out sooner than expected in every case, as though the catalyst was losing its activity."

Ueno (*J. Chem. Ind. Tokio*, 1918, 21, 749) concluded that the rate of hydrogenation of fatty oils is expressed by the simple unimolecular mass-action equation given above.

Ubbelohde and Svanoe (Z. angew. Chem., 1919, 32, 257, 269, 276) have compared the general efficiency of various types of hydrogenation apparatus, and have noted that linolein yields a mixture of oleins (ordinary olein and "iso-olein") on hydrogenation. Moore has made the further observation (this J., 1919, 320) that ordinary olein (the glyceride of liquid oleic acid) itself is partially transformed during hydrogenation into "iso-oleins" (derived from elaidic and solid iso-oleic acid).

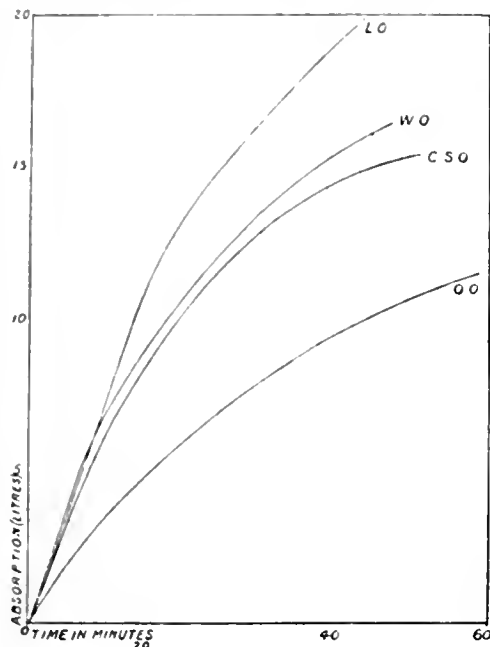


Fig. 1.

Thomas (this J., 1920, 10 r), employing a closed apparatus, has given a mathematical analysis of observations upon the rate of hydrogenation of olive oil (consisting mainly of tri-olein) based upon the unimolecular mass-action equation, and allowing for the simultaneous proceeding of the two hydrogenations—that of linolein and that of olein. The unimolecular constants he obtained are in good agreement with the experimental data.

On the other hand, experiments previously described by us (Proc. Roy. Soc., 1919, A, 96, 137) with olive, cottonseed, linseed and whale oils, in which the hydrogen was continuously passed through the hydrogenation apparatus, and the absorption measured at short intervals by meter readings before and after passage through the latter, showed that oils from various sources are hydrogenated, broadly speaking, at a constant rate until only about 10–20% of glycerides more unsaturated than olein is left, after which an abrupt change occurs. Soon, however, the rate settles down again to approximate constancy; then, after an interval, it gradually declines as a state of saturation is approached.

From the mainly linear nature of the curves thus obtained, it was concluded that the action which was actually measured represented the decomposition by hydrogen of an unstable complex formed between nickel and the unsaturated compounds. The first process was held to be a relatively fast equilibrium action, with the balance strongly in favour of the unassociated constituents, whilst the second (measured) action was relatively slow. It is important to bear in mind that, in any study of the velocity of a chemical change, only the

slowest of a series of actions taking place at the same time is being measured.

In all such cases it is of first importance to obtain the correct chemical interpretation of the mathematical results, for the latter, after all, serve only to interpret the form of the curve which represents the general relation between the chemical action and the time taken, and do not necessarily point to the reason which causes the curve to take a particular form. There is thus at present a conflict of evidence between logarithmic and approximately linear relations for the amount of hydrogen absorbed/time.

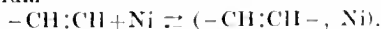
Now, a logarithmic curve represents a continuous decline in the amount of chemical action as time progresses, whereas a true linear curve represents chemical action as independent of the progress of time. Consequently, an approximate linearity indicates that the action, after a given time, has proceeded to a greater extent than if it was of the logarithmic type. It is certain, then, that a true logarithmic action could not approach linearity owing to any abnormal decline of catalytic activity, since this would deflect the curve still further towards the horizontal (time) axis. It should be emphasized that the absolutely linear portion of our curves extends well beyond the limits of experimental error; thus, in the case of various hydrogenations in presence of nickel, the following table shows the relation between the total absorption of hydrogen obtained, and the time taken, to the absorption of hydrogen and time occupied in tracing the initial completely linear portion of the curve:—

Unsaturated compound.	Temp. ° C.	Total hydrogenation.		Initial linear portion.	
		Litres H ₂ absorbed in mins.		Litres H ₂ absorbed in mins.	
Cottonseed oil	180°	30.3	73	12.8	17.7
Linseed oil ..	180°	22.9	59	10.4	13.8
" ..	140°	19.0	69	9.3	24.8
" ..	230°	22.7	56	9.9	9.5
Ethyl cinnamate	140°	12.55	29.7	11.25	23.4
" ..	180°	12.55	22.4	10.25	15.9
Anethol ..	140°	13.15	49.9	7.50	20.3
" ..	180°	13.40	30.3	10.10	18.6

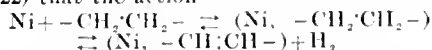
The absolutely linear portion may thus occupy from 40 to over 80% of the total absorption of hydrogen, and from 15 to over 60% of the total time occupied by the action.

On the other hand, it is obvious that the action could not possibly express itself in a linear form throughout its whole course, for the following reasons:—

(i.) As previously pointed out (Proc. Roy. Soc., A., 1919, 96, 137), the diminution of the amount of unsaturated compound must eventually affect the equilibrium



(ii.) The evidence which goes to show (*ibid.*, 1919, 96, 322) that the action



although feeble at 180° C., is in certain cases sufficient to permit of the transference of hydrogen from a saturated molecule of one species to an unsaturated molecule of another indicates that the saturated grouping—CH₂CH₂—when present in sufficient concentration (*i.e.*, towards the close of the action) may commence to compete with the diminished amount of unsaturated compound in the equilibria set up with nickel.

(iii.) As in all these actions, by-products more or less toxic to the catalyst will gradually accumulate, and tend to exert a definite influence (although but small in the hydrogenation of highly purified com-

pounds) upon the activity of the nickel and the speed of the action.

Many investigators in this field have employed a closed type of reaction-apparatus from which no gas escapes during the action, so that any volatile products, toxic or merely inert, must accumulate in the system.

Experiments are quoted later which were conducted with the same apparatus, but in which the customary method of operating (with a free passage of hydrogen) was replaced by a "closed" system in which the hydrogen was simply drawn in to replace absorbed gas as the action proceeded. The results emphasise the extent to which traces of gaseous impurities will accumulate when a relatively large volume of hydrogen is absorbed with only a small closed gas-space, and show that it is necessary under these conditions to pay due attention to the altering concentration of hydrogen in the gas-space.

The study of the hydrogenation of some synthetic ethylenic compounds has been undertaken since these substances are not only entities themselves, but are more easily purified than the glycerides and possess only one ethylenic linkage. The compounds employed, which were carefully purified by fractional distillation in a vacuum before hydrogenation, were: ethyl cinnamate, $C_6H_5.CH:CH.COOC_2H_5$, and anethol, $CH_3O.C_6H_4.CH:CH.CH_3$. For comparison with our present data, the curves (fig. 1) obtained in hydrogenating, at 180° C. in presence of 0.2% of nickel, olive, cotton-seed, linseed, and whale oils are reproduced. Olive oil contains but little linolein, cotton-seed about 50% linolein and 25% olein, linseed oil about 20% olein, and the remainder mainly linolein and linolenin, whilst whale oil contains too complex a mixture of glycerides of acids of varying degrees of unsaturation to be of any utility for the present purpose.

I. Hydrogenation of ethyl cinnamate and of anethol at 140° and 180° C.

100 grms. of each of these compounds was hydrogenated in presence of 0.15 gm. of nickel under conditions comparable to these in the hydrogenation

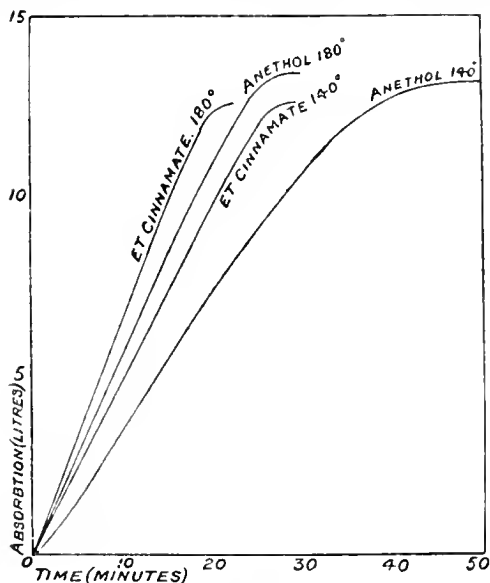


Fig. 2.

tion of oils (*loc. cit.*), a flow of electrolytic hydrogen of 50 litres per hour being maintained in all cases.

The data are shown graphically in fig. 2 (hydrogen

absorbed in litres as ordinates against time in minutes as abscissæ), and are also reproduced in the tables. The latter give details of time in minutes (t) and hydrogen absorption in litres (v), with the respective constants for a linear relation ($K=v/t$) and for a unimolecular action,

$$K' = 1/t \log V'/(V'-v).$$

V' represents the total volume of hydrogen (measured at the temperature of the meter) required to saturate 100 grms. of the organic compound.

For ethyl cinnamate (meter temp. 19°) $V'=13.60$, and for anethol (meter temp. 15.5° C.) $V'=16.00$.

ETHYL CINNAMATE.

At 140° C.				At 180° C.			
t .	v .	K .	K' .	t .	v .	K .	K' .
2.8	1.50	0.536	0.0181	3.1	1.85	0.597	0.0204
5.5	2.80	0.509	0.0182	6.3	4.10	0.651	0.0247
8.1	3.95	0.488	0.0184	9.5	6.20	0.653	0.0278
11.2	5.45	0.486	0.0198	12.7	8.25	0.650	0.0318
14.1	6.90	0.490	0.0218	15.0	10.25	0.645	0.0383
17.2	8.40	0.488	0.0243	19.1	11.95	0.626	0.0480
20.3	9.85	0.485	0.0275	22.4	12.55	0.560	0.0497
23.4	11.25	0.481	0.0326				
26.5	12.40	0.463	0.0398				
29.7	12.55	0.423	0.0375				

ANETHOL.

At 140° C.				At 180° C.			
t .	v .	K .	K' .	t .	v .	K .	K' .
2.9	0.60	0.207	0.0057	2.9	1.70	0.586	0.0168
5.8	1.70	0.293	0.0084	6.0	3.40	0.567	0.0173
8.7	2.90	0.333	0.0100	9.1	5.10	0.560	0.0185
11.6	4.10	0.353	0.0111	12.3	6.85	0.557	0.0197
14.5	5.25	0.362	0.0119	15.4	8.45	0.549	0.0212
17.4	6.35	0.365	0.0126	18.6	10.10	0.543	0.0233
20.3	7.50	0.369	0.0135	21.5	11.50	0.535	0.0256
23.2	8.50	0.367	0.0142	24.4	12.75	0.522	0.0284
26.1	9.50	0.364	0.0150	27.3	13.30	0.487	0.0283
29.1	10.40	0.357	0.0157	30.3	13.40	0.442	0.0261
32.0	11.15	0.348	0.0162				
35.0	11.90	0.340	0.0169				
38.0	12.45	0.327	0.0172				
40.9	12.80	0.313	0.0171				
43.9	13.00	0.296	0.0166				
46.9	13.10	0.279	0.0158				
49.9	13.15	0.263	0.0150				

II. Hydrogenation of ethyl cinnamate and anethol at 180° C.—"closed system."

The hydrogenations at 180° C. were next repeated under somewhat different conditions—the usual exit of the apparatus was connected to the hydrogen supply through one of the meters (the former gas inlet being closed), thus connecting the gas-space above the hydrogenation system to the gas supply, and hydrogen was drawn in as the action proceeded to replace that which had been absorbed.

The results were different from those previously obtained and, when plotted (fig. 3), were found to give curves frequently closely related to the logarithmic type. Experiments were made in each case with undried hydrogen and with hydrogen passed over a long layer of calcium chloride granules, with numerical results as given on next page.

These results are typical of a number of experiments; some give increasing unimolecular "constants," others moderately concordant values, and others (notably those with undried gas) give "constants" which eventually decline considerably. On resuming hydrogenation by the continuous gas-flow method, the usual approximately linear graphs had been employed the rate was found to have declined—i.e., the catalyst had lost activity.

ETHYL CINNAMATE ($V'=13.60$, meter temperature 19°)

Dried hydrogen.				Undried hydrogen.			
t .	v .	K.	K' .	t .	v .	K.	K' .
2	1.04	0.520	0.0173	2	1.27	0.635	0.0213
4	2.31	0.577	0.0202	4	2.49	0.622	0.0220
6	3.46	0.577	0.0211	6	3.55	0.592	0.0219
10	5.65	0.565	0.0233	8	4.46	0.557	0.0216
12	6.70	0.558	0.0245	10	5.13	0.513	0.0206
14	7.62	0.544	0.0255	12	5.77	0.481	0.0199
16	8.53	0.533	0.0268	15	6.50	0.433	0.0188
18	9.39	0.522	0.0283	20	7.51	0.376	0.0174
20	10.14	0.507	0.0297	25	8.20	0.328	0.0160
25	11.90	0.476	0.0361				
28	12.63	0.451	0.0409				
30	12.83	0.428	0.0416				
35	12.88	0.368					

ANETHOL.

Dried hydrogen ($V'=16.20$, meter temp. 19°). Undried hydrogen ($V'=16.00$, meter temp. 15.5°).

t .	v .	K.	K' .	t .	v .	K.	K' .
2	0.77	0.385	0.0106	2	0.95	0.475	0.0133
4	1.69	0.422	0.0120	4	1.65	0.412	0.0118
6	2.47	0.412	0.0120	6	2.21	0.368	0.0108
8	3.18	0.397	0.0119	8	2.73	0.341	0.0097
10	3.81	0.381	0.0117	10	3.18	0.318	0.0096
15	5.04	0.336	0.0108	15	4.00	0.267	0.0083
20	6.06	0.303	0.0102	20	4.58	0.229	0.0073
25	6.80	0.272	0.0095	25	5.01	0.200	0.0065
30	7.40	0.247	0.0088	30	5.35	0.178	0.0059
				35	5.60	0.160	0.0053
				40	5.81	0.145	0.0049
				50	6.13	0.126	0.0042
				60	6.36	0.106	0.0037

remaining experiments were confined to the case of ethyl cinnamate, which was proved to yield ethyl β -phenylpropionate quantitatively both at 180° and 140° C.

It was natural to consider whether the action was likely to proceed by a different mechanism merely by altering the mode of admission of hydrogen to the system, or whether the small amount of gaseous impurities present in the hydrogen or emanating from the organic compound diminished the concentration of hydrogen in the gas-space notably.

If S is the total gas-space available, v the volume of hydrogen absorbed at time t , and P the percentage of hydrogen in the gas employed, the volume of gaseous impurity in the gas-space when v litres have been absorbed is $v(100-P)/P$, so that concentration of H_2 in gas-space at time t

$$= \frac{P}{100S} \left(S - \frac{100-P}{P} \cdot v \right)$$

Assuming the rate of absorption is proportional to this concentration,

$$\frac{dv}{dt} = \frac{KP}{100S} \left(S - \frac{100-P}{P} \cdot v \right)$$

S & P are constant for the same apparatus and supply of hydrogen so that this may be written in the form

$$dv/dt = K''(V'' - v)$$

where $V'' = PS/(100-P)$, i.e. = the total volume of hydrogen which can be absorbed before the gas-space is completely filled with impurities.

$$\text{Hence } K'' = 1/t \log V''/(V'' - v).$$

On attempting to apply this hypothesis to results of the type indicated above, it was found that in some cases more hydrogen had been absorbed than the value V'' permitted, possibly in consequence of diffusion in the connections during the slower stages of the absorption.

A further series of experiments was therefore made, in which carefully dried hydrogen was employed as before, and a long capillary tube inserted between the drying-tube and the inlet to the hydrogenation apparatus.

It was calculated that a flow of only 0.02 litre per minute through the capillary employed required a linear velocity of about 30 cm. per sec., so that back diffusion would be practically suppressed. The volume of the gas-space in the apparatus was measured, allowing for the volume occupied by 100 grms. of ethyl cinnamate at 180° C. (120 c.c.) and found to be 648 c.c. This volume of gas at 180° C. would occupy 412 c.c. at 15° , the temperature of the meter throughout the next series of experiments.

The percentage of hydrogen available for absorption was then calculated as follows:—In an experiment X litres of (dry) hydrogen had been absorbed. A sample of the gas in the gas-space was then extracted and analysed, and from the percentage of hydrogen (p) the volume of hydrogen in the vessel was found (x litres)

$$\text{Whence, } \% H_2 = \frac{X + x}{X + 0.412} = P$$

so that the total volume required to fill the beaker space completely with gaseous impurity ($=V''$) = $9.412P/(100-P)$ litres. Obviously, this expression takes account of impurities originally in the hydrogen and of those resulting from the organic compound, and is only strictly valid if we can assume that the latter accumulate in the same way as the former.

It was found, however, that the residual gas generally contained 2–3% CO_2 , 4–10% calculated as CH_4 & 40–60% H_2 , so that the bulk of the impurity was nitrogen, evidently from the original hydrogen. The "organic" gaseous impurities are therefore relatively small in comparison with the nitrogen.

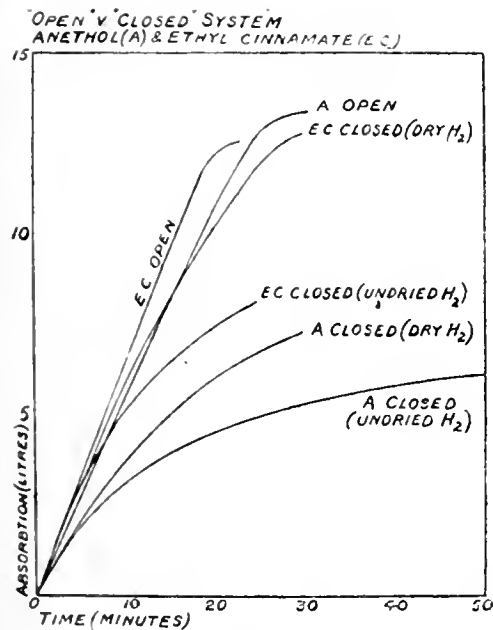


Fig. 3.

In the other cases no perceptible diminution in rate was observed. It may be added that anethol was observed to furnish small quantities of a solid crystalline compound (at present under investigation) as well as the normal product of hydrogenation, dihydroanethol, when treated at 180° , and especially in the "closed" apparatus. Since it was desired to eliminate any possible action other than that of saturation of the ethenoid bond, the

Experiments were then made, using the "closed" apparatus, with 100 grms. of ethyl cinnamate and 0.15 gm. of nickel in each case, employing dried gas from four different cylinders of electrolytic hydrogen, with the capillary tube inserted to eliminate back-diffusion.

The next table contains the results obtained, giving

t , v , and $K = v/t$ as in the previous cases.

K' , the "unimolecular constant" calculated with respect to the total hydrogen absorbable by the ethyl cinnamate present ($V' = 13.40$ at 15°).

p , the percentage of hydrogen in the residual gas.

P , the effective percentage of hydrogen calculated as shown above.

K'' , the "unimolecular constant" calculated with respect to the total hydrogen absorbable when the accumulated impurities would occupy 648 c.c. at 180° (or 412 c.c. at 15°) (V'' calculated from P as described).

The extreme variability of V'' as P increases from 98% upwards is to be emphasised, and is a consequence of the asymptotic nature of the curve corresponding to $V'' = 0.412 P/C (100 - P)$. (V'' becomes ∞ when $P = 100\%$).

Cylinder	t	v	K	K'	p	P	V''	K''
No. 1	2	1.50	0.750	0.0258	75.8	99.21	51.78	0.0064
	4	2.78	0.695	0.0253				0.0060
	6	4.13	0.688	0.0267				0.0060
	8	5.33	0.666	0.0275				0.0059
	10	6.62	0.662	0.0296				0.0060
	12	7.80	0.650	0.0315				0.0059
	14	9.00	0.643	0.0345				0.0059
	16	10.03	0.627	0.0375				0.0059
	18	11.01	0.612	0.0416				0.0058
	20	11.89	0.595	0.0474				0.0057
	22	12.39	0.563	0.0510				0.0054
	24	12.47	0.520	0.0483				0.0050
No. 2	2	0.94	0.470	0.0158	65.1	98.47	26.52	0.0078
	4	2.25	0.562	0.0200				0.0096
	6	3.36	0.560	0.0209				0.0098
	8	4.36	0.545	0.0214				0.0097
	10	5.28	0.528	0.0218				0.0096
	12	6.20	0.517	0.0225				0.0096
	14	7.10	0.507	0.0234				0.0097
	16	7.90	0.494	0.0242				0.0096
No. 3	2	0.88	0.440	0.0115	46.4	98.27	23.40	0.0083
	4	2.21	0.552	0.0196				0.0108
	6	3.57	0.595	0.0207				0.0120
	8	4.78	0.597	0.0239				0.0124
	10	5.90	0.590	0.0252				0.0126
	12	7.08	0.590	0.0272				0.0130
	14	8.12	0.580	0.0289				0.0132
	16	9.04	0.565	0.0305				0.0132
	18	9.96	0.553	0.0329				0.0134
	20	10.78	0.539	0.0354				0.0134
	22	11.44	0.502	0.0379				0.0133
	24	12.14	0.501	0.0428				0.0132
No. 4	2	0.89	0.445	0.0150	36.6	97.89	19.11	0.0103
	4	2.13	0.532	0.0188				0.0128
	6	3.24	0.540	0.0200				0.0134
	10	5.08	0.508	0.0207				0.0134
	12	5.94	0.495	0.0211				0.0135
	14	6.76	0.483	0.0218				0.0135
	16	7.43	0.464	0.0219				0.0134
	18	8.15	0.453	0.0226				0.0134
	20	8.78	0.439	0.0231				0.0134
	25	10.07	0.403	0.0242				0.0130
	32	11.45	0.358	0.0262				0.0124
	40	12.27	0.307	0.0268				0.0112

It is deduced that:—

(i.) The excellently concordant values of K'' show that the curves obtained in a "closed" apparatus are a measure of the diminishing concentration of hydrogen in the gas-space, and have no bearing on the mechanism of the actual hydrogenation;

(ii.) With increasing purity of hydrogen the ratio v/t approaches the mainly linear type always obtained in the hydrogenation of pure compounds with continuous passage of hydrogen;

(iii.) The values of K' increase throughout, but tend to become more concordant as the purity de-

creases (in the above series they would, of course, be constant for $V' = 13.40$, i.e., "effective % H_2 " = 97.0);

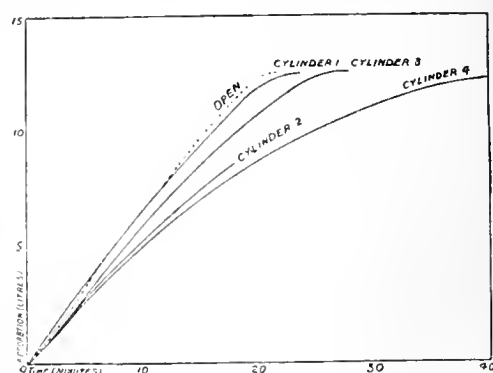


Fig. 4.

(iv.) As the purity of the hydrogen and the speed of the action (measured by v/t) increase, the absolute values of the constants K'' fall; this is simply the mathematical expression of the fact that the "rate of reaction" really measured is that at which the gas-space is being filled with gases other than hydrogen—the rate being smaller the purer the hydrogen.

The data given in this paper therefore show that:—

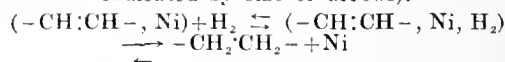
(i.) with free passage of hydrogen an ethylenic compound is hydrogenated in the liquid state at a rate which, for more than half of the complete action, is independent of the concentration of the unsaturated compound

(ii.) in a closed system, the rate is governed by the decreasing concentration of the hydrogen above the liquid, caused by the accumulation of gaseous impurities from the hydrogen and (to a less extent) from the organic compound.

The additional facts now brought forward appear considerably to strengthen the theory that catalytic hydrogenation may be represented by the scheme:—



(relatively fast equilibrium in the general sense indicated by size of arrows).



(relatively slow, measurable action).

It is plain that progressive alteration of the hydrogen concentration must affect the rate at which the second phase of the change proceeds.

In conclusion, without attaching undue finality to Irving Langmuir's suggestive view that "adsorption" is conditioned by a single layer of molecules held at a surface by forces which are none other than chemical in nature (*J. Amer. Chem. Soc.*, 1916, 38, 2221; 1918, 40, 1361), it may be stated that this view of adsorption is indistinguishable from unstable chemical combination of the kind which, according to our view, is concerned in the mechanism of catalysis of organic compounds at the surface of metals or metallic oxides, and which sixteen years ago was similarly used by one of us (*Proc. Roy. Soc.*, 1904, 73, 500) to explain the hydrolytic action of colloidal enzymes.

We would add an expression of our indebtedness to Mr. J. Allan for criticism and general discussion of the results obtained throughout the progress of these experiments.

Communication.

THE ACCELERATION OF VULCANISATION.

BY D. F. TWISS AND S. A. BRAZIER.

Three methods are available for the speeding-up of the vulcanisation process: (1) raising the temperature, (2) increasing the proportion of sulphur relative to rubber, (3) introducing an accelerator.

Effect of temperature.

The effect of alteration of temperature is similar to that for other chemical reactions, the temperature coefficient being between 2 and 3 (for 10°C.). The suggestion has been made that some accelerators, lead oxide in particular, are not genuine catalysts, but that they merely react with part of the sulphur with evolution of heat, thereby raising the temperature of the reacting mass above that of the surrounding heating medium (Seidl, *Gummi-Zeit.*, 1911, 25, 710, 748). The insufficiency of this explanation is evident from the fact that such an effect should be almost negligible at the surface of the rubber in contact with the moulds, whilst in the interior it would be marked; thick slabs also would vulcanise much more rapidly than thin sheets; both these consequences of the theory are contrary to experience. It is quite possible, however, that many vulcanisation accelerators do exert a slight thermal effect in addition to their purely catalytic influence.

The curves given in Fig. 1 represent results of some of our experiments as to the rate of vulcanisation at temperatures ranging from 138°C. (35 lb. steam pressure) to 168°C. (95 lb.) for a mixture of pale crêpe rubber (90) and sulphur (10); the corresponding curves for 128°C. will be found in Fig. 4. Pale crêpe rubber was chosen as showing greater uniformity in rate of vulcanisation than other forms of rubber, and was taken as far as possible from one case. For the introduction of the sulphur a stock mixing of sulphur with approximately an equal weight of rubber was used; the composition of this mixture was checked by analysis, and the correct proportion weighed out for mixing with the remainder of the rubber. In this way the almost inevitable loss of sulphur dust during the customary method of mixing was avoided.

The progress of vulcanisation may be followed in at least three different ways: (a) by the combination of sulphur; (b) by the gradual decrease in the extensibility of the rubber at a definite load (or increase of the load necessary to produce a definite elongation); within the range of the experiments in this paper, this shows a roughly rectilinear relationship with the period of vulcanisation. Unfortunately, this method is influenced to a greater extent than either (a) or (c) by the mechanical treatment of the rubber during mastication and the subsequent mixing operation, increased working or heating having a tendency to exaggerate the extensibility figure. The approximation of the curve to a straight line and the close proximity of extensibility values for duplicate samples (see p. 130 r) are evidence of the considerable degree of accuracy attainable with careful working. For the purpose of comparison between different samples an elongation of 600% (including the original length) at a load of 0.5 kilo. per sq. mm. has been arbitrarily assumed as a standard throughout this paper; (c) by the time required to produce maximum tensile strength.

The last method, although of less importance than might be expected in technical practice, is of considerable value in experimental work as supplying a convenient and rapid method for comparing rates of vulcanisation, e.g., of different rubbers or at different temperatures, the maximum tensile

strength, determined within three days of vulcanisation, being observed with a product containing approximately 5% of combined sulphur calculated on the rubber. The actual value of the breaking strength of a rubber test piece is always more or less fortuitous; however, as vulcanisation beyond the condition necessary for the attainment of the maximum strength causes a very rapid weakening, the position of the maximum is relatively easily determined. The peaked curves in Fig. 1 indicate the position of the maximum rather than the actual magnitude of the values.

It will be observed that the temperature coefficient manifests no tendency to any regular increase or decrease with rise of temperature, the mean value calculated (by all three methods of comparison) from the figures represented in the curves for the rubber-sulphur mixture at 123° to 168°C. approximating to 2.3.* This appears to indicate that the allotropic forms ordinarily present in molten sulphur in relative proportions dependent on the temperature, must possess equal or at least comparable vulcanising activity (see Twiss, Annual Report, 1919, 4, p. 327).

The extent to which the vulcanisation period in technical work can be reduced by raising the temperature is limited by various considerations, not the least of which is the poor thermal conductivity of rubber and the consequent danger of unequal heating, involving local irregularities in the degree of vulcanisation.

Increase of proportion of sulphur.

Many old compounding ingredients and specifics of the rubber trade, often bearing fanciful names, contained—undeclared—considerable proportions of free sulphur; mixtures of sulphur with various waxes and also with antimony sulphide may be quoted as better class examples. As the rate of vulcanisation of rubber-sulphur mixings containing less than, roughly, 10% of free sulphur is directly proportional to the percentage of free sulphur, the effect of such additional sulphur-containing ingredients on the rate of vulcanisation is obvious, and to-day is commonly realised.

For experimental work on the relative rates of vulcanisation of different rubbers or on the effect of various catalysts, the selection of a mixture of rubber and sulphur containing as much as 19% of the latter is advisable, not only because this quantity is more than sufficient to permit the progress of the change to well beyond the characteristic maximum strength, but also because it is sufficiently high to reduce in extent any disturbance arising from slight inaccuracies in the proportion of sulphur present. The possibility must always be borne in mind, however, that the activity of an accelerator may possibly be influenced by the proportion of free sulphur simultaneously present.

Use of a catalyst.

Almost all basic substances can act as catalysts to the vulcanisation process, e.g., magnesium oxide, lead oxide, lime, sodium hydroxide, potassium hydroxide, and also substances such as sodium amide, potassium glyceroxide and sodium phenoxide, which, by interaction with the traces of water invariably present in rubber, are able to give rise to alkalis or bases; typical examples of organic accelerators are piperidine, quinine (the crude alkaloid mixture known as "quinoidine" is commonly used), aniline, naphthylamine, *p*-phenylenediamine, hexamethylenetetramine, anhydroformaldehydeaniline, benzylideneaniline, aldehyde-ammonia, and simple carbon bisulphide or

* On account of the inconveniently rapid progress of vulcanisation at 168°C. and its slowness at 128°C. the measurements at 138°C. to 158°C. are possibly to be preferred; a mean value of 2.6 is then obtained for the temperature coefficient.

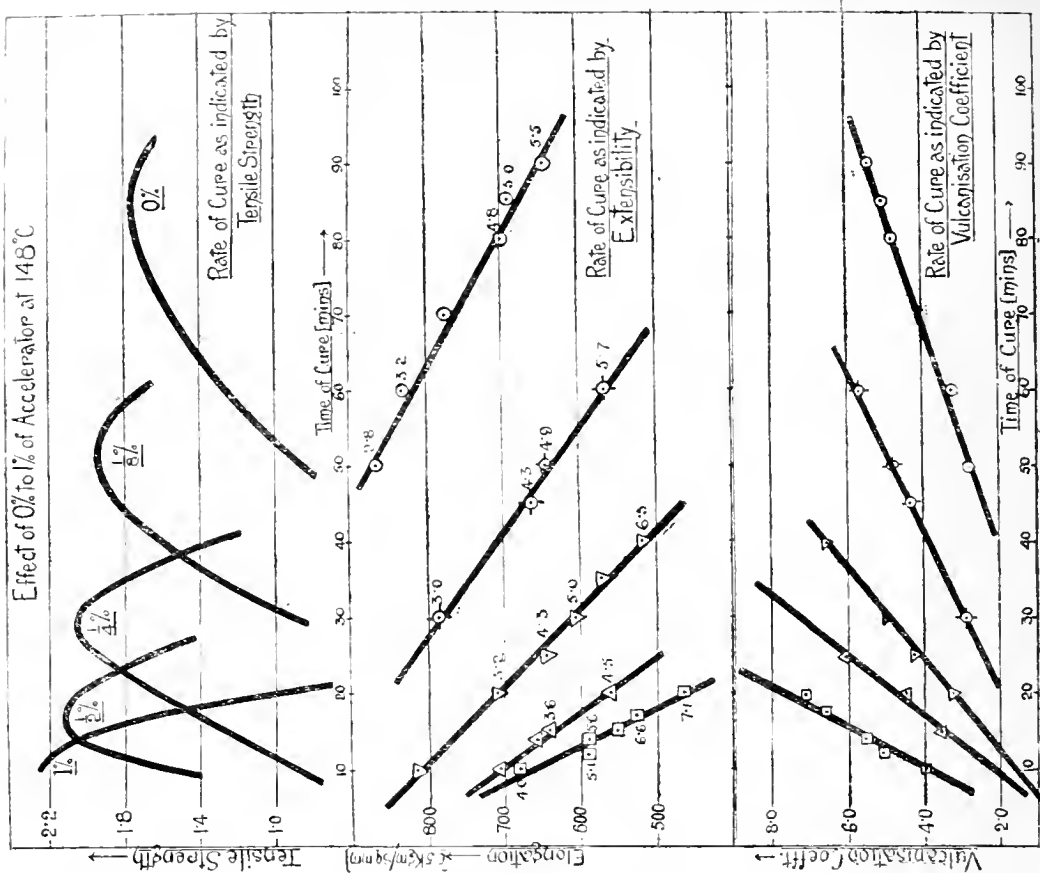


Fig. 1.

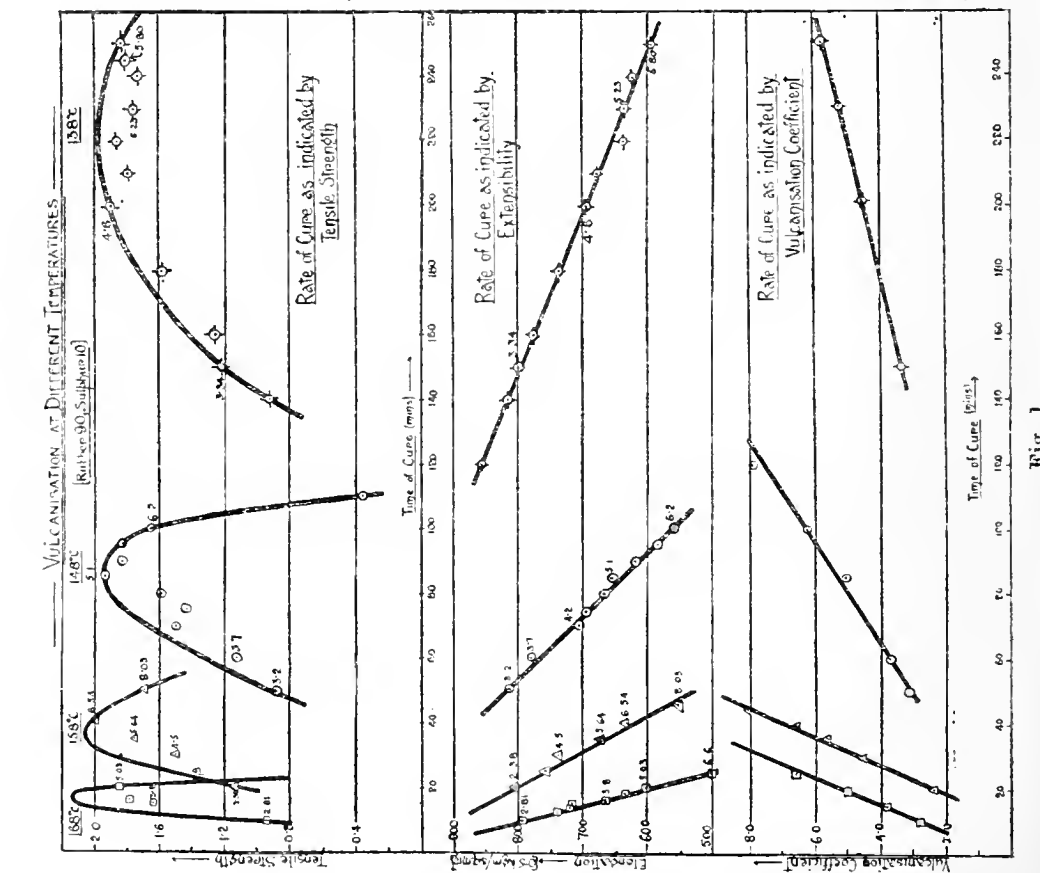


Fig. 2.

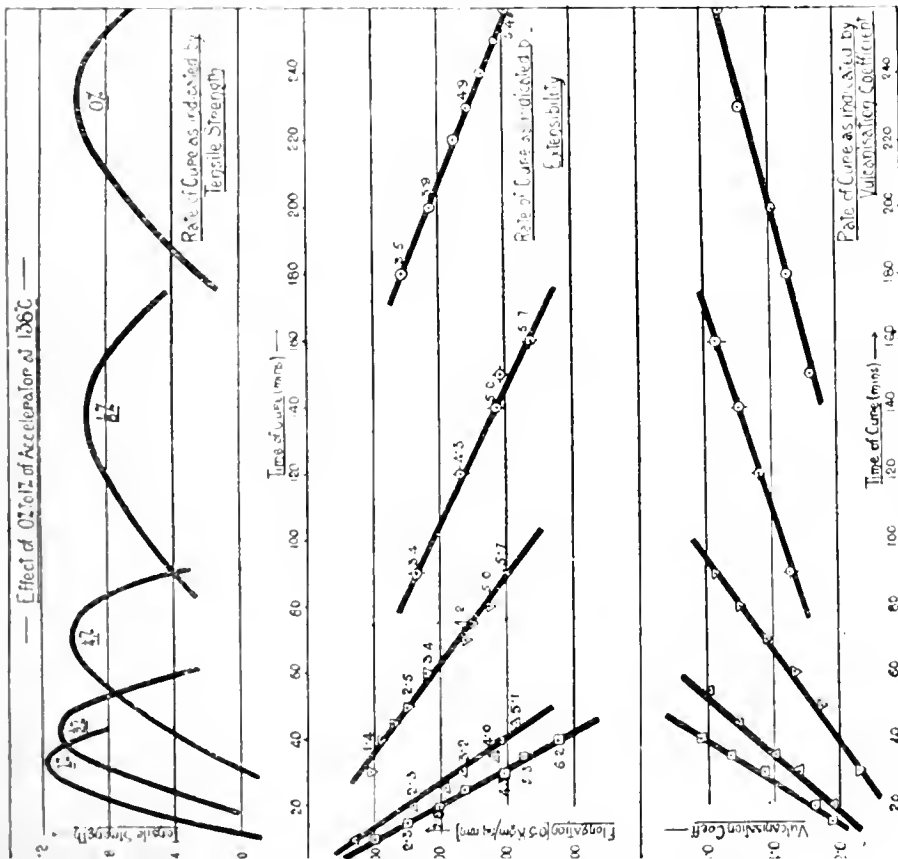


Fig. 3.

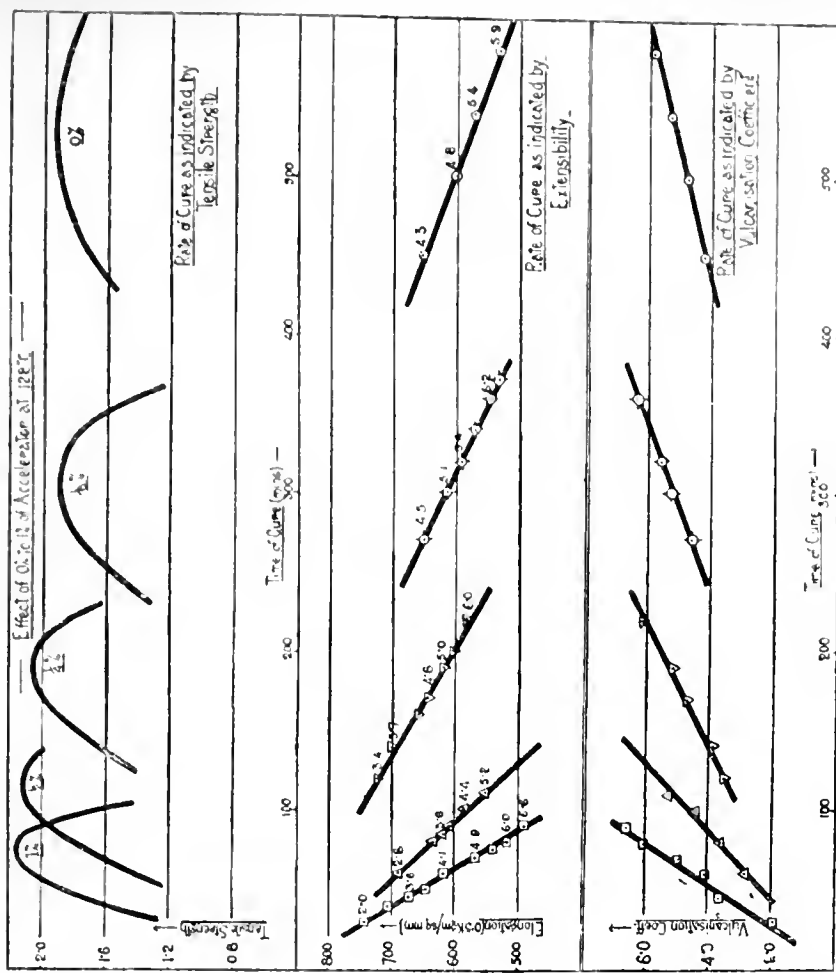


Fig. 4.

clot, freshly coagulated from the latex, is kept for several days before being rolled and washed, partial decomposition of the nitrogenous constituents of the retained serum sets in with the formation of organic bases. These are not eliminated by the subsequent rolling and washing, so that the resulting rubber exhibits exceptionally rapid vulcanisation.

As is illustrated clearly by our results in Fig. 1, the curve representing the rate of combination of rubber and sulphur does not follow the course expected from a simple chemical reaction, but, with less than 10% of free sulphur, is approximately rectilinear until the almost complete exhaustion of the sulphur (Spence and Young, this J., 1911, 817; 1912, 81, 785). This is probably to be explained by the occurrence of autocatalysis; with mixtures of rubber and sulphur containing more than 10% of the latter, the progress of the fixation of sulphur follows the sinuous S course, which is commonly regarded as characteristic of an autocatalytic process (Skellon, Rubber Industry, 1914, 172; van Iterson, Comm. Nederlands Govt. Inst. for Advising Rubber Trade, 1916, 7, 247). In the presence of an artificial catalyst, therefore, the compensation relation between the effect of the disappearance of sulphur and the extent of the increasing catalytic effect may be disturbed, so that the fixation of sulphur no longer follows a rectilinear course (van Iterson, *loc. cit.*).

Although the results as to the rate of vulcanisation of a simple sulphur mixing, as decided by these three methods, are comparable, in the presence of an extraneous catalyst, this is not necessarily so. The chemical action of sulphur on the rubber induces the physical alterations which constitute the advantage to be gained by vulcanisation, but the chemical and physical processes are not necessarily strictly proportionate, and some "accelerators" influence one more than the other. In the presence of certain accelerators the physical or mechanical alteration is disproportionately rapid, and the tensile strength attains its maximum* at a coefficient of vulcanisation (comb. sulphur $\times 100 \div$ rubber) well below the normal value of 5 (see p. 130 T).

Other accelerators, on the other hand, reduce the sharpness of the optimum, so that the peak of the curve is less pronounced. In yet other cases the catalyst may give rise to a vulcanised rubber with an abnormal extensibility relative to its coefficient of vulcanisation. Most of them, but not all, by reducing the time of heating necessary, cause the production of a rubber with a higher tensile strength than would be obtained by more tardy vulcanisation at the same temperature without the catalyst, and in this direction reduction of the time of vulcanisation by using an increased percentage of sulphur can have a similar effect (van Rossem, *loc. cit.*, p. 210).

The effectiveness of one of the above-named organic catalysts, *viz.*, aldehyde-ammonia, is demonstrated by the accompanying curves (Figs. 2-6) representing the results of experiments made, like those given in the earlier curves, with a mixture of pale crêpe and sulphur (90:10), at various temperatures from 148° C. (51 lb. steam pressure) downwards, in an oil bath vulcaniser. Even at a concentration of 1% the effect is still clearly observable while, with 1%, vulcanisation occurs so readily as to be possible in a reasonable period at 108° C., or less than 5 lb. steam pressure; Fig. 7,

indeed, records the progress of vulcanisation at 98° C. It will be seen that the temperature coefficient calculated by the ratio of the speed of reaction at intervals of 10° C. from 108° to 118° is practically the same as for the reaction in the absence of an artificial catalyst (p. 125 T), the average value for the accelerated mixings represented in Figs. 2-6 being 2.4. This observation militates against the belief of some investigators in this field that vulcanisation catalysts are not themselves able to expedite vulcanisation, but that during the early stages of the process they combine with sulphur, giving rise to substances which possess the desired activity. This view may be correct in certain cases, but evidently cannot be accepted generally for all vulcanisation catalysts. The average values of the coefficient with the various proportions of accelerator are given in the table:—

Average Temperature Coefficient.

Proportion of accelerator,	1% (108° - 148° C.)	3% (118° - 148° C.)	1% (118° - 148° C.)	1% (118° - 148° C.)	Nd (128° - 168° C.)
Method of testing—					
(a) Combination with sulphur ..	2.4	2.3	2.4	2.3	2.3
(b) Elongation at 0.5 kilo. per sq. mm. ..	2.5	2.5	2.5	2.4	2.3
(c) Maximum tensile strength ..	2.5	2.5	2.2	2.5	2.4

It is a striking fact that although the individual values of the temperature coefficient between 108° and 148° C. without exception oscillate closely about the mean value of 2.4, the interval 98°-108° C. shows a much greater value exceeding 5.0. This is doubtless due to the melting of the sulphur between these two latter temperatures, the normal melting point being lowered under the obtaining conditions. This observation supplies a confirmation of the argument on page 125 T as to the comparable effectiveness of the various allotropic forms.

The fact observed above, that the temperature coefficient possesses comparable values for mixings with and without an artificial catalyst, appears from other results in our possession to be a general one for all catalysts. This facilitates the representation of the relative effectiveness of different catalysts by means of a numerical factor. A factor representing the ratio of the respective periods of vulcanisation required for the attainment of a definite state of vulcanisation in a rubber-sulphur mixing, with and without a definite proportion of accelerator, will be independent of the temperature; it will be essential that this comparison be made at a stage of the vulcanisation process when the mixture still contains a considerable proportion of uncombined sulphur. The resulting "acceleration factor" may differ according to which of the three possible criteria—percentage of combined sulphur, maximum tensile strength and extensibility at definite load—is taken as fixing a definite state of vulcanisation. In the case of the accelerator used in the experiments now quoted, the behaviour may be described as normal, all three methods giving comparable results. The effectiveness calculated in this way for the accelerator at various concentrations and at different temperatures is given in the following table; the figures in each case represent the mean of the values obtained by the three different methods for the comparison of rate of vulcanisation.

Stated otherwise, the presence of 2, 4, 1, and 1% respectively of the accelerator increases the rate of vulcanisation at any ordinary vulcanising temperature to 1.7, 3, 5, and 7½ times the normal. A

* This does not refer to the so-called "technical optimum" of vulcanisation for which the corresponding coefficient of vulcanisation in the presence of a catalyst may fall as low as 2 or 1. (Kratz and Flower, J. Ind. Eng. Chem., 1919, 11, 39.) The technical optimum cure is probably capable of less definite measurement than the maximum tensile strength in the type of mixing used above.

"Acceleration factor."

Percentage	1	2	3	4
Temperature—				
118°–128° C.	1.7	2.8	5.1	7.5
128°–138° C.	1.7	3.0	5.4	7.6
138°–148° C.	1.7	3.0	4.8	7.1

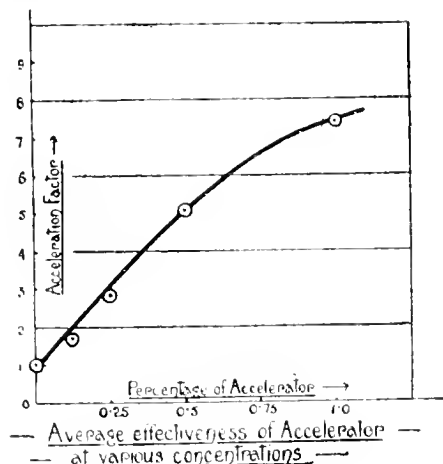
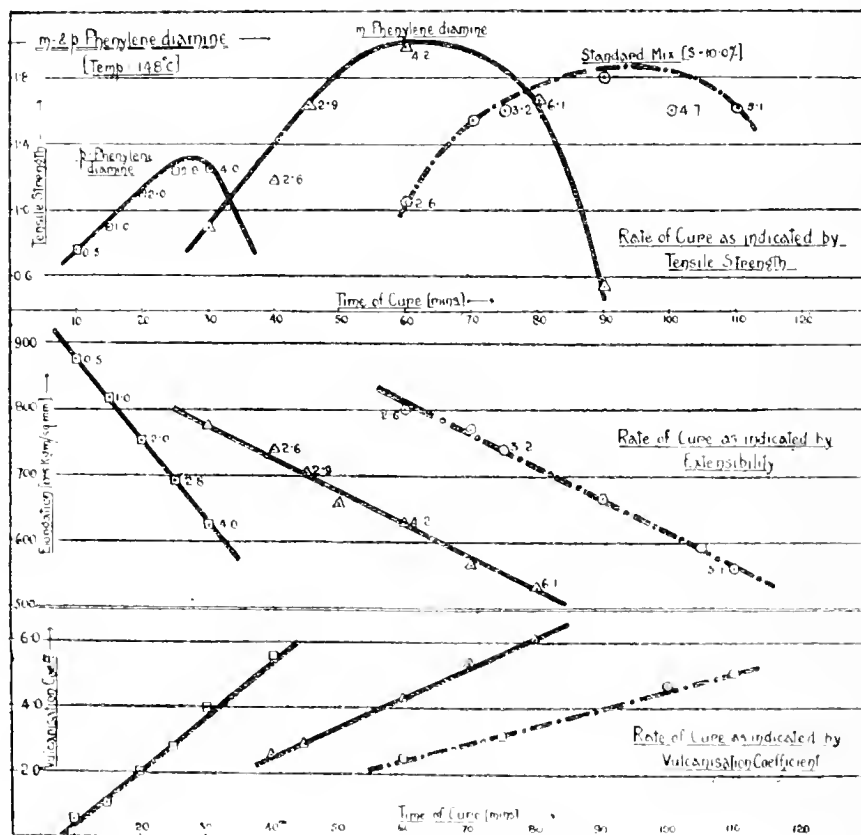


Fig. 8.

Fig. 8. The slight divergency (from the course of the smooth curve) of the points for the lower percentages is doubtless due to the relatively greater effect of the inevitable small loss of accelerator by vaporisation during the mixing operation.

Fig. 9 includes curves showing the relative effectiveness of isomeric substances, viz., *m*- and *p*-phenylenediamine as accelerators. The meta-compound is notably less active than its para-isomeride. It is of interest to note that the effectiveness of these two substances towards vulcanisation falls in the same order as their affinity constants as determined by Bredig in 1891. The low value of the coefficient of vulcanisation at the maximum tensile strength of the *p*-phenylenediamine mixing will also be noticed. The absence of points on the extensibility curve beyond a coefficient of 4 is due to the samples concerned being over-vulcanised and too weak for the test.

In Fig. 10 are given the results of tests on four different occasions and with three separate mixings, containing 1% of *m*-phenylenediamine; they are of importance, particularly in the extensibility section, as demonstrating the comparableness of figures obtained, even in independent experiments, with the standardised conditions adopted in this investigation.* The "acceleration factor" for *m*-phenylenediamine, calculated from the results in Fig. 8 has a mean value of approximately 1.5; for *p*-phenylenediamine judged by the physical methods the factor is approximately 3.3, whereas the rate of combination with sulphur indicates a value of only 3.0.



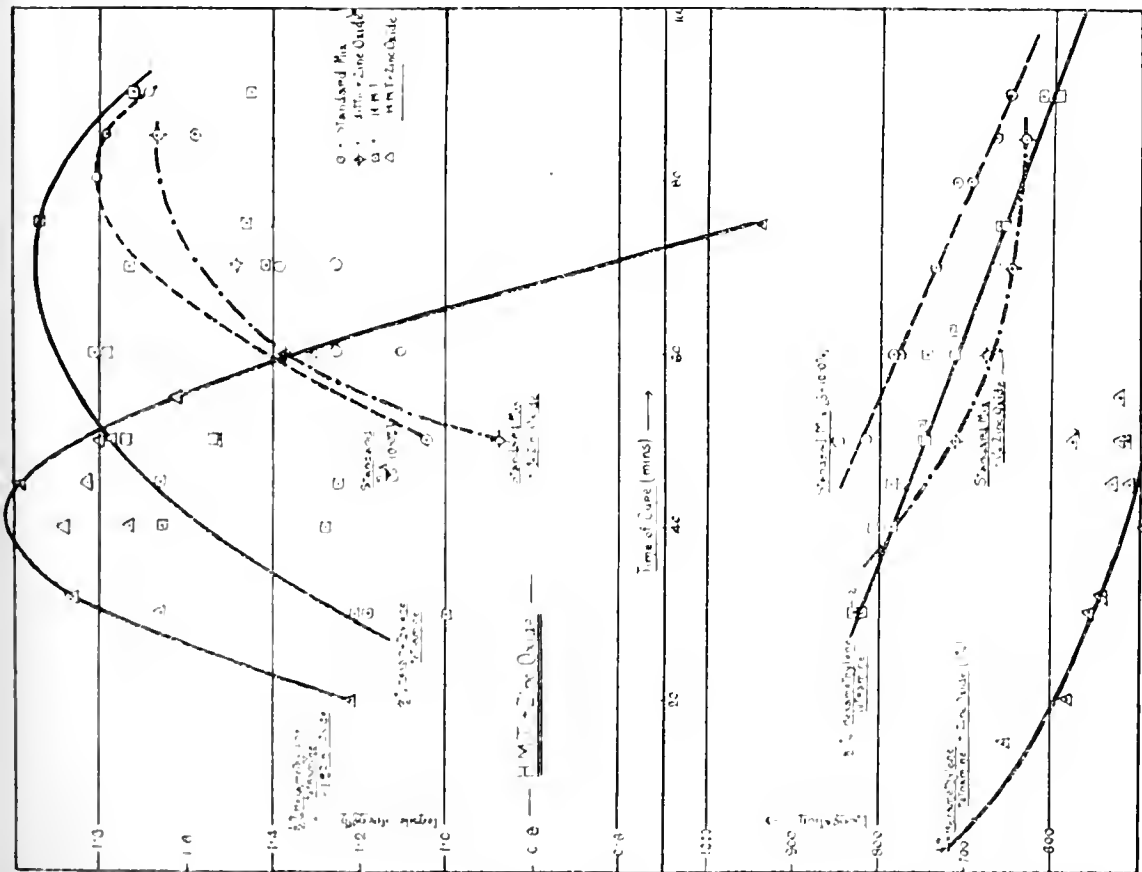


Fig. 11.

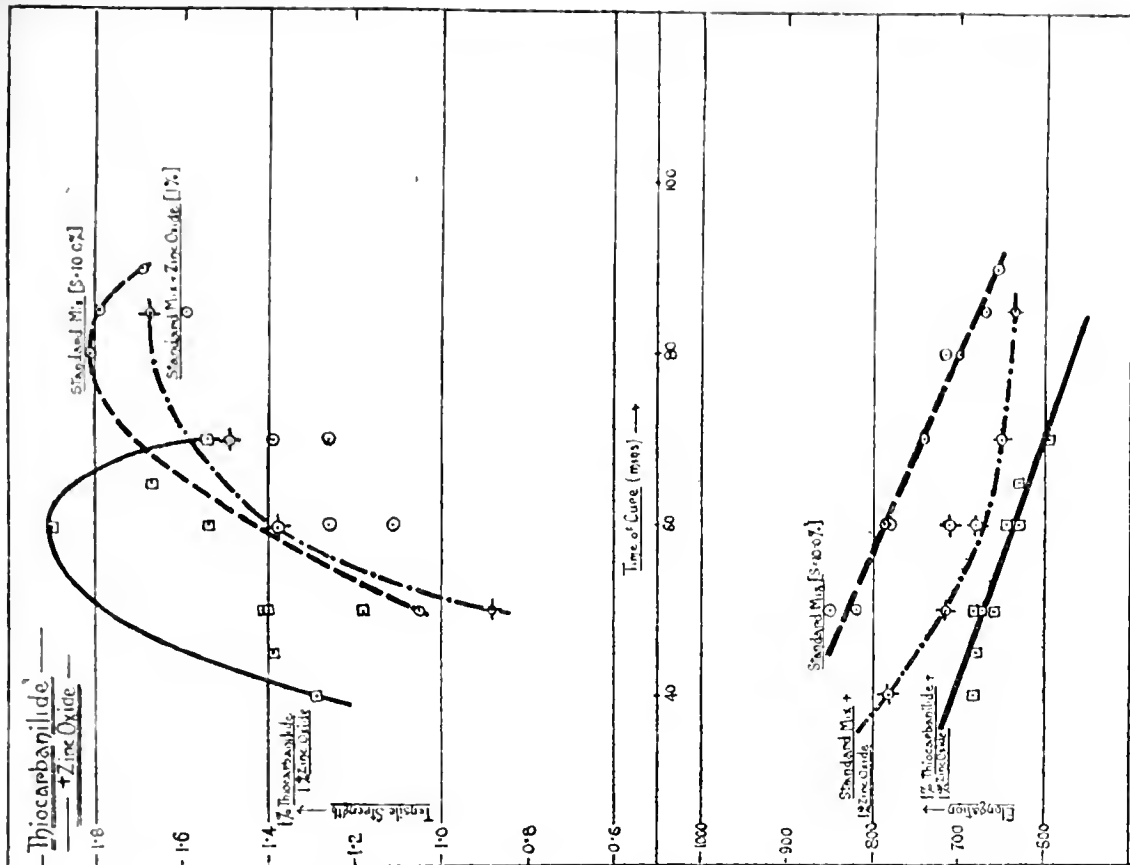


Fig. 12.

Mixed Catalysts.

The effectiveness of a mixture of catalysts in an ordinary chemical reaction is well known not to coincide invariably with the sum of the effects produced by each independently. This peculiarity is also observable with vulcanisation catalysts (Dittmar, *Gummi-Zeit.*, 1915, 29, 424). Lead oxide with magnesium oxide, and *p*-nitrosodimethylaniline with aniline or one of its homologues, are cases which have already been quoted in the literature.

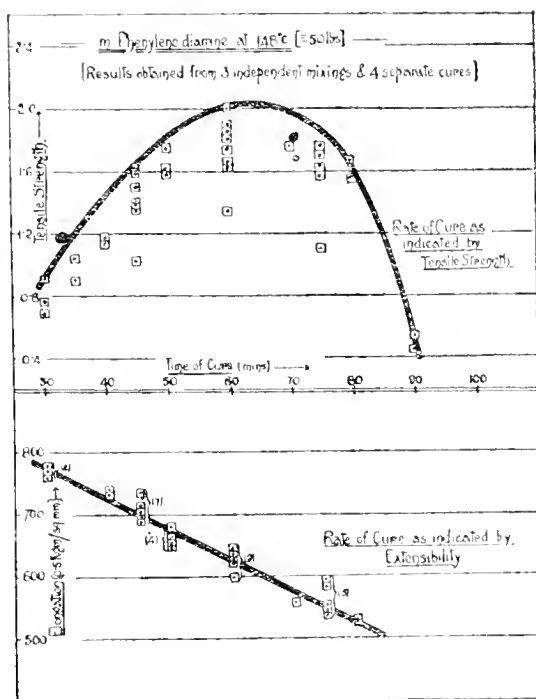


Fig. 10.

A related phenomenon probably is also the power of zinc oxide, which alone does not accelerate vulcanisation, to increase considerably the effectiveness of other organic catalysts such as hexamethylenetetramine and thiocarbonyl (see Figs. 11 and 12). As the latter by itself is practically inert, we have the interesting case of a mixture of two inactive substances exerting a distinct accelerating effect. In other cases we have found two vulcanisation catalysts to be "incompatible" in the sense that the effectiveness of the more active catalyst is actually decreased by the presence of the other.

As has been intimated already, no acceptable general explanation is yet possible as to the mode of action of vulcanisation catalysts. In addition to the theories already mentioned, others have also been proposed. An observation of considerable interest in this connection is that rubber in solution or wetted with benzene becomes vulcanised by successive treatment with sulphur dioxide and hydrogen sulphide at the ordinary temperature (Peachey, *this J.*, 1919, 688A). This reaction appears to be very suggestive in connection with the action of vulcanisation catalysts. None of the three modifications, S_{λ} , S_{μ} , and S_{π} present in liquid sulphur appears to be in possession of exceptional chemical activity towards rubber (see above), but there is evidently a possibility that there is capable of existence yet another form of sulphur of much greater vulcanising power. Such a view, needing considerable modification however, has already been tendered (Dubosc, *India-rubber World*, 1918, 59, 78; 1919, 59, 248), but the scope for investigation in this direction is enormous; indeed, the evidence available as yet is insufficient even to exclude the possibility that vulcanisation accelerators may activate the rubber and not the sulphur.

We are pleased to acknowledge the valuable assistance of Mr. C. W. H. Howson, B.Sc., in connection with the experimental work recorded above, and our indebtedness to the Dunlop Rubber Co., in particular to Mr. J. V. Worthington, Technical Superintendent and Director, for permission to publish this paper.

Bristol and S. Wales Section.

Meeting held at Cardiff on March 3, 1920:

MR. W. S. BIRD IN THE CHAIR.

RESEARCHES ON COAL.†

Part II.—An investigation of certain coking coals:
A theory of coking.

BY S. ROY ILLINGWORTH, B.Sc., F.I.C., A.R.C.S.

The various theories that have been advanced from time to time to explain the phenomena accompanying the production of coke from coal are reviewed by V. B. Lewes in Chapter 8 of "The Carbonisation of Coal" (1912). The author states: "Most observers look upon the luting body in the coal as tar residues." Wedding differs from this view and attributes the production of coke to the deposition of carbon within the mass of coal by virtue of the destruction of gaseous hydrocarbons. Donath and others consider that the production of a coke is due to internal change in the nature of the carbon molecule. Lewes advanced the opinion that the formation of coke arises from the successive deposition and decomposition of the heavy tars from layer to layer of the charge. The distillation of the heavy tars and their subsequent decomposition is due to the gradual flow of heat from the hot oven walls into the charge. Anderson (this J., 1898, 1027) ascribes the coking qualities of a coal to the presence in the coal substance of resinic bodies, and from the different behaviour of certain Scotch coals under the action of caustic soda, together with the different stabilities of these coals at 300° C., he divides the resins into two classes:—(a) One class decomposed below 300° C. and saponified or oxidised by caustic soda; (b) a class stable above 300° C. and not altered by caustic soda. Wheeler and Stopes in their "Monograph on the Chemistry of Coal," p. 9, make the following comment on Anderson's work:—"The compounds extractable by alkali solutions are undoubtedly ulmin compounds . . . moreover their presence in coals has been shown to exert a detrimental effect on the coking properties of the coal." Anderson's views must therefore be considered as not based upon conclusive evidence as to the nature and properties of the resinic substances in coal. Finally, Bedson, in 1900, showed that the cementing principle in certain coals was capable of extraction by pyridine, a view that was subsequently enlarged by Wheeler when he showed that the pyridine-soluble portion of a coal could be separated by extraction with chloroform or benzene into two portions, only one portion of which—the resinic or gamma compound—was possessed of coking qualities. Any endeavour to formulate a theory of coking must not solely take into account the mere formation of a coke, but in addition some light must be shed on the variations of strength, porosity, etc., of the cokes from different types of coals.

The differences in behaviour of the four coals dealt with in the first part of this paper (J., 1920, 1117) cast considerable light upon the fundamental causes influencing the nature of the cokes resulting from various coals.

The coal substance can be split into three types of substance by the "pyridine-chloroform" method of extraction. In conformity with the observations of Wheeler, Bedson, and others it was observed that

only the resinic constituents of the coals examined left a coherent residue when carbonised at temperatures varying from 450° to 900° C. Tests with mixtures of any two of the three components of the coals proved that only those mixtures containing resinic substances were capable of giving a residue of a coherent nature at 900° C. Consequently the property of causing the cohesion of a coal into a coke on carbonisation must be ascribed solely to the resinic portions.

The investigation of the decomposition of the coals at 450° C. proves that under the continued influence of this temperature a progressive destruction of the resinic portion of the coal substance results in the production of residues containing decreasing quantities of this type of substance. The residues obtained were ground to the same degree of fineness, and 1 grm. was heated at 900° C. under standard conditions in order to determine if a residue were formed which failed to produce a coke. The minimum amount of resinic substances necessary for the production of coke must lie between the amount present in that residue which produced by the shortest period of heating fails to yield a coke, and the residue anterior to it in series which does coke.

TABLE XXIII.

The coking properties and percentage volatile of residues from the various coals.

Residue.	Temp. of formation.	Volatile 950° on residue.	Resinic subs. in residue.	Nature of coke.
No. 2 Llantwit.				
1½	450	35.10	7.56	Dense hard coke.
1½	450	24.30	7.37	do.
3½	450	24.00	—	Very weak coke; scarcely coked; much detritus.
5½	450	24.10	5.33	do.
9½	450	23.20	5.63	do.
24½	450	21.00	4.00	No coke.
4½	350	32.00	—	Dense hard coke.
10½	350	30.85	0.30	do.
10½	400	—	—	No coke.
No. 3 Rhondda.				
1½	450	21.63	9.86	Dense hard coke.
3½	450	21.70	7.58	do.
5½	450	21.65	5.30	Dense coke.
24½	450	19.40	5.73	Coked; a little detritus.
No. 2 Rhondda.				
1½	450	20.51	7.99	Dense coke.
2½	450	14.56	5.01	Coked; much detritus.
5½	450	12.08	3.12	No coke.
12½	450	11.49	2.60	do.
Two foot nine.				
1½	450	19.18	8.62	Dense hard coke.
2½	450	16.70	6.36	do.
5½	450	16.10	4.51	Coked; much detritus.
24½	450	14.82	1.12	No coke.

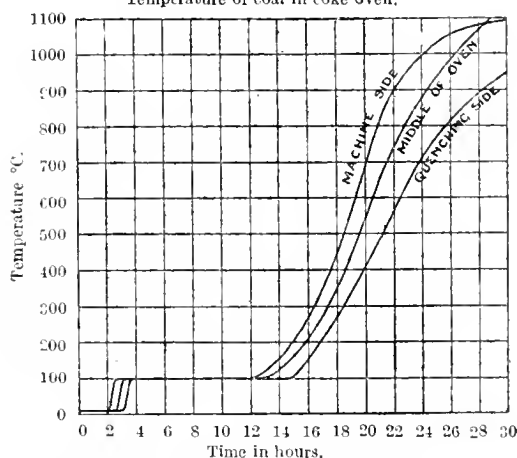
The above results indicate a difference in the cementing properties of the various resinic substances in the different coals; those in the No. 2 Llantwit have a weaker coke-producing quality than the others. The minimum amount of resinic substances in the residues from the various coals necessary to yield a coke lies between the following limits:—For the No. 2 Llantwit, 6–7%; No. 3 Rhondda, 5.30; No. 2 Rhondda, 5.0–5.5%; Two Foot Nine, 4.5–6.36%. The mean figure for the majority of the coals is about 5.5%, but probably for those coals containing less than 84% of carbon in the coal substance this figure will tend to increase. The investigation detailed in Part I. has led to the formulation of a new theory of coking, which will be first enunciated, and subsequently the facts upon which it is based will be indicated. It has been there shown that every coking coal is characterised by a fairly well-marked temperature

† Part I. of this investigation was published in the May 15th, 1920, issue of this Journal, p. 1117.

at which coking commences. The view is held, in agreement with Anderson, that all coking coals soften and become plastic, but this temperature is regarded as not far removed from 400° C. The β -cellulosic constituent of the coals examined by Wheeler (*J. Chem. Soc.*, 19—, —, —) were devoid of any tendency to melt or soften, but he found that the resinic substances melted at 150° C. Observations on the coals examined in the present research reveal the same characteristics of the two cellulosic types of substance, and confirm the fact that the resinic substances readily melt at 200° C., and become practically fluid at 350° C. in an inert atmosphere. The plasticity of the coal is therefore attributed to the liquation of the resins by heat, and the subsequent flow of the fluid or semi-fluid resinic matter around the other solid ingredients of the coal; possibly a definite absorption of the liquid resins by the other solid constituents takes place. The degree of plasticity in a coal is conditioned by the following factors: (a) the amount of resinic matter present; (b) the melting point and the point of complete fluidity of the resinic constituents; (c) the temperature to which the coal is subjected; and (d) the proximity of that temperature to the point of decomposition of the resinic constituents. The greater the amount of resinic substance present the more plastic the coal will become; too great an amount of resinic substance will result in a "molten" coal capable of flowing like a liquid. The initial cementation into a coke of the residues arising from the destruction of the constituents of a coal is due to the skeleton of carbon deposited between the non-melting ingredient by decomposition of the resinic substances.

The process of coking consists of the decomposition of the various substance in the coal with the evolution of volatile matter and the formation of a very highly carbonaceous residue. The decomposition must take place in the order of the thermal stability of the substances in the coal, for (in the modern type of coke oven) a very gradual temperature gradient exists between the hot walls of the containing vessel and the centre of the charge. The curves in Fig. 8 are constructed from the measurements of the temperature at various places in a coke oven, made by Simmersbach and detailed on page 335 of the first edition (1916) of Wagner's "Coal and Coke."

Fig. 8.
Temperature of coal in coke oven.



No doubt during certain intervals of time several substances are simultaneously undergoing decomposition, due to the temperature gradient in the oven increasing at a more rapid rate than permits of fractional decomposition of the coal substance.

The early stages in the carbonisation of a coal comprise, as the above results have shown, the destruction of the resinic and β -cellulosic substances, and consequently that phase of carbonisation taking place at temperatures below say 500° C. is the phase concerned with destruction of plasticity in the hot mass of coal. As a generalisation 5.5% is the minimum amount of resinic matter necessary (in a coal) for the formation of a coke, and since the passage through various degrees of plasticity is a characteristic feature of coking coals under the influence of heat, 5.5% of resinic substance may be taken as the minimum amount necessary to render a coal mass plastic. Throughout the period that a coal undergoing decomposition contains above 5.5% of resins the resulting plastic mass will tend to be forced into a spongy state by the internal pressure resulting from the generation within the mass of gaseous and other products volatile at the existing temperature. The progressive destruction of the resinic substances results in a change of the hot coal from the state of initial maximum "fluidity" through increasing degrees of "viscosity," the approach to a very viscous or semi-solid state, and finally the absence of any plastic nature due to the reduction of the resinic content of the mass below 5.5% of the residue. When the plastic mass is most fluid the volatile matter evolved will tend to pass through the mass and escape from it without forcing it into a spongy state, but as the whole becomes more viscous the volatile matter will tend to be retained within the mass as isolated bubbles, and will force the semi-coke into a spongy form. There must ultimately arise a state of affairs approximating to the stage at which the resinic substance in the residue has been reduced to some 5% or 6%, under which the volatile matter is generating within a medium so viscous that it takes a very appreciable period for that portion capable of escaping against the resistance of the mass to escape. The next phase is the loss of plasticity in the mass due to the further destruction of the resinic substances. It can readily be appreciated that the structure of the coke will be fixed since the final production of the carbon skeleton binding the mass into a rigid body has now taken place. Vesicles or pores will thus be formed in the coke, and their size will be conditioned by the pressure exerted by each occluded pocket of gaseous matter on the semi-solid mass just prior to it losing the final low degree of plasticity. The fundamental consideration determining the degree of porosity of a coke according to the theory here advanced is first the number of gaseous bubbles occluded in the coal mass when the resinic content is in the neighbourhood of 5.5%, and secondly the pressure exerted by the gaseous matter thus occluded. These two factors are both determined by the amount of volatile matter evolved from the coal substance in that period just prior to the decrease of the resinic matter to below 5.5%. The volatile matter evolved in the early periods is of lesser importance as regards its influence on the porosity of the coke, since so long as the mass is decidedly plastic it can "rise and fall" with any fluctuations in the evolution of gaseous substance; moreover, even if the volatile matter should force the coal into a spongy state, such a mass would sink down as soon as the evolution of volatile matter had decreased. It is not suggested that a coal becomes actually fluid during carbonisation. The terminology of plasticity etc. adopted above must be taken more as an analogy than as a dogmatic statement of physical state. The successive stages indicated are not visualised as ensuing throughout the whole mass of the charge at the same time, but as a cycle of events taking place from layer to layer of the charge in a direction parallel to the hot walls of the oven and conditioned by the rate of penetration of heat towards the centre of the charge. The fact that considerable pressure must exist within a coke

is evident from the following figures due to J. Parry and quoted by Lewes ("The Carbonisation of Coal," 1912, p. 221):—20 grms. of a coke of the following composition: C, 89.45; H, trace; O+N, 1.305; S, 0.795; ash, 8.45 %, gave rise to 361.5 c.c. of gas after heating for 2½ hours, and all told yielded 1117.2 c.c. after 14½ hours' heating. The gas evolved in the first 2½ hours contained CO, 22.8; CO, 13.4; H, 50.0; CH₄, 13.8 % by vol. The presence of methane in this gas is evidence of the occlusion of gases evolved at temperatures below 700° C., for Burgess and Wheeler (J. Chem. Soc., 1910, 97, 1917) state that paraffins cease to be produced above this temperature. The volatile matter evolved from a coal during any period of time at temperatures below 500° C. is the sum of the volatile matter arising from the resinic and β -cellulosic components undergoing decomposition. The resinic constituents are the source of the substance which cements the various residues from the coal substance into a coherent coke; associated with the formation of the "cement" is the evolution of a definite amount of volatile matter derived from the resinic substances undergoing degradation to carbon. This volatile matter *per se* must result in the production of porosity in the coke, the degree of which is determined by the amount of volatile matter evolved in the period just prior to the destruction of coking qualities in the layer of coal considered during the period of time when the resinic content of the semi-carbonised mass is between 5 and 6%.

Some indication of the difference in the amount of volatile matter evolved at various temperatures from the resins in certain of the coals examined is evident from the following table, the values in which were determined by heating 1 gram. of the resin at the specified temperature for seven minutes in a platinum crucible:—

TABLE XXIV.

Percentage of resins volatile at various temperatures.

Coal.	425° C.	600° C.	950° C.
No. 2 Llantwit	29.43	60.03	81.04
No. 3 Rhondda	8.9	56.00	69.45
No. 2 Rhondda	12.87	54.60	64.21

The marked difference in volatility of the resinic constituents of the No. 2 Llantwit (and the No. 2 Rhondda) is very evident; it constitutes a characteristic difference in property of the resins in the gas and coking coals. This greater volatility at the lower temperature in conjunction with the fact that of the coals examined the No. 2 Llantwit is the only one to decompose (to any great extent) at 350° offers some explanation of the greater porosity of the coke from this coal, but other factors have yet to be considered. The previous results have shown that the true coking coals contain an apparently homogeneous β -cellulosic constituent which is decidedly more unstable than the resinic constituents in these coals, and is practically destroyed in the first three hours of carbonisation at 450° C. On the other hand, the gas coals contain a β -cellulosic substance of the same order of stability as their resinic constituents, and it is associated to a considerable extent with the residual resinic remaining in the coal after long periods of carbonisation at 450° C. The true coking coals lose their β -cellulosic constituents as such in the very early periods of coking when the coal is still possessed of considerable plasticity, and the volatile matter arising from the decomposition of this type of substance is largely able to escape from the mass. The result is that during the critical period of carbonisation the viscous coal mass is only under the leavening influence of the volatile matter evolved from the resinic

substance, an amount much smaller than that evolved from the resinic substance of the gas coal, hence the texture in the case of the coking coal is more compact than that arising under similar conditions from the gas coals. Due to the greater stability of the β -cellulose in the gas coals and to its resultant association with the resinic matter throughout long periods of carbonisation at 450° C., the coal mass of the gas type of coal is, during the critical period, not only under a greater leavening influence arising from the greater amount of volatile matter evolved from the resins themselves, but added to this must be the effect of the volatile matter evolved from the β -cellulosic substance remaining in the semi-coke and undergoing decomposition concurrently with the resinic substances. The greater stability of the β -cellulosic substance and high yield of volatile matter from the resinic substance are among the chief causes of the porosity of the cokes from the gas type of coals.

Fig. 9.

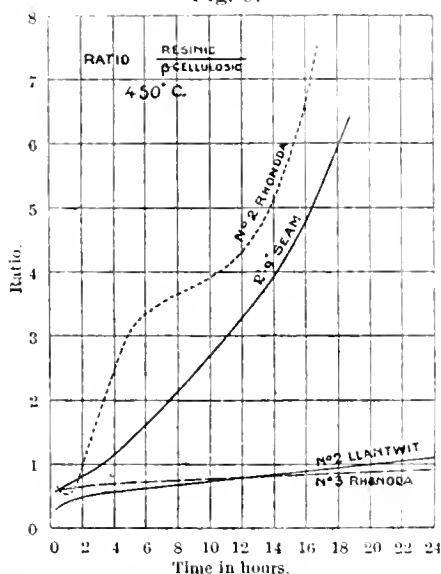


Fig. 9, showing the variation of the ratio of resinic to β -cellulosic constituents in the various residues derived from the coals investigated, illustrates the rapid rise towards the maximum of these curves for the true coking coals compared to the very slow rise of the curves for the gas coal—facts due to the greater relative stability of the β -cellulose in the latter type of coal.

TABLE XXV.

Percentage of β -cellulosic constituents volatile at various temperatures.

Coal.	425° C.	600° C.	950° C.
No. 2 Llantwit	14.01	20.83	37.45
No. 3 Rhondda	10.08	22.16	27.01
No. 2 Rhondda	9.42	14.81	28.69

The results in the above table were obtained in the same manner as the results given in Table XXIV. The greater amount of "volatile" evolved at the lower temperature in the case of No. 2 Llantwit is a feature that still further increases the tendency of this coal to produce a porous coke. The volatile matter evolved during any interval of time by decomposition of resinic and β -cellulosic material in the coal is actually the volatile matter evolved from that portion of the coal substance soluble in

pyridine and decomposed in the interval of time considered. This figure can be determined, since it is the loss of weight of the coal substance for any period divided by the decrease of pyridine-soluble constituents in that period. The loss of weight of the coal substance of the various coals in any given period is readily obtained from the data detailed in Table VII., whilst the corresponding amount of pyridine-soluble substance destroyed can be obtained from Tables VIII., XI., XIV. and XVII. The percentage ratio figures thus obtained are for brevity termed the "period volatile."

Fig. 10.

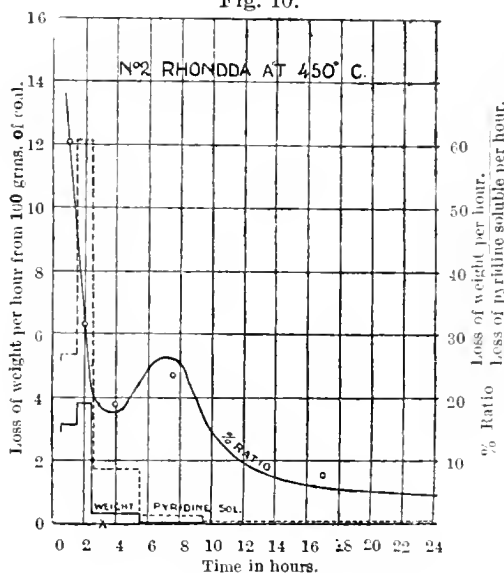
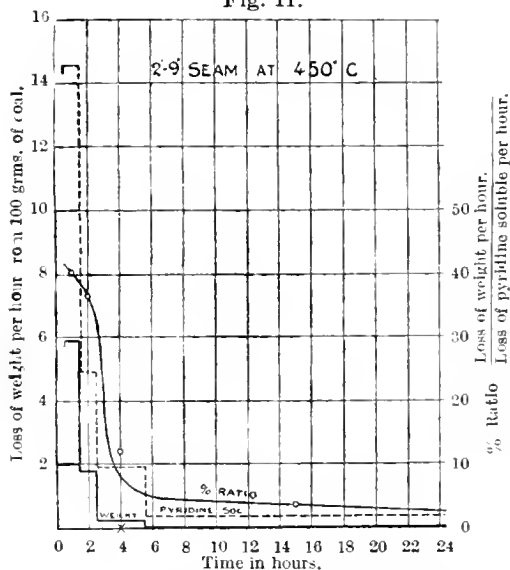


Fig. 11.



The loss of weight and the loss of pyridine-soluble substance per hour per 100 grms. of coal substance are represented for each coal in figs. 10-13. From these results the ratio curves have been constructed. It is readily apparent that the order of the coals as regards the amount of volatile matter yielded at any instant by the decomposition of the pyridine-soluble substance is as follows:—No. 3 Rhondda, No. 2 Llantwit, No. 2 Rhondda, Two Foot Nine

Seam. In the case of the coking coals the curves reach a maximum value in the early period of heating and fall rapidly towards a minimum. The crucial characteristic of any coal as regards porosity

Fig. 12.

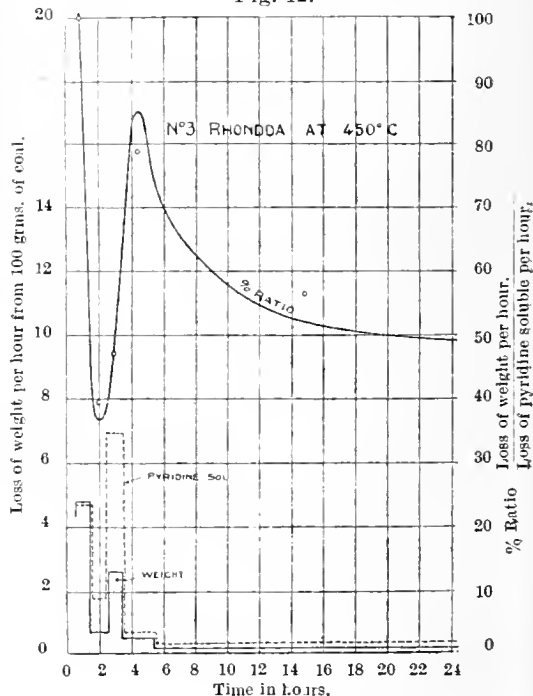
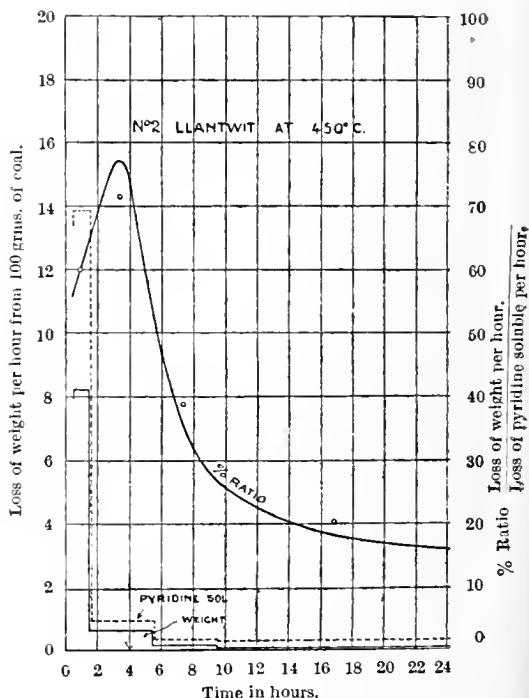


Fig. 13.



of the resulting coke is the amount of volatile matter evolved when the hot mass is in its most viscous state and about to pass to a fixed structure by virtue of destruction of the resinic. In the case

of No. 2 Llantwit this change occurs between 3½ and 5 hours' heating at 450° C.—in all probability at the end of 4 hours' heating. At this time the volatility of the pyridine-soluble substances undergoing decomposition approaches the maximum, and hence the viscous mass just prior to fixation of form is under the influence of the maximum leavening effect that arises from this coal. The destruction of coking qualities in the Two Foot Nine and No. 2 Rhondda occurs after 4½ and 2½ hours' heating at 450° C. respectively, at which time the percentage of the volatile matter evolved from the pyridine-soluble substance undergoing change is far removed from the maximum value and approximating towards the minimum value. Hence the leavening influence in this type of coal is comparatively small.

The leavening effect during the critical stages is the total volatile matter evolved during this period, the value of which from 100 parts of coal substance can be calculated by multiplying the amount of pyridine-soluble substance undergoing decomposition by the percentage volatile of the substances decomposed. The values for the various coals at their critical period are given in the following table:—

TABLE XXVI.

Leavening effect based on 100 parts of coal substance at critical periods.

Coal.	Loss wt. per hour.	% volatile from pyridine-soluble.	Total volatile per hour (porosity factor).
No. 2 Llantwit ..	1.0	73	0.73
No. 2 Rhondda ..	1.8	18	0.32
Two Foot Nine ..	1.9	8	0.16
No. 3 Rhondda ..	0.2	45	0.09

The figures in column 3 of the above table are termed the "porosity factor," and it is interesting to note that the numerical values of the factors for the individual coals are in the same order as the porosity of the cokes they give rise to.

No. 3 Rhondda is intermediate in nature between the gas and coking coals. On an industrial scale it is utilised for both purposes. In many of the features so far discussed for the several coals this particular coal has behaved more like the gas coals than the coking coals, but it will now be shown that No. 3 Rhondda has all the special characteristics of a true coking coal. The data given in Table XXIII. show that after 24 hours' heating at 450° C. the coking qualities of this coal are not destroyed. The curves in fig. 5 indicate the greater relative stability of the pyridine-soluble constituents of this coal. In addition the general curvature of the curves in fig. 3 leads to the conclusion that the β -cellulosic constituents will be wholly destroyed at the end of 48 hours' heating at 450° C.; at the expiration of this time there will remain in the residue some 5–6% of resinic matter, an amount sufficient to cause the residue to coke. The critical period in the production of a coke occurs after a long lapse of time, and it is evident from the extrapolated results derived from the curves in fig. 12 that during the critical phase the pyridine-soluble constituents will be undergoing decomposition at a rate not exceeding the destruction of 0.2 grm. per 100 grms. of coal substance, and that the volatile figure of the constituents destroyed will be 45% by weight.

The porosity factor will thus have a value 0.1, which is lower than that of any of the coals considered, and hence the coke from this coal will be of a dense nature. The very low rate of decomposition of the resins in the coal coupled with the resulting longer periods of time that the hot mass is in a plastic state allows the volatile matter

evolved to escape from the mass, and it is this long period of coke formation which nullifies the highly volatile nature of this coal and its propensity to result in a porous coke.

The theory of the causes of porosity in a coke is thus developed from experimental data. In photomicrographs taken by direct illumination of the surface of the residues of No. 2 Llantwit coal, the large vesicles and stream lines of the molten resins are evident in the one-hour and two-hour residues. The 5-hour and 24-hour residues are devoid of large vesicles, individual particles of the charge are not evident, and the whole mass has settled down to a compact coke.

Hardness of coke.

The hardness of a coke is determined by two factors: (a) The total amount of cementing material in the coke; (b) the distension of the cementing material, i.e., the volume over which it is dispersed, a factor conditioned by the porosity of the coke produced.

The initial cementing material in a coke results from the amount of fixed carbon left after decomposition of the resinic substance. The total hardening or cementing material is the initial material plus that amount of carbon subsequently deposited by the degradation of hydrocarbons etc. at higher temperatures—say above 600° C. Work is at present proceeding in order to ascertain the maximum amount of resinic substance from the coals discussed. Meanwhile the maximum amount so far obtained is taken as a basis for calculating the initial cementing materials in the coals. This figure is obtained by multiplying the percentage of resinic substances present by the amount of fixed carbon they yield when carbonised; the results are detailed in Table XXVII.

TABLE XXVII.

Coal.	Resin-ic matter	Fixed carbon.			Carbon per 100 coal substance.		
		425° C.	600° C.	950° C.	425° C.	600° C.	950° C.
No. 2 Llantwit	9.65	70.57	38.97	18.96	6.81	3.76	1.83
No. 3 Rhondda	14.07	91.10	44.00	30.55	12.81	6.19	4.30
No. 2 Rhondda	10.18	87.13	49.54	35.79	8.89	5.04	3.64

It is evident that the gas coals give rise to a smaller amount of initial cementing material at any temperature and must hence produce a coke of weaker mechanical properties than the other coals. The initial cementing material, in my opinion, is the main cause of the hardness of a coke, for the amount of oil left behind and non-volatile at 450° C. in the various residues arising at this temperature never exceeded 1% by weight of the coal. The figures given above indicate the reason for the brittleness of low-temperature cokes due to incomplete carbonisation of the resin. The thermal stability of the resinic substances influences the hardness, since a very gradual decomposition of these substances may result in the small amount of fixed carbon produced at one instant being disrupted by a rise in a semi-coked mass at a subsequent period. On the other hand, a rapid decomposition of resinic substance means a rapid fixation of structure and will per interval of time produce more fixed carbon, a stronger structure and one least liable to fracture by internal pressures arising during any subsequent interval.

The brittleness of certain batches of coke from the No. 3 Rhondda is to be ascribed to the periodical rising and falling of the semi-coked mass resulting in the alternate formation and partial fracture of structure, a sequence of events very possible since this coal contains a remarkably stable resinic substance and the "period volatile" of the pyridine-

soluble constituents reaches a very high figure by comparison with the other coking coals. It is interesting to compare the views here developed with the phenomena arising in actual practice. This aspect of the question will be developed at length in another communication; it will suffice for the moment to recall that the gases resulting in the very early stages of carbonisation are weak gases poor in hydrocarbons and comparable with the gas obtained by Wheeler by distillation of β -cellulose.

The endeavour in actual practice to shorten the period of coking in ovens by increasing the temperature of the oven walls often results in the production of a porous coke from a coal which under normal conditions yields a good furnace coke. The higher temperature of the walls results in a steeper time-temperature gradient in the oven and a considerable shortening of the time during which the charge is under the influence of temperatures below 500° C. The relative stability of the β -cellulosic and resinic substances will be the less differentiated the higher the temperature to which these substances are subjected; moreover, the critical period of the charge will be shortened by the higher temperatures. The result with higher flue temperatures will be that the fixation of structure in the coke will ensue nearer to the period of the maximum "period volatile" of the pyridine-soluble substances, a fact which will tend to produce a more porous coke.

Finally, it may be mentioned that, arising out of this series of researches, new processes have arisen that give every promise of bringing about the coking of coals at present unsuitable for production of metallurgical coke, improving the qualities of certain types of coal which yield inferior cokes under modern procedure, and thus materially enlarging our present gradually decreasing supply of coking coals—a class of coal so essential to the iron and steel industry. The processes have developed to the stage of large-scale trials, and they have been provisionally protected. I hope shortly to deal with this aspect of the question.

This investigation has been carried out in the Chemical Department of the South Wales and Monmouthshire School of Mines, Treforest. I desire to tender my best thanks to the members of the Board of the School for the facilities so kindly provided, and to Principal Knox and Mr. Robert Metcalfe for their interest and assistance throughout the work.

This work is being extended both in the direction of submitting the coals here considered to the influence of higher temperatures and the examination of a greater number of coals along the lines indicated.

Chemical Engineering Group.

Conference held at Newcastle-on-Tyne on December 15, 1919.

(Abstracts of Papers.)

HANDLING OF PETROLEUM IN BULK AT THAMES HAVEN.

BY N. A. ANFILOGOFF, F.I.C., M.I.MECH.E.

The Petroleum Storage Wharf at Thames Haven has a tank with a capacity of over 500,000 tons. The tank steamer is taken alongside any one of the four deep-water piers, and there empties its cargo by means of its own pumps through various pipe lines into the storage tanks on the wharf.

For high-flash petroleum the ship's pumps are

supplied with steam generated on board ship, while for discharging low-flash petrol or crude oils steam is supplied from shore. The steam is supplied by high-pressure boilers situated 800–1000 ft. from the ship and provided with superheaters, calculated to give sufficient superheat to insure the steam arriving at the tanker's pump cylinder at a temperature not less than that of saturated steam of that pressure. In practice it has been found that steam with 160 lb. gage pressure and a superheat of 140° F. travelled through a 3-in. main a distance of 850 ft., and at the end there was registered a temperature of 378° F., i.e., still showing some 20° F. superheat above the temperature of saturated steam. The loss of superheat worked out at about 1° F. for every 7 ft. of main.

When the cargo is discharged into the storage tanks of the ocean dépôt or installation the various products, in addition to those obtained from refining operations, have to be transported either direct to consumers or to inland or smaller dépôts for distribution. This is carried out by means of the ubiquitous square petrol can, by wood or galvanised steel barrels and drums, by either horse-drawn or self-propelled road tank car, by rail tank car, by towed bulk tank barges, or by small coasting tank steamers. At Thames Haven during 1918 the average daily handling of petroleum in bulk amounted to more than 2500 tons (750,000 galls.).

The works being spread over an area of nearly 200 acres, decentralisation had to be adopted rather than centralised power plant, and the 26 large B. & W. and Lancashire boilers are divided amongst eight boiler houses. A pumping installation is in close proximity to each boiler-house, the pumps employed being direct driven steam pumps of either the single cylinder or the duplex type. No type of pump will give equally good service with all grades of petroleum products under all and any conditions.

The duplex pump, used for limpid or non-viscous petroleum products, is of the usual "Worthington" pattern, where two steam cylinders side by side have pistons connected directly to two pump plungers. A D-valve is employed and the engine works without expansion. In the tandem, compound type of duplex high-duty pumps the steam is used expansively through high-pressure and low-pressure cylinders, and again each engine works its neighbour's valves. Some makers have fixed a Meyer type cut-off valve gear, which enables the engine to work without expansion for a certain predetermined part of its strokes and expansively for the remainder.

The general rules and details to be observed are that: (1) The pump rods, cylinder liners, piston rings (packing rings), valves guards, and springs should be of brass or gunmetal. (2) Valves guards, springs, and valve seats should be accessible. (A solid valve seat plate is preferable to separately screwed-in or pinned-in valve seats.) (3) Valve guards should be of ample strength to withstand the constant jar and should be pinned in addition to being screwed into valve seat. (4) Snuff or pet cocks should be provided both above and below delivery valve plate; and (5) the valve lift should be ample; if multiple valves are used the sum of the valve areas should be at least 25% greater than the area of the suction pipe. These pumps are recommended for such liquids as petrol, kerosene, and gas oils. Of two pumps capable of delivering the same quantity, the pump with the longest stroke (and fewer in number per minute) is to be preferred.

As in pumping petrol, for instance, the pump gets "gassed," a delivery air vessel with relief cock should be provided with every pump. Where the oil is very "gassy," or where long suction pipes are employed, a suction air vessel (similar to a delivery air vessel) should be provided. In pump-

ing petrol, especially, it is desirable that the pipes and pumps should be of ample dimensions to convey the quantity required without undue pressure. The suction pipes, if long, should be preferably larger than the inlet to the pump to avoid friction. The best packing for glands is "metallic" packing.

The provision of filter gauzes on the suction side of a pump is an advantage, but they must be designed in duplicate boxes, where the change can be easily effected, and must provide at least 25% more unrestricted area than the suction pipe itself.

For pumping the heavier and more viscous oils the single-cylinder pump is used. These direct-acting steam pumps consist of a steam piston at one end of the piston-rod and a plunger at the other. The pumps occupy little space and have very few working parts. They have no external valve gear, and the steam cylinder is so constructed that all the steam is utilised for effective work on the piston. The side valve operation is arranged so that there are no minute steam passages and consists of a very simple valve mechanism which is entirely enclosed. They have the longest stroke obtainable with the least number of strokes per minute, and automatically slow down at the end of each stroke; the valve gear and steam ports are constructed so that the piston does not strike the cover when running at excessive speed.

In pumping viscous oils a simple calculation from piston speed and size of bore and stroke will not necessarily give the number of gallons the pump is capable of delivering, because so much depends upon the viscosity of the oil, the temperature at which it is being pumped, and also upon the friction in the pipes. At ordinary temperatures 1° F. will change the friction somewhere about 5%. At Thames Haven, working with viscous fuel oils, the velocity through an 8-in. pipe frequently exceeds 6 ft. per second. The difficulties of pumping viscous liquids are enhanced by the fact that even when the oil is heated before it is pumped, when long lengths of pipe lines are used for delivering, there are a series of velocities inside the pipe, the highest being in the centre and the lowest where the oil comes in contact with the pipe itself, so that the internal liquid friction is considerable.

It is practically impossible to deduce formulae to provide definite data from which to select the size of pump required.

In practice, for the more viscous oils, especially those of which the viscosities cannot be measured in Redwood's No. 2 viscometer at 32° F., or such oils which have a setting point of, say, 50° F. or less, the storage tank is provided with steam coils and is kept at such a temperature as will reduce the viscosity sufficient to enable the oil to be pumped (usually between 80° and 100° F.).

The pipe-lines are in some cases enclosed in wooden boxes, with the steam pipe running alongside, but it would seem to be preferable to have a steam pipe running inside the oil pipe with the steam inlet at the end furthest away from the pump, so that the steam enters at presumably the coolest part of the pipe, and travels in an opposite direction to the oil. To prevent leakage of steam into the oil, glands are provided to permit the steam pipe to expand freely without becoming distorted in the pipe line, and taper-thread sockets are used for the steam pipe.

In pumping viscous oils it is essential that the suction and delivery pipes be of ample proportions, that a suction air vessel be used, that where filter gauzes are employed the area of such gauzes be at least 100% greater than the area of the suction pipe, and that the pump preferably have the least possible number of valves. These valves should each be of the largest possible size, and the valve seats should be so constructed that the webs do not unnecessarily break up the stream.

GENERAL TYPES OF PUMPING MACHINERY FOR CHEMICAL WORKS.

BY J. H. WEST.

The handling of liquids in chemical works involves a large variety of problems, ranging from the lifting of water from deep wells, fire service and boiler feeding, to the handling of highly corrosive liquids and of very thick and viscous liquids. A great variety of apparatus is available for dealing with these problems.

In selecting a pump for a particular purpose, reliability should be the first consideration, even beyond first cost or efficiency. It is mistaken economy to work a pump at maximum rating, since this involves very great wear and tear. It is usually recommended to work pumps for boiler feeding at half the piston speed given for general service duty, and it is more economical to use only 70 or 80% of the maximum piston speed for the latter. A long-stroke pump is preferable to a short-stroke one, since the wear on most parts is dependent on the number of reversals.

The direct-acting steam pump is cheaper than the flywheel steam pump. In the duplex form of pump a very simple steam valve gear can be used, since the motion of one rod actuates the valve on the other cylinder. These pumps are not economical in steam, owing to the necessity for considerable clearance at the ends of the stroke to allow of cushioning, and the duplex pump, unless well designed, is apt not to work its full stroke on both cylinders.

A ram pump has the advantage over one of the bucket type in that any slip past the plunger is apparent at the stuffing boxes, whereas considerable slip past a bucket plunger may take place without being detected.

The belt-driven centrifugal pump is probably the lowest in first cost of all types of pump, and the efficiency is high for large quantities but only moderate for medium quantities, especially at high lifts, and distinctly low for small quantities. The necessity of priming centrifugal pumps before starting, unless the liquid is supplied from a higher level than the pump, is a disadvantage for chemical work in most cases, but the absence of valves, and the fact that the decrease in efficiency owing to wear is probably less than in the case of reciprocating pumps, especially with the open type impeller, is in their favour. Trouble at the stuffing boxes is often due to faulty packing. A centrifugal pump with only a single stuffing box, and that on the suction side, has many advantages for chemical work.

In the case of pumps for corrosive liquids the question of safety and reliability far outweighs that of first cost or efficiency, since the quantities to be handled are generally comparatively small.

The choice of suitable valves has much to do with the satisfactory working of pumps lifting chemical liquors, and the provision of efficient strainers of ample area on the suction pipes is of great assistance to the proper operation of the valves.

PUMPING EFFICIENCY.

The following table gives the yearly cost of power for pumping water at various rates against a head of thirty feet, assuming 4000 hrs. to be worked in the year, and the cost of one h.p. hr. to be 14d., which is equivalent to electric power delivered to a motor of 85% efficiency at 1.42d. per unit.

In order to have a basis of comparison between the efficiency of steam pumps and those driven by belt or motor, it is necessary to assume a figure for the steam which represents a horse-power-hour, and in what follows this figure is taken at 35 lb.

The efficiency of small pumps of all types is very low, owing to the small hydraulic mean radius of

the passages, and this is particularly marked with centrifugal pumps.

Gallons per hour.	Yearly cost of power in pounds at various efficiencies.							
	100%	70%	60%	50%	40%	30%	20%	10%
500	1.6	—	—	—	4.0	5.3	7.9	15.8
1,000	3.2	—	—	6.4	8.0	10.7	16.0	32.0
5,000	16.0	—	26.7	32.0	40	53.0	—	—
20,000	64	91.4	106.7	128	160	213	—	—
50,000	160	229	267	320	400	533	—	—

A 1-in. centrifugal pump throwing 1000 gallons per hour against a head of 30 ft. will have an efficiency of 15% to 18%, a 2-in. pump throwing 5000 gallons per hour will show about 40%, a 3-in. pump throwing 10,000 to 12,000 gallons 55% to 60%, while efficiencies of 70% and over are only attainable in pumps of 6-in. or 7-in. and over, handling 50,000 gallons per hour upwards.

For any given centrifugal pump there is a certain set of conditions as to speed, quantity, and head, for which the efficiency will be a maximum, and any deviation from these conditions will mean a decrease in efficiency. It is desirable, therefore, to some extent, to design every centrifugal pump for the particular conditions under which it is to work, in order to get the best results.

The efficiency of duplex steam pumps will vary from about 20% in small sizes up to 40% in large sizes, corresponding to steam consumptions of 175 lb. per h.p.hr. to 88lb. That of direct-acting pumps will range from 25% up to 50%.

The efficiency of flywheel pumps is usually several per cent. better than that of direct-acting pumps of the same capacity, owing to the smaller clearances in the steam cylinder and the greater ease in working expansively. The steam consumption can be reduced considerably by compounding the steam cylinders, and also by fitting condensers to the exhaust.

Where the exhaust steam from pumps can be used for heating liquors in chemical works, the steam consumption is of little consequence, since nearly 90% of the heat in the live steam is recovered in the exhaust steam.

The efficiency of diaphragm pumps ranges from about 14% in small sizes up to 20% in the largest sizes usually made.

TYPES OF PUMPS.

Pumps may be classified as follows:—

- (1) General service pumps, *viz.*, those for water and non-corrosive chemical liquors. This type includes reciprocating pumps and centrifugal pumps.
- (2) Pumps for gritty and thick viscous liquids. These may be of the reciprocating or rotary type.
- (3) Pumps for corrosive acids, of reciprocating or rotary types.
- (4) Pumps operated by compressed air. These include the well-known acid eggs, either plain or automatic, and the air-lifts.

In using the egg for liquids containing solids in suspension, it is a good plan to introduce the air through the perforated pipe at the bottom of the vessel, instead of at the top, to keep the liquid agitated and prevent an accumulation of solids.

The efficiency of eggs and automatic elevators is a subject upon which little or no information based on actual tests is available, and it will be of interest, therefore, to consider the overall efficiency of the system.

Take the case of an automatic elevator in which the compressed air is admitted by a float or other mechanism as soon as the liquid reaches a certain level in the egg. As soon as air is admitted, pressure begins to build up and the liquid begins to rise in the delivery pipe, going on until it begins to flow out from the discharge top of the pipe. The rate at which the air pressure builds up will depend upon the size of the air inlet, but the air space above the liquid being very small, it is safe to assume, unless a reducing valve or regulator is used, that in most cases it will reach the full pressure in the air main almost instantaneously and the column of liquid in the discharge pipe will have been accelerated up to the maximum velocity corresponding to the air pressure used by the time the liquid reaches the top of the discharge pipe. In any event equilibrium will very quickly be attained at a velocity such that the "static" head plus the friction head equals the air pressure.

For instance, if water is being blown through a straight vertical 3-in. cast iron pipe 60 ft. long, the static head is 26 lb. per sq. in. and if the air pressure were only 26 lb. per sq. in. it would just raise the water to the top of the discharge pipe without causing any delivery, while an additional air pressure will cause a flow at the following velocities:—

Air pressure in excess of 26 lb. per sq. in.	Velocity of flow ft. per sec.
0.3	2
1.1	4
4.3	8
15.6	16

Thus if the air supply is at a pressure of 40 lb. per sq. in. the water will flow out with a velocity of about 15 ft. per sec.

This condition of equilibrium will be maintained, provided the air supply pressure remains constant until the egg is empty, with the exception of a slight decrease in the static head owing to the lowering of the liquid level in the egg, the depth of liquid in the egg of course balancing a corresponding column in the discharge pipe.

In automatic elevators where the air supply valve is gradually closed by a float which falls with the liquid, a certain amount of wire-drawing takes place, and the final air pressure may not be greater than that required to discharge the liquid with a moderate velocity; in fact, in some elevators, such as the Kestner, a regulator is provided to ensure this, but in any event the minimum amount of air used will be equal to the volume of the egg at the supply pressure.

In the case of hand-operated eggs, under the best conditions where the workman stands with his hand on the air-control valve and his ear against the discharge pipe, he is able to shut off the air immediately he hears the last of the liquid leave the egg, and he probably uses no more air than an automatic elevator, but if the air is not shut off at the proper moment, the waste of air may be very considerable, as will also be the case if the egg is not completely filled with liquid.

In comparing hand-operated eggs with automatic elevators, it must be remembered that the former are usually much larger than the latter, being often of 500 up to several thousand gallons capacity, as against 3 to 20, or sometimes 100 gallons in the latter, and the advantage in air economy is all in favour of one big blow instead of numerous small ones.

There is no reason why automatic elevator mechanism should not be applied to large eggs, but actually it is not often done. On the other hand, where a more continuous supply is required,

as in the case of acid supply to towers, the use of small eggs operating frequently is necessary.

The discharge pipes from eggs and elevators should be as nearly vertical as possible, otherwise at the end of the blow the air may break through the column of liquid and escape along the upper side of the pipe. The clearance or air-space at the top of an egg or elevator must be reduced as far as possible, since it represents so much wasted air.

The efficiency of blow eggs decreases with increasing pressure, owing to the greater amount of energy contained in the air left in the egg at higher pressures.

The homogeneous lead lining of steel eggs affords a convenient protection against weak sulphuric acid etc. The use of ebomite lining for eggs has been adopted for hydrochloric acid.

The air-lift.—The air-lift provides an exceedingly simple method of raising liquids, and one that is fairly efficient if properly designed. It is not used in chemical works to anything like the extent it deserves. Compressed air is injected at the bottom of a discharge pipe so that alternate piston-like layers of air and water are formed in the pipe, thus lightening the column in the pipe and allowing the liquid to be blown out with an air pressure which may be as low as $\frac{1}{2}$ to $\frac{1}{4}$ lb. per foot of actual lift with water.

The use of air-lifts with hot liquids offers special advantages, since the heat energy of the liquid contributes to the work of pumping by expanding the air, and pumping efficiencies of 100% and over can be obtained in this way. Air-lifts are also used for lifting molten metals, and in such cases the thermal expansion of the air will be still more marked owing to the higher temperatures.

The correct proportioning of the "submergence," or ratio of the depth below the free level of the liquid in the well or feed tank at which the air is applied, to the total lift, has much to do with the efficiency of an air-lift, and while no hard and fast rules for determining this factor can be given, since it depends to some extent upon the size of the eduction pipe and the nature of the liquid, it is generally admitted that the lower the lift the greater should be the submergence, and that the greater the submergence the higher the efficiency. For lifts up to 100 ft. the submergence will usually lie between 50% and 70% of the lift, decreasing to about 35% with a lift of 500 ft. Efficiencies of over 30% can generally be obtained with a well-designed air-lift, and 40%, or even higher, can be reached in some cases with cold liquids.

In a method of applying compressed air in pumping known as "the return air system," reciprocating pumps of the steam driven type are used with compressed air instead of steam, and on the closed circuit system, i.e., the exhaust from the pumps returns to the compressor for re-use. In this method the economy in compression at the higher pressures is utilised, and losses of air through leakage are made up by the small booster compressor.

Steam ejectors are often useful for draining pits and sumps. Their low efficiency is against their use for other purposes.

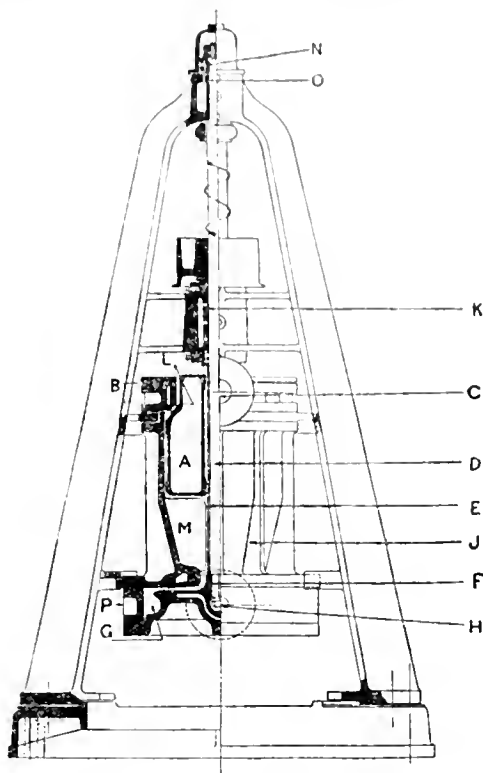
The author described a large number of different types of pumps with illustrations, and also gave a number of general data on pumping.

THE MARCLEN PUMP.

BY R. A. STEWART.

This pump differs from other centrifugal pumps in being glandless. Essentially, it consists of a casing and a rotor or impeller with a vertical spindle. This method of construction has been adopted in order that glands may be dispensed with.

A belt-driven type of pump specially intended for sulphuric acid is shown in the accompanying figure.



The annular float, A, controls the admission of liquid through the inlet, C, and the annulus, L. The upper sloping part of the float seats itself on the corresponding sloping part of the annular part, which really constitutes a valve seating. The float moves freely in a vertical direction, but is not in contact with the spindle, as it is guided by the small ribs shown as upper extensions of the ribs, M. These ribs, M, of which there are four, form a rest for the float, and guide the liquid steadily into the eye of the impeller, F, which subjects it by centrifugal action to pressure in the chamber, G, whence it is forced through the outlet, H, into the delivery pipe. The impeller, F, is mounted on the spindle, D, which revolves in large bearings, K and O, the spaces between the bushes and main casting in each case forming grease reservoirs. The weight of the spindle and impeller is taken by a ball thrust bearing, N, which revolves in a covered grease chamber. All wearing parts are fitted with automatic grease lubricators. No parts liable to wear are exposed to the action of the acid. The body of the pump, J, the impellers, F and F', the sheathing of the spindle, E, the float valve, A, and annular inlet chamber, B, can be made of any suitable acid-resisting metal, stoneware, ebomite, or glass.

When working a full supply of acid is allowed to flow into the inlet, C, and, should this be too much, the float valve, A, rises and temporarily restricts the supply, but falls again when the level of the acid in the pump body becomes lower.

By a comparatively small modification of the design the pump is equally well adapted for raising other acids. For such purposes the pump may be made in ebomite, earthenware, or silicon-iron alloy, or even in stone. As a matter of fact these pumps have been working for over two years with various corrosive liquids, including concentrated and dilute sulphuric and phosphoric acids and hydro-

chloric acid. Also very strongly alkaline liquors, at the boiling temperature, containing a heavy sediment mixed with ashes.

The following are some of the uses for which this pump is well adapted:—The elevation of hot saline solutions to crystallising vessels; the removal of salts deposited from solutions undergoing evaporation; the emptying of caustic soda or sodium sulphide finishing pots; mixing liquids and solids in dissolving tanks—employed for this purpose the pump vigorously agitates the contents of a relatively very large vessel.

THE ELEVATION OF ACIDS BY THE KESTNER PATENT AUTOMATIC ELEVATOR.

BY J. A. REAVELL.

The use of the acid egg tends to great loss of air, and is generally inefficient. The Kestner

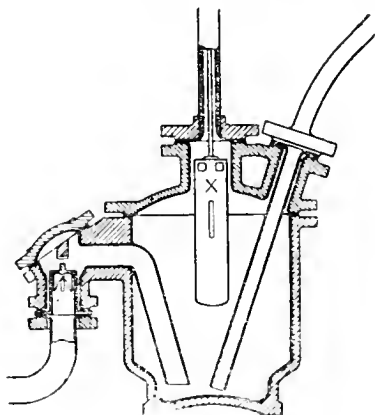


Fig. 1.

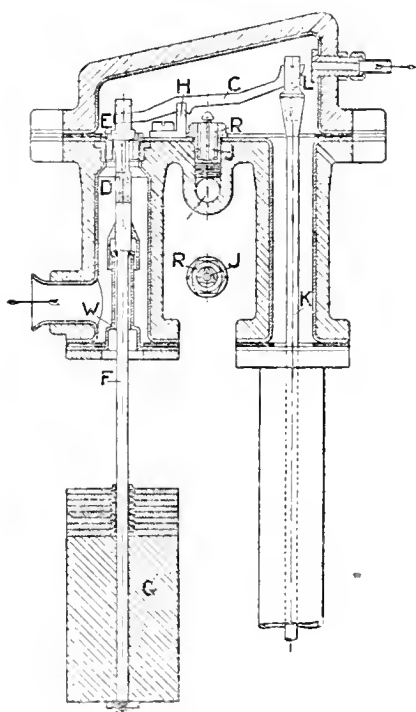


Fig. 2.

elevator was designed to overcome this waste and to do away with the labour required to operate the valves.

The standard elevator is shown in fig. 1, and the movements whereby the air is admitted and exhausted in fig. 2. Acid flows into the body of the elevator through the inlet valve which is coupled to a feed tank placed above it. As soon as the acid rises to a pre-determined height in the body, the float, X, operates. To obtain a quick action the float is carried into the narrow portion

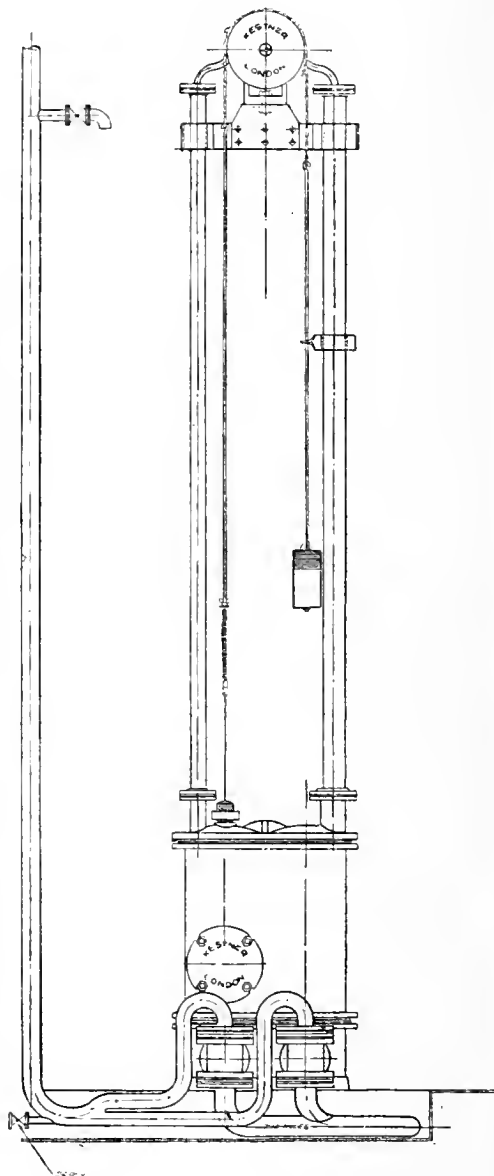


Fig. 3.

of the top cover, and the acid flowing in rapidly fills this. The lever, C (fig. 2), works on the fulcrum, H, and the rod, K, which is coupled to the float, rises when the body is full. This at once releases the pressure from the air valve, R, and air is admitted into the head, passing down the standpipe into the body. At the same time, the pressure of the air closes the air exhaust valve, D. The compressed air blows the acid from the body up the delivery pipe to the receiving tank. A balance weight rod, F, provided with balance weights counterbalances the weight of the float.

As soon as the acid is blown out of the body, the float comes back to its original position and the exhaust valve opens at once, allowing the air to exhaust. The acid inlet valve then opens, and the cycle is repeated. The movement of the lever is very slight, the air valve having a travel of only about $\frac{1}{8}$ inch, so that the wear and tear is infinitesimal.

The stand-pipe should be of sufficient length so that the elevator head is above the level of the acid in the feed tank, thus preventing the flooding of the head with acid.

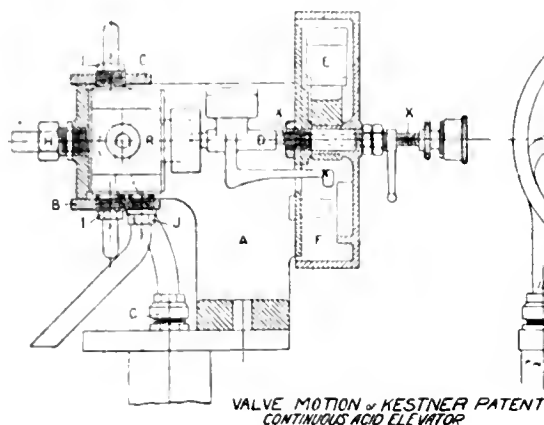


Fig. 4.

In a modification of this elevator, made of earthenware, for pumping nitric acid, the special feature is the method of connecting the float to the lever by means of a wire. Originally this wire was of platinum, but recently a special alloy has been used.

A continuous elevator has been designed for delivering acid into sprays, and for other purposes. This is shown in fig. 3. The lower portion consists of two bodies which may be made of cast iron lead lined, or unlined, as required, or of potware.

One of these bodies contains the float, which is operated by the valve shown in fig. 4. This consists of a taper plug type valve operating in a valve box, both of which are made of potware. The air is delivered into the head at H. The plug valve, R, is arranged with four ports which communicate with the openings marked 1 on the horizontal plane, and 2 on the vertical plane. As the float rises, owing to the inlet of the acid, the wheel, F, is rotated until the trigger, K, which operates in a slot in the wheel, comes in contact with the wheel. This then moves over B and causes the weight, E, to fall sharply, thus operating the valve, R, quickly. This operation turns on air to the cylinder that has just filled, and shuts it off from the cylinder that has just been emptied, exhausting this to atmosphere. There are two openings for each cylinder. For one cylinder the diagram shows these at 1 and 1A. Both these connect in a common pipe to the cylinder, 1B. If, for example, the valve has just operated on No. 1 cylinder due to it being full, then the compressed air enters through 1 into No. 1 cylinder and the exhaust from No. 2 cylinder leaves through pipe, 2B, by way of the valve, 2A, and vice versa—each cylinder filling and emptying alternately.

These elevators, which have been used for discharging acid through sprays at 40 to 60 lb. pressure on the sprays, are simple in operation and have no working parts revolving in acid, the only parts subject to the acid being the inlet and outlet acid valves. Some of them have been con-

tinuously in operation in chemical works for over fourteen years and the cost of upkeep amounts only to some few shillings a year.

CENTRIFUGAL PUMPS FOR SULPHURIC AND NITRIC ACIDS.

BY S. J. TUNGAY.

The centrifugal pump has many advantages over pumps of other types for lifting sulphuric acid. Their efficiency is high as compared with the use

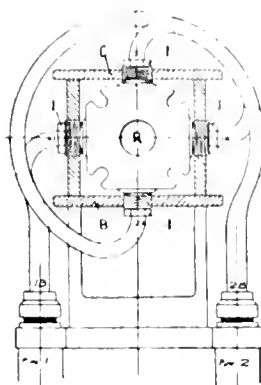


Fig. 1.

Fig. 1 shows a 3-stage centrifugal pump again so arranged that there is but one stuffing box and

gland, with a thrust collar at the pressure side. This has been arranged with a by-pass leading to the first or suction chamber so that it is always subject to suction while working, and any leakage or overflow at this point is carried back to the suction side. This forms a very compact and highly

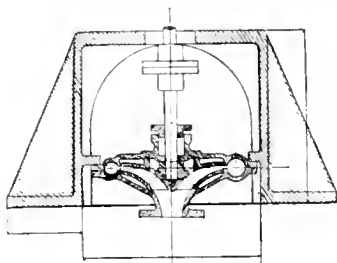


Fig. 2.

efficient design, the impellers being carefully balanced and internal friction reduced to a minimum.

Fig. 2 shows a single-stage vertical type centrifugal pump arranged for driving by vertical spindle, motor-coupled direct to pump shaft. The pump chamber has a bottom suction fitted with a balanced impeller, with a faced ring on the impeller working in a machined groove on the body in such a way that the gland packing has very little work to do. A ball bearing is arranged on the motor spindle and the pump works with but little internal friction. The multi-stage pumps are often arranged in the same manner.

A centrifugal pump which has been very successful for pumping nitric acid is shown in fig. 3. This pump is constructed entirely of acid-resisting iron, with the exception of the bed-plate. Arranged as

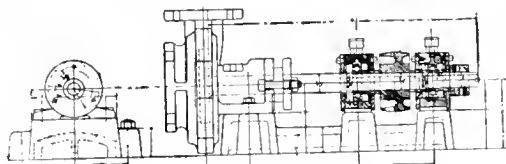


Fig. 3.

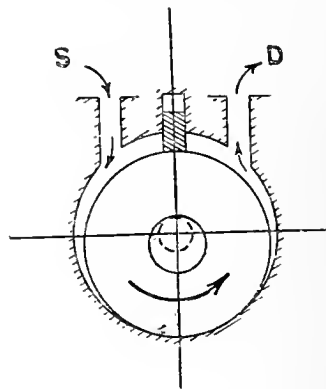
shown with an end suction, the impeller and spindle are in one piece. A long stuffing box and sleeve are adopted and oil grooves machined in the spindle and kept filled with ceresin wax lubricant prevent trouble from leakage at the gland. The spindle is supported by two ball bearings, totally enclosed, enabling the pump to be run at comparatively high speeds with a minimum of friction or wear and tear. Some of these small pumps have raised 85% nitric acid to a height of 60 ft. continually at temperatures up to 52° C. In one case five pumps pumped 22,000 tons of nitric acid without expenditure for maintenance.

THE AVAMORE "SQUEEGEE" PUMP.

BY R. A. PELMORE.

This pump, invented by Mr. N. G. Beckwith, comprises a rotor consisting of a disc loosely mounted on an eccentric on the shaft rolling round the inside of the pump casing. No sliding takes place between casing and rotor, but rolling motion is imparted to the rotor, which turns round the eccentric, the latter acting as a bearing. At the top of the casing a metal piece, usually with some spring tension behind, works up and down in a slot, keeping contact at all times with the rim of

the disc. Inlet and outlet ports are immediately to right and to left of this metal piece or valve. "Squeegee" action drives the liquid round in front of the rotor; the liquid, being sealed from the suction side by the valve piece, is driven through the delivery port, while at the same time liquid follows behind the rotor through the suction port.



The pump consists of a casing, end covers, a shaft and two discs with two valve pieces. The discs and valves are duplicated for perfect balance, even flow, and silent running.

The casing and rotor do not have sliding wear against each other. The sliding surfaces are the main bearing bushes for the shaft and the bore of the rotor disc on the eccentric. The discs rotate round their own centres at a speed proportional to the difference in diameter of casing and rotor, so that for a pump speed of, say, 1000 r.p.m. the rim of the disk only makes about 60 complete turns per minute past the valve piece, so making the wear there negligible.

The constructional simplicity and the restriction of principal wearing surfaces to bearings, as well as the absence of valves, without sacrificing the positive action or ability to run without priming, make a pump of extraordinary reliability.

By the addition of a simple attachment the delivery and suction connexions remain the same for either direction of rotation.

METHOD OF TRANSPORTING AND DISTRIBUTING LIQUIDS ABOUT A FACTORY.

BY H. W. CROW.

The device which is the subject of this note† provides a means of putting any given vessel in a factory into communication with any other by a kind of switch table, avoiding cocks, valves, and branch pipes, either directly or through the intermediary of a pump. Pipe lines are brought from a number of vessels to one point, terminating in such a way as to make a circle of short vertical pipes at the circumference of a table. The end of each short pipe is fitted with a cone union. Revolving round the centre of this circle are two radiating arms, one end of each capable of connection to any of the cone unions and the other ends connected to two loops, one above and one below, the further ends of which are connected with one another either through two bridge pieces and the suction and delivery of a pump or through one bridge piece direct. In this way any one of the short vertical pipes of the circumference of the table can be brought into connexion with any other and therefore any one of the vessels with any other of the group. By making

† See Eng. Pat. 130,946.

one or more of the short vertical pipes lead to corresponding short vertical pipes of other tables further groups of tanks can be brought into inter-communication.

It is preferable to arrange the tables at a higher level than the highest vessel. If this is not practicable small pet cocks should be fitted at the highest point in the pipes leading to the various vessels to prevent siphoning.

The table may be used in conjunction with its own pump or not. When the pump is not required one of the bridge pieces is swung on one side and the other connected straight across the two loop terminals. When an operation is completed it is possible, by slowing down the pump, to sweep the pipes all but free from liquid by allowing air to be sucked through.

PUMPING ACIDS AND OTHER CORROSIVE LIQUIDS, WITH PARTICULAR REFERENCE TO THE ARMoured CERATHERM CENTRIFUGAL PUMP.

BY W. HAYHURST.

Pumps for acid or corrosive liquids should be simple in construction and should, if possible, have no internal rubbing surfaces or bearings; there should be no contact between the corrosive liquid and material with which it can react; valves should be avoided if possible; any oil used should not come into contact with the corrosive liquid; the pressure developed by the pump should be easily variable from zero to about 50–60 lb. The type of pump which best fulfils these requirements is the centrifugal. Ceramic material is the most suitable for the construction of pumps for the chemical trade. The type chosen by the author for his pump was a form of Ceratherm, with considerably increased tensile strength and with sufficient heat-resisting capacity to enable the pump to be worked alternately on hot or cold liquids without liability to fracture.

In the Guthrie Ceratherm pump the casings are made exceedingly thick and strong. They are set into the iron armourings by means of acid-resisting cement. The inlet and outlet are so constructed that coupling up can be accomplished without any fear of damaging the pump. The acid or corrosive liquid is fed into the pump on the gland side. No trouble is experienced in packing this gland, which is continually under suction, a small quantity of wool, soaked in paraffin wax, if desired, being pressed home with the fingers. Heads of 200 or 300 ft. can be attained without any leak or trouble at the gland.

To fix the impellers on to the steel shaft the interior of the shaft of the impeller is filled with a cementing material of very high acid resistance, which receives a screw thread and the shaft is then screwed into the impeller, which is ground and balanced on its own shaft. The impellers are of very substantial construction. They have been tested up to fully 3000 revs. The Manx impeller, which is now being used largely, is almost self-balancing so far as thrust is concerned and gives very little thrust along the shaft.

In chemical works it is generally required to pump a small quantity (say 20–100 galls. per min.) to a head of anything up to 120 ft., whilst running the pump at low speeds. This means that the ratio of the diameter of the impeller to the width of its blade is necessarily high, and as this ratio is a factor which governs the theoretical efficiency of any centrifugal pump a corresponding reduction of efficiency would be expected.

In the neighbourhood of 40 ft. head efficiencies of 60% have been attained with these pumps, and with 120 ft. head about 40%.

Yorkshire Section.

Meeting held at Queen's Hotel, Leeds, on February 23, 1920.

MR. W. McD. MACKEY IN THE CHAIR.

THE DETERMINATION OF ALKALINITY IN TANNERY LIME LIQUORS.

BY W. R. ATKIN, M.Sc., AND W. E. PALMER, B.A.

(Abstract.)

The methods to be described have been devised with a view to overcoming certain defects in the methods at present employed in the determination of alkalinity in lime liquors. The factors which contribute to the alkalinity of lime liquors are: (a) calcium hydroxide, (b) sodium hydroxide, (c) sulphhydrates of calcium and sodium, (d) ammonia and amines, (e) sodium and calcium salts of weak acids or products of hydrolysis of hide proteins.

Total alkalinity is due to all the substances enumerated above and is determined by the method described by Bennett,¹ in which 25 c.c. of the filtered lime liquor is boiled with excess of N/10 sulphuric acid and the excess titrated with N/10 caustic soda, using methyl red as indicator. In all the lime liquors examined a precipitate was formed by this treatment with acid; these precipitates are due doubtless to dissolved keratinous matter and not to true hide substance, as was demonstrated by one of us recently.² This point was further tested by determining the total alkalinity of a solution of gelatin in lime water, in which case no precipitate was obtained on addition of acid. A similar result was noted with a solution of hide powder in lime water, although both solutions were very rich in nitrogen. By using N/10 reagents throughout, the titration for total alkalinity is a measure of $a+b+c+d+e$.

Caustic alkalinity is an important figure for the tanner, as it is a measure of the plumping power of the lime and has a pronounced influence on the subsequent leather produced. This caustic alkalinity is due to the hydroxides of calcium and sodium or to $a+b$.

Bennett³ has devised a method to determine this figure which is very ingenious, but in one important respect unsound. It depends on two titrations of the lime liquor after treatment with formaldehyde.

First titration.—10 c.c. of 40% formaldehyde solution diluted with 10 c.c. of distilled water is neutralised to phenolphthalein with N/10 caustic soda until a faint pink colour appears; 25 c.c. of the clear filtered lime liquor is added, and, after mixing, titrated with N/10 hydrochloric acid until the pink colour just disappears. During this titration the hydroxides of calcium and sodium are neutralised by the acid and not affected by the formaldehyde. Ammonia reacts with the formaldehyde to form neutral hexamethylenetetramine. Formaldehyde reacts with sulphhydrates and liberates an equivalent amount of hydroxide: $\text{NaSH} + \text{HCHO} + \text{H}_2\text{O} = \text{H.CH(OH)(SH)} + \text{NaOH}$, and thus all sulphhydrates are estimated. Formaldehyde also reacts with amino acids, producing methylene-amino acids, which react acid to phenolphthalein: $\text{NH}_2\text{R.COOH} + 11\text{HCHO} = \text{CH}_2\text{:N.R.COOH} + \text{H}_2\text{O}$. Consequently, after adding formaldehyde to a lime liquor methylene-amino acids are formed which neutralise a certain amount of the caustic alkalis, so that the caustic alkalinity and that due to sulphhydrates are reduced by this acidity produced by the amino acids. In other words, this titration

¹ Bennett. Coll. (London) 1915, 260.

² Thompson and Atkin. J. Soc. Leather Tr. Chem., 1920, 15.

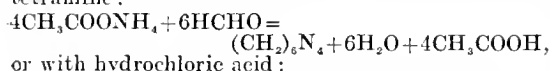
³ Bennett. Coll. (London), 1915, 321.

affords a measure of $a+b+c$ —amino acids. The above explanation is that put forward by Bennett, and is quite sound. The calcium and sodium salts of the hydrolysis products are not affected in this titration.

Second titration.—25 c.c. of filtered lime liquor is neutralised to phenolphthalein with glacial acetic acid until the pink colour is discharged, and $N/10$ iodine added until an unabsorbed excess is observed. $N/10$ caustic soda is then added until a pink colour appears, and finally 10 c.c. of neutral formaldehyde. After mixing the solution is titrated with $N/10$ caustic soda until the pink colour reappears. Later this titration (originally described by Stiasny)⁴ was modified by using hydrochloric acid instead of acetic acid, as the end-point was found to be sharper.

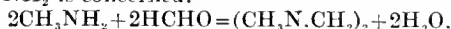
Bennett states that this second titration is a measure of the acidity produced by the amino-acids, the caustic alkalis having been neutralised by the acid and the sulphhydrates decomposed by the iodine; consequently the number of c.c. of $N/10$ caustic soda found in the second titration added to the number of c.c. of $N/10$ hydrochloric acid for the first titration gives a measure of $a+b+c$.

Unfortunately he has ignored the fact that the free ammonia often present in considerable quantities has been neutralised with the formation of ammonium acetate (or ammonium chloride), which reacts with formaldehyde to liberate an amount of acid equivalent to the amount of ammonia present in the lime liquor and also to form hexamethylene-tetramine:



This omission is curious, as Bennett⁵ himself advocated the use of formaldehyde to replace the distillation with caustic soda in the Kjeldahl method of estimating nitrogen.

No mention has yet been made of the action of amines on formaldehyde. Primary amines, such as methylamine, seem to react with formaldehyde in a somewhat analogous way to amino acids as far as the NH_2 is concerned.



With their hydrochlorides hydrochloric acid is liberated, just as in the case of ammonium chloride. This point was tested experimentally with methylamine hydrochloride.

The hydrochlorides of secondary and tertiary amines apparently do not behave in the same way as those of the primary amines. However, Effront⁶ has tested for trimethylamine in many different putrefactions, but has not succeeded in detecting it, and states that only materials containing betaine or trimethylglycine give rise to trimethylamine.

The constitution of the amino-acids known to be components of hide proteins does not suggest the likely formation of di- or tri-methylamine by putrefaction. Accordingly only a relatively small error will be introduced by assuming that the whole of the ammonia and amines obtained by distillation reacts with formaldehyde, causing the liberation of an equivalent amount of hydrochloric acid. Thus the second formaldehyde or Stiasny titration measures the amino-acids plus ammonia and amines, or d +amino acids. The sum, therefore, of the two formaldehyde titrations is a measure of $a+b+c+d$, and by subtracting $e+d$, that is c.c. $N/10$ acid required for ammonia, amines, and hydrosulphides, $a+b$ is obtained, which is the caustic alkalinity of the lime liquor.

⁴ Stiasny. Coll., 1908, 373.

⁵ Bennett, this J., 1909, 292.

⁶ Effront. Biochemical Catalysts in Life and Industry, p. 665 (Wiley.)

Communications.

ESTIMATION OF ALCOHOL IN MIXTURES OF ALCOHOL, SULPHURIC ACID AND WATER.

BY G. S. BUTLER, B.A., F.I.C., AND H. B. DUNNICLIFF, M.A., B.Sc., A.I.C.

In the course of an investigation on the possibility of recovering sulphuric acid from nitre cake by spirits of wine, a large number of solutions of sulphuric acid in alcohol were obtained in which it was necessary to have a reliable method of estimating the constituents. It was hoped that the method due to F. G. Benedict and R. S. Norris (J. Amer. Chem. Soc., 1898, 20, 293) would be suitable, but it was not found possible to get concordant results by this or by several other methods.

In all alcoholic sulphuric acids partial esterification is observed, even in the presence of considerable quantities of water. The method to be described involves the estimation of the alcohol both as ethyl hydrogen sulphate and as free alcohol. It has been found that if the free sulphuric acid is estimated by titration by the method to be described, the results show good agreement with those obtained by precipitation of the free sulphuric acid as barium sulphate and weighing the precipitate. This shows that, during titration, no appreciable hydrolysis takes place.

The combined alcohol is estimated by determining the amount of acid which has undergone esterification. A weighed quantity of the alcoholic sulphuric acid is titrated against standard alkali. This "direct" titration gives b (see Table I.) the percentage of acid present in terms of sulphuric acid, i.e., it gives the free sulphuric acid and the acidity due to ethyl hydrogen sulphate (in terms of sulphuric acid).

The "total" acidity is found by evaporating a weighed quantity of the alcoholic sulphuric acid to a pasty consistence with excess of standard caustic soda or potash, heating in an air oven at 120°C . for half an hour, and then, after extraction with distilled water, titrating the excess alkali with standard acid. From this is calculated the percentage acidity of the alcoholic sulphuric acid before any esterification took place. The value is expressed as percentage of sulphuric acid, a . Thus, the percentage of acid esterified = $2(a-b)/a \times 100$.

The weight of esterified alcohol in 100 grams of the alcoholic sulphuric acid is $2(a-b)/a \times 100 \times 46 \div 98$ grams.

TABLE I.

Composition of the mixture calculated from the proportions mixed: Sulphuric acid, 29.10%; ethyl alcohol, 68.98%; water, 1.92%.

No. of days elapsed after mixing.	Sulphuric acid.		Combined alcohol calculated from a and b . %	Free alcohol by distillation. %	Total alcohol found. %
	By direct titration. %	Total. %			
3	19.51	29.08	9.02	59.99	69.01
7	18.95	29.05	9.48	59.20	68.68
13	18.20	29.10	10.20	58.88	69.08
14	18.10	29.10	10.30	58.88	69.18
15	17.96	29.10	10.45	58.56	69.01
16	17.95	29.10	10.46	58.56	69.02
17	17.83	29.10	10.59	58.24	68.83
21	17.64	29.10	10.76	58.24	69.00
23	17.53	29.10	10.81	57.92	68.73
28	17.36	29.10	11.02	57.92	68.94

* Mean of five determinations.

The free alcohol may be determined by weighing out 25 grms. of the alcoholic sulphuric acid, making

faintly alkaline with sodium carbonate, and distilling until about 95 c.c. of distillate has been collected. This treatment does not hydrolyse the ester, and the distillate contains all the uncombined alcohol present. The distillate is made up to 100 c.c., and its density at 60° F. determined. The percentage of alcohol present is obtained from a table, and this quantity multiplied by four gives the weight of uncombined alcohol present in 100 grms. of the alcoholic sulphuric acid.

TABLE II.

The alcoholic sulphuric acids were made up from 97.6% H₂SO₄ and 98.34% C₂H₅OH.

Date.	Uncombined alcohol by distillation method, %	Combined alcohol by titration method, %	Total alcohol estimated, %	Composition of solution calculated from proportions mixed.		
				C ₂ H ₅ OH.	H ₂ SO ₄ .	H ₂ O.
				%	%	%
10-1-20	50.24	7.07	67.21	67.18	30.92	1.00
21-1-20	50.76	10.57	67.33			
12-1-20	55.36	9.98	65.34	65.60	32.50	1.00
21-2-20	54.16	11.44	65.60			
12-1-20	66.52	7.78	74.30	74.09	24.24	1.67
21-1-20	65.28	8.74	74.02			

Table I. gives the results of determinations made for the same solution and showing the slow but continuous increase in esterification up to a maximum with lapse of time. Table II. gives a series of results obtained for alcoholic sulphuric acids of various concentrations. The solutions were made up in each case by mixing weighed quantities of alcohol and sulphuric acid, both of known strength.

The Cordite Factory,

Arurankadu, S. India.

THE ACTION OF CONCENTRATED SULPHURIC ACID ON IRON.

PART III.*

BY CHARLES E. FAWSITT.

There exists some uncertainty as to the safety of transporting concentrated acid in iron or steel drums, and much more investigation is required on the action of sulphuric acid on iron on account of the importance of the matter in chemical industry.

Some of the factors which influence the action are:—(a) the kind of iron used—this depends on the chemical composition and also on the structure of the metal; (b) the nature of the surface of the iron, i.e., whether rough or smooth, or covered by any film of solid, liquid, or gaseous substance (Fawsitt, this J., 1906, 1134); (c) the concentration of the acid; (d) the temperature; (e) the presence of impurities, or additions of foreign substances to acid or iron; (f) mechanical influences, such as shaking.

The factors under headings (a) and (d) were to some extent discussed in the first paper (this J., 1914, 234). Some work under heading (c) has been done by Knietsch (this J., 1902, 343), and a record of further work has appeared in a recent paper (Fawsitt and Pain, Proc. Roy. Soc. N.S.W., 1918, 52, 396; see this J., 1920, 193 A).

The manner in which the addition of water increases the rate of action on iron depends apparently on the kind of iron used. Using steel wire containing C 0.51, P 0.016, Si 0.083, S 0.058, Mn 0.48%, the velocity of action, found by Fawsitt and Pain, is as indicated in Table I. When the acid and steel were shaken together the rate of action was measured by weighing the steel before

and after action, but these results are also calculated to show the average volume of gas evolved.

TABLE I.

Weight of steel used, 5 grms.; surface, 36 sq. cm.; temperature, 30° C.; time, 28 days.

Concentration of sulphuric acid (% of H ₂ SO ₄).	Average vol. (for 28 days) of gas evolved in c.c. per hour per sq. cent. of steel surface.	Steel and acid shaken together.	
		Loss of weight of steel in 28 days (grms.).	Calc. average vol. of gas evolved during 28 days (c.c. per hr. per sq. cent.).
97.4	—	0.090	0.17
91.0	0.27	0.861	1.46
90.0	0.31	1.222	2.07
89.3	0.66	0.140	0.21
87.9	0.28	0.523	0.89
85.0	0.30	0.526	0.89
80.0	2.5	2.915	4.91

With the particular class of steel used here, the velocity increases as the acid is diluted from 97% to 90.9% H₂SO₄; it then appears to drop rather suddenly at the next concentration (89.3%). After the concentration has dropped to 85% the velocity increases very decidedly. It would be of interest to examine the effect of concentration on velocity rather exactly between 91% and 88% H₂SO₄.

Further experiments under heading (f), carried out since the publication of the first paper, all show that shaking has an important effect in increasing the velocity of action.

Experiments would indicate that with commercial acid and any particular kind of iron, a certain maximum rate of action is sometimes capable of being realised. When a drum of acid is being transported it is safer to assume that the maximum rate of action may take place, and the maximum pressure of gas, after a given time, could be calculated from the dimensions of the drum, the volume of the air space above the acid, the temperature, and laboratory experiments with the same iron and the same acid. The maximum rate of action is, however, not always attained.

It has sometimes been found that although there may be a pressure of gas in one drum of acid, there is little or no pressure in other drums of the same kind, filled with the same acid, and exposed to exactly the same conditions, as far as can be judged. So long as calculations are based on the maximum rate of action, no harm can result from a smaller rate being actually observed, but it is disconcerting to find this great difference in behaviour between a number of drums.

Among other considerations, it is possible that the reason the iron is not always acted on at the maximum rate is that the iron may sometimes go into a passive state, and so not be acted on by the sulphuric acid. There may be more than one kind of passive iron, but the readiest means of producing a passive iron is to leave iron for a few moments in nitric acid of sp. gr. 1.4. It may easily be shown by E.M.F. measurements that iron, lying in concentrated sulphuric acid, and apparently not being acted upon by the acid, is not usually in a passive state. On the other hand, passivity is recorded (Heathcote, this J., 1907, 899—917) as being produced on iron sometimes simply by exposing iron to air. The author has never come across a case where a piece of iron or steel, that had been lying in the air (and otherwise untreated), showed passivity towards concentrated sulphuric acid; but if the iron of a drum could become wholly (or perhaps even partially) converted into passive iron, by lying in the air or otherwise, it is quite possible that such iron might retain its passivity after the sulphuric acid had been put in.

* For Parts I. and II. see Fawsitt and Powell, this J., 1911, 234.

Many experiments have been carried out by the author, where steel that had been wholly treated by nitric acid of sp. gr. 1.4 was passive towards concentrated sulphuric acid. The passivity is detectable both by E.M.F. measurements and by the observation that little or no solution of the steel takes place. Every case tried did not succeed.

A curious effect has been noticed by the author. If a piece of steel is dipped for only part (say half) of its length into nitric acid of sp. gr. 1.4 and then dipped wholly into concentrated sulphuric acid, in many cases no action on the steel is apparent for several days at least,* either on the lower (passivified) part or the upper part. This might be thought to be due to a "wave of passivification" passing over the whole iron (just as the activating proceeds by propagation from one part of the iron to the other; see Heathcote, this J., 1907, 899—917). This is not the real explanation, however, as the upper portion originally not acted on by the nitric acid becomes immediately coated with a white film of ferrous sulphate, while the lower portion, which has been acted upon, is unaffected. The upper part, although originally of a greater solution pressure than the lower, probably becomes polarised and will no longer dissolve.

Experiments were also carried out with the addition of certain substances to the acid in the hope that the action might be found to be decreased by certain additions, and the results are shown in Table II. Unfortunately no experiment has yet shown that any addition to the acid is decidedly helpful in preventing action. In these experiments the tubes containing 40 c.c. of acid and 3.5 grms. of steel were not shaken. The steel was weighed before and after each experiment. On lifting the tube from the thermostat, the acid was poured off as completely as possible and the steel washed twice with alcohol, then with water, and finally with alcohol; it was then dried and weighed. The surface of the steel exposed to the acid in each case was approximately 25 sq. cm. Before using fresh steel wire in these experiments it was first treated with sodium hydroxide solution, and then with water. In many cases, however, the steel was used in a second experiment; it was then used exactly as it came from the first experiment (after washing with alcohol and drying).

The same steel wire was used in all the experiments as already described for Table I. (*loc. cit.*)

It will be noticed that although preliminary treatment with concentrated nitric acid may to a certain extent preserve the steel from being afterwards attacked by the concentrated sulphuric acid, putting nitric acid or nitrates into the sulphuric acid increases the rate of action very much.

While dipping iron into concentrated nitric acid either wholly or partially and then placing in concentrated sulphuric acid may bring about a state of passivification which persists for some considerable time, this is not by any means always the case.

When the passivification is either not effected, or only effective for a few hours or days, the small quantity of nitric acid in the solution enormously increases the rate of action of the acid on the iron. This is shown by the results of several experiments at 30° C. for 44 days with 96.3% H_2SO_4 , using 3.5 grms. of steel in each case. In the first two experiments, in which the steel received no preliminary treatment, the loss of weight was 0.132 gm. and 0.238 gm. respectively. In another experiment one-third of the steel was covered with concentrated nitric acid; this was poured off and concentrated sulphuric acid added to cover (the same) one-third of the steel and poured off. Then 40 c.c. of concentrated sulphuric acid was added. In this case the loss of weight was 0.047 gm. In a further experiment with the same treatment as the last, but with only one-quarter of the steel treated with

nitric acid, the loss of weight was 1.37 grms., whilst when the whole steel was treated with concentrated nitric acid the loss was 1.52 grms. It appears, then, that although preliminary treatment of the steel with concentrated nitric acid cannot yet be recommended as a means to be generally used in preventing subsequent action of the sulphuric acid, this is not an unpromising line for further experiment.

TABLE II.

No. of experiment.	Temperature.	Concentration of sulphuric acid (by analysis).	Additions to sulphuric acid.	Time (in days) of immersion in acid.	Loss of weight of steel, grms.
1	28° C.	91.5	None.	18	0.48
2	28° C.	91.5	0.04 gm. potassium persulphate.	18	0.44
3	28° C.	91.5	0.08 gm. sodium sulphate.	18	0.43
4	28° C.	91.5	0.06 gm. sodium hydrosulphite.	18	0.44
5	28° C.	91.5	0.09 gm. potassium bisulphate.	18	0.22
6	30° C.	91.5	None.	4	0.068
7	30° C.	91.5	0.019 gm. potassium nitrate.	4	0.124
8	30° C.	91.5	None.	13	0.19
9	30° C.	91.5	0.016 gm. arsenious oxide.	13	0.103
10	30° C.	96.3	None.	13	0.064
11	30° C.	96.3	0.06 gm. arsenious oxide.	13	0.078
12	30° C.	96.3	0.052 gm. lead nitrate.	13	0.393
13	30° C.	96.3	0.08 gm. HNO_3 (sp. gr. 1.4).	13	0.58
14†	30° C.	96.3	None.	13	0.015
15†	30° C.	96.3	None.	13	0.0257

† In experiments 1—13 the iron received no special preliminary treatment. In experiment 14 the steel was wholly covered with nitric acid of sp. gr. 1.4, this was poured off, sulphuric acid added and poured off; a little more sulphuric acid added and poured off; then the usual 40 c.c. conc. sulphuric acid added. In experiment 15 one half of the steel was covered with nitric acid of sp. gr. 1.4 which was then poured off and 40 c.c. conc. sulphuric acid added; strong action took place at first, but after 2 days apparently no action was going on.

It was also thought advisable to try the effect of putting certain metals in contact with iron while immersed in concentrated sulphuric acid. A metal might be expected to dissolve more readily in an acid if it were in contact with a metal electro-negative to it. This effect, however, is sometimes obscured by polarisation effects when both the metals are completely covered by the acid. Variable and no encouraging results were obtained with the use of either aluminium or platinum in contact with iron (Table III.).

It was not thought worth while to try metals like magnesium or zinc, because it was considered that as concentrated sulphuric acid acts on these metals, gas would in any case be evolved, even if they protected the iron.

TABLE III.

Temperature.	Concentration of acid, H_2SO_4 , %.	Metal touching steel.	Time of immersion in acid, Days.	Loss of weight of steel, grms.
30° C.	91.5	None.	4	0.068
30° C.	91.5	Mercury.	4	0.061
25° C.	91.5	None.	11	0.134
25° C.	91.5	Platinum.	11	0.123
25° C.	91.5	Aluminium.	11	0.071
25° C.	96.3	None.	27	0.048
25° C.	96.3	Platinum.	27	0.048
25° C.	96.3	Aluminium.	27	0.055

A possible line of investigation seems, therefore, to be one where the iron is previously made passive under such conditions that no nitric acid gets into the sulphuric acid.

Chemical Department,
University of Sydney.

* A momentary action takes place on the untreated portion.

Birmingham Section.

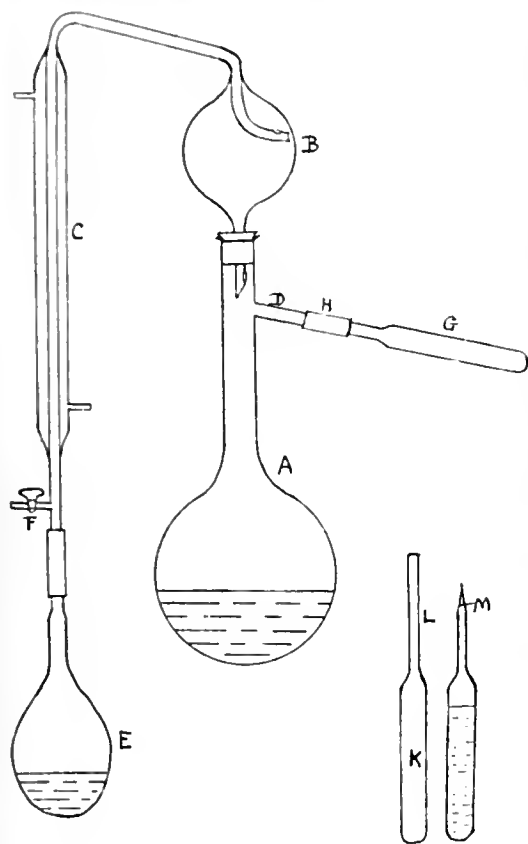
Meeting held at Birmingham University on
Thursday, March 25th, 1920.

MR. L. P. WILSON IN THE CHAIR.

THE ESTIMATION OF CARBON DIOXIDE AND FERMENTABLE SUGARS.

BY ARTHUR SLATOR, D.S.C.

The apparatus described in this communication was devised originally to estimate the total amount of carbon dioxide formed when sugars are fermented by yeast. It was subsequently found to be of general use, and can be employed in most cases where it is necessary to estimate this gas. The apparatus consists of a distillation flask, A, con-



nected to a condenser, C, in the manner shown in the diagram. The large trap, B, prevents spray and froth being carried over during the distillation of carbon dioxide from A to the "assay" flask, E, which contains standard baryta water. G is a test tube to which has been sealed a piece of glass tubing. This tube can be connected to the side piece, D, by means of pressure tubing, H. The whole apparatus can be exhausted through the stopcock, F. The estimation of carbon dioxide in sodium carbonate is carried out in the following manner: 25 c.c. of N/10 sodium carbonate is placed in A, and G is filled with dilute sulphuric acid. Into E is introduced 30 c.c. of N/10 baryta water. The apparatus is exhausted, a screw clip being

placed at H to prevent the acid in G passing into A. F is then closed and the clip removed. The acid in G is warmed and boils into A. Both A and G are then well boiled and E shaken to absorb the gas. When the distillation is complete, air free from carbon dioxide is allowed to enter the apparatus through F. The excess of baryta in the turbid solution in E is then titrated with standard hydrochloric acid, using phenolphthalein as indicator. Previous tests made by titrating baryta water containing a suspension of barium carbonate showed that rather more standard acid is required to change the bright pink colour to a faint pink than is necessary when no carbonate is present. The correction amounted to 0.15 c.c. of N/10 solution. In these estimations this correction is added to the apparent amount of alkali neutralised by the carbon dioxide. 25 c.c. of N/10 sodium carbonate was found to yield carbon dioxide sufficient to neutralise 25.05, 25.15, 24.7, 25.05, 25.05 c.c. of N/10 baryta water (theory 25.0 c.c.).

A drinking water was analysed for total carbonate. Dilute sulphuric acid was placed in A. G was replaced by a funnel through which the water to be analysed was run into the exhausted apparatus. 250 c.c. was found to yield CO_2 equivalent to 14.25, 14.25 c.c. of N/10 alkali. The same water showed 15.5° temporary hardness (parts CaCO_3 in 100,000) by direct titration, using methyl orange as indicator. The figures show slight excess of lime over the carbon dioxide calculated as bicarbonate. The carbon dioxide in calcium carbonate can be estimated by distillation with phosphoric acid. A specimen which was practically pure (0.2 g. neutralised 40.1, 39.9 c.c. of N/10 HCl) gave the following result: 0.2 g. CaCO_3 gave CO_2 which neutralised 40.0, 39.95 c.c. of N/10 baryta (theory 40.0 c.c.).

Free carbon dioxide in liquids can be estimated by filling G with the liquid and subsequently distilling off the carbon dioxide. Carbon dioxide formed in certain oxidation reactions can be collected and estimated by means of this apparatus. 0.0953 g. of potassium tetraoxalate $\text{KH}_2(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$ was oxidised in the apparatus with slight excess of acidified permanganate solution and found to yield CO_2 to neutralise 29.9, 30.15 c.c. of N/10 baryta water (theory 30.0 c.c.).

These examples show the general use to which the apparatus can be put for estimating carbon dioxide. Special precautions have to be observed if volatile acids other than carbon dioxide are present, and if insoluble salts are present care has to be taken that all the carbonate is acted upon by the acid, but in most cases the method is very satisfactory.

The estimation of sugars by alcoholic fermentation offers possibilities which are of some interest. If the fermentation reaction can be put on a quantitative basis, it should be possible to estimate sugars and mixtures of certain sugars and other carbohydrates without utilising the optical activity or the reducing properties of the sugar. Pure cultures of yeasts containing definite enzymes have been used successfully by Davis and Daish (J. Agri. Sci., 1913, 5, 437) to estimate sugars. The fall in reducing power or the change in optical activity after complete fermentation measures the amount of sugar present. The idea here is to extend the use of these pure cultures and employ the fermentation reaction itself to estimate the sugar. The matter was tested in the following way.

Into tubes (K) were introduced measured quantities of solutions of pure sugars, 2 c.c. of yeast water (sp. gr. 1.017) and water to make up the volume to about 12 c.c. The tubes were plugged with cotton wool and then sterilised. Each tube was seeded with a pure culture of yeast, the tube heated at L and the glass pulled out to a fine capillary. The tube was then exhausted and sealed at L. Fermentation was allowed to proceed at 25° C., the tubes

being shaken each day. The carbon dioxide produced was estimated in the following way. A file mark was made at M on the tube, which was then connected to the apparatus. After exhausting the flask and boiling the water in A, F was shut off and the end of the tube broken at M in the pressure tubing. The tube and flask were then both warmed and all the carbon dioxide collected in E.

The following results were obtained by fermenting about 0.1 g. of sugar with a pure culture of brewery yeast and with a culture of *S. apiculatus*, which ferments dextrose and levulose but not maltose or sucrose.

The yeast water gave $\text{CO}_2 = 0.15$ c.c. N/10 baryta water, and the correction was made on the final titration.

In Table I A=c.c. N/10 baryta water neutralised by CO_2 from 0.0988 g. of dextrose (theory 21.9 c.c.) by fermentation with a pure culture of brewery yeast. B=ditto from 0.09895 g. of sucrose (theory 23.2 c.c.). C=c.c. from 0.0960 g. of dextrose fermented by the *apiculatus* yeast (theory 21.35 c.c.).

TABLE I.

	A.	B.	C.
After	cc.	cc.	cc.
3 days	20.2 ..	19.5 ..	18.8 ..
4	21.35 ..	21.15 ..	20.3 ..
5	21.85 ..	22.6 ..	20.65 ..
7	21.95 ..	23.15 ..	20.3 ..
8	— ..	— ..	20.7 ..
11	21.75 ..	23.0 ..	20.75 ..
14	21.95 ..	23.3 ..	20.7 ..
23	21.85 ..	— ..	— ..
Average of last 3	21.85 ..	23.15 ..	20.7 ..
Per cent. of theory ..	99.8 ..	99.8 ..	97.0 ..

In A and B carbon dioxide equivalent to the sugar originally present has been evolved, in C lower results have been obtained, but even here the degree of accuracy is as great as one would expect from a biological method of this kind. In the first two cases the claim is not made that the sugar is completely fermented according to the equation $\text{C}_6\text{H}_{12}\text{O}_6 = 2\text{CO}_2 + 2\text{C}_2\text{H}_5\text{OH}$. Probably about 95% of the carbon dioxide is produced according to this equation, another 2% comes from the auto-fermentation of the yeast, and the rest from the auto-digestion of the yeast. In the last case carbon dioxide may be produced from substances other than sugar.

A test was made to determine if the method could be applied to estimate some of the constituents of ordinary brewery wort. A wort of gravity 1.040 contains approximately 10 g. of dissolved substance in 100 c.c. Of this 10 g. about 1 g. is dextrose and levulose, $\frac{1}{4}$ g. is sucrose, $\frac{5}{16}$ g. maltose and fermentable maltodextrins, 2 g. dextrin and unfermentable maltodextrins, and $\frac{1}{2}$ g. proteins, mineral salts, etc. Fermentation with the *apiculatus* yeast should estimate dextrose and levulose only. 1.5 c.c. of wort (gravity 1.055) was introduced into the tube, diluted and fermented in the manner described. It gave CO_2 neutralising 3.7, 3.8, 3.6 c.c. of N/10 baryta water. Further experiments were made in which small quantities of dextrose (0.05—0.10 g.) were added to the wort before fermentation. The increase in the amount of the carbon dioxide produced corresponded to 92.6, 92.6, 92.6, 93.5, 91.9, 92.6 (average 92.6) % of the theoretical amount. The results are consistent, but only 92.6% instead of 97% of the theoretical yield of carbon dioxide is obtained. Using this factor the amount of dextrose and levulose in the original wort = $3.7 \times 100 \times 100 \div (222 \times 92.6 \times 1.5) = 1.20$ g. per 100 c.c. The total amount of fermentable carbohydrates in wort can be determined in the following way: the wort is fermented with yeast, filtered, and the alcohol distilled off from a measured volume. The residue is made up again to this volume and the process repeated. From the gravity of the final residue the total amount fermented can be

calculated. A wort of gravity 1.0393 produced a residue 1.0136. This corresponds to a spirit indication 5.85 or 3.21 g. of alcohol per 100 c.c. This is equivalent to 3.07 g. CO_2 . In a fermentation of this kind about 95% of the theoretical yield of alcohol is obtained. The theoretical yield of carbon dioxide in the above case would be 3.23 g. 2.25 c.c. of wort diluted and fermented gave CO_2 neutralising 32.1, 32.2, 32.4 c.c. of N/10 baryta water (average 32.2). 100 c.c. wort therefore gives $32.2 \times 100 \times 2.2 \text{ g.} \div (2.25 \times 1000) \text{ g.} \text{CO}_2 = 3.15 \text{ g.}$ Though the calculation is only approximate, it is evident that the total amount of fermentable carbohydrates can be obtained in this manner with a fair degree of accuracy.

The conclusion drawn from a number of experiments carried out in this manner is that small quantities of sugars can be estimated by this fermentation method, but that it is advisable to carry out control experiments with pure sugars before using the method for any special purpose. It has been shown that small quantities of sugars can be fermented away by large amounts of yeast within a few hours (J., 1908, 653). It is possible to carry out such fermentations in sealed tubes and estimate the sugars by titrating the carbon dioxide produced. A blank experiment has to be carried out to correct for the auto-fermentation of the yeast. The method, however, is inconvenient, and details of the estimations are not included in this communication.

The presence of certain enzymes in yeasts serves to characterise and identify different races of yeast. Care has to be taken in detecting such enzymes, and some of the results given in the literature are certainly incorrect. The following method is recommended for carrying out such tests. For the detection of invertase tubes containing yeast water, sucrose (0.2 g.) and dextrose (0.02 g.) are inoculated with a pure culture of the yeast. The tubes are exhausted and sealed up. After about a week the carbon dioxide is distilled off and estimated. If no invertase is present in the yeast, carbon dioxide to neutralise about 4 c.c. of alkali is obtained; if present, up to 40 c.c.—50 c.c. is neutralised. The presence of dextrose is an advantage for growth and a certain amount of fermentation is insured. Maltase can be detected by using maltose in place of sucrose. The presence of or absence of galactozymase can be shown by using galactose.

The following results were obtained:

TABLE II.

Yeast.	Enzyme.	Alkali neutralised.	Presence or absence of enzyme.
		cc.	
<i>Apiculatus</i>	Invertase	3	—
	Maltase	3.7	—
<i>Cerevisia</i>	Galactozymase	31	+
Wild yeast A	..	34	+
.. B	..	2.5	—
.. C	..	32	+

Many methods of estimating carbon dioxide have been described in the literature on the subject, and no great originality can be claimed for this apparatus. It is, however, of general use, and can be applied especially to estimate carbon dioxide from fermentation reactions.

Prof. G. T. MORGAN said that in 1904 he had suggested the use of phosphoric acid in the estimation of carbon dioxide in naturally occurring carbonates. The non-volatility of the acid effected a simplification in the apparatus, since pumice impregnated with dry copper sulphate was not required as was the case when hydrochloric acid was used to decompose the carbonate. A mixture of

phosphoric and chromic acids enabled one to estimate in a similar way the carbon present in organic compounds, even when these were very resistant to oxidation, and the process could be used for carbon in steel (Chem. Soc. Trans., 1904, 85, 1004).

Dr. E. W. SMITH said that in testing for carbon dioxide in the atmosphere he had found that where great accuracy was required it was necessary to use bottles coated with paraffin wax. Did the author consider that the precautions of the Pettenkofer method were worthy of consideration for his purpose?

Dr. SLATOR said, in reply, that he had not tested other oxidation reactions, but they could doubtless be carried out in the apparatus, and the resultant carbon dioxide estimated. Care must be taken not to employ reagents which produce free oxygen. No difficulties such as occurred in estimating carbon dioxide by Pettenkofer's method were met with in these experiments.

Yorkshire Section.

MR. W. MCD. MACKEY IN THE CHAIR.

SOME OBSERVATIONS ON THE "SPRINGING" OF "CARBOLATE" AND THE RECOVERY OF PHENOL FROM THE RESULTING AQUEOUS LIQUOR.

BY H. M. DAWSON.

In the "springing" of "carbolate" by the addition of sulphuric acid a considerable proportion of the phenol remains in the resulting aqueous solution of sodium sulphate which is run off to the drain. The proportion of carbofic acid which is thereby lost depends largely on the conditions under which the "carbolate" is "sprung." The percentage loss is mainly determined by the volume of the aqueous liquor, by the concentration of the sodium sulphate in this solution, and by the temperature at which the separation of the phenol layer takes place. At a given temperature the percentage loss of phenol increases with the volume of the aqueous solution, but more rapidly than this, in that the resulting diminution of the concentration of the sodium sulphate lowers the "salting out" effect of the electrolyte on the phenol. When the volume of the aqueous solution and the concentration of the sodium sulphate are fixed, the percentage loss of phenol increases with rise of temperature.

The influence of the sodium sulphate concentration (C_2) on the phenol content of the aqueous liquor (C_1) is shown by the following numbers obtained from experiments made at 25° C. The concentrations are expressed in grams per litre, and in the third column are given the phenol concentrations calculated from the empirical equation—

$$\log C_1 = 1.92 - 0.0036 C_2 \dots (1).$$

Relation between phenol and sodium sulphate concentrations.

Sodium sulphate.	Phenol concentration.	
	Observed	Calculated
0	85.5	83.2
50	53.0	55.0
100	36.2	36.3
150	24.0	24.0
200	16.0	15.8
250	11.5	10.5

From the above relation between the phenol and sulphate concentrations it is possible to calculate

the percentage loss of phenol at 25° under any specified conditions of the springing process. Assuming that the quantities of phenol, sodium hydroxide, and sulphuric acid are equivalent, so that the "springing" is quantitatively represented by the equation



the percentage loss may be derived as follows:—Let W be the total quantity of phenol in the carbolate, C_1 the concentration of phenol, and C_2 the concentration of sodium sulphate in the resulting aqueous solution in grms. per litre. Then $W/142/188$ is the quantity of sodium sulphate produced, $142W/188C_2$ is the volume of the aqueous solution in litres, $142WC_1/188C_2$ is the quantity of phenol in the aqueous solution, and p , the percentage loss of phenol, is given by

$$p = 142WC_1/188C_2 \times 100/W = 75.5 C_1/C_2 \dots (2).$$

Since C_1 is a function of C_2 , the percentage loss of phenol can be expressed in terms of the sodium sulphate concentration. Substituting for C_1 the value given by equation (1), we obtain for p under the specified conditions:—

$$p = 75.5 (10^{1.92 - 0.0036C_2})/C_2 \dots \dots (3)$$

For the range of sodium sulphate concentrations which may be said to be of practical significance the percentage losses of phenol are shown in the following table, the numbers under p being derived from equation (2), and those under p_1 from equation (3).

Sodium sulphate concentration.	p	p_1
100 grms. per litre	27.3%	27.4%
125 " "	18.1%	17.8%
150 " "	12.1%	12.1%
175 " "	8.5%	8.4%
200 " "	6.0%	6.0%

In practice the conditions of the "springing" process vary considerably, and there is a correspondingly wide variation in the percentage loss of phenol. Assuming that the "carbolate" is obtained by the use of a 10% solution of caustic soda, that the dissolved phenol is equivalent to the sodium hydroxide in solution, and that the solution of the phenate is sprung by the addition of 65% H_2SO_4 (110° Tw.), the loss of phenol is about 10%. A laboratory experiment carried out under these conditions at 25° C. gave a loss of 9.5%.

It seems probable that the loss under actual working conditions may sometimes be appreciably greater than 10%, for the solution obtained under the above conditions contains sodium sulphate in such quantity that it deposits Glauber's salt at temperatures not much below 25° C., and this deposition is, of course, to be avoided in practice.

The loss of this phenol was at one period during the war a matter of considerable importance, and, at the suggestion of the Explosives Supply Department of the Ministry of Munitions, experiments were made with a view to the recovery of the phenol from the waste sodium sulphate liquors.

Preliminary observations, in which methods depending on adsorption of the phenol by solid substances and on extraction by liquid solvents were tried, led to the conclusion that the removal of the phenol from the aqueous liquors was more likely to be achieved by the use of liquid extraction media. In such extraction processes the efficiency is determined by the distribution ratio, i.e., by the ratio of the concentrations of the phenol in the extracting liquid and in the aqueous solution when the two media are in equilibrium. Apart from the magnitude of this ratio the extracting liquid should be readily available, should be only slightly miscible with water, and should be such that it can be easily separated from the phenol, which, by its use, has been extracted from the waste liquors.

The literature affords but little information as to the distribution of phenol between organic solvents and water, although the behaviour towards benzene and water has been examined in some detail. For dilute solutions, not exceeding, in the case of the aqueous layer, a concentration of about one gram per litre, Philip and Bramley (J. Chem. Soc., 1915, 107, 377) have shown that the ratio (r) of the concentration in the benzene to that in the water layer is independent of the absolute concentration. These experiments give $r=2.18$ at 25° . At higher concentrations experiments of Rothmund and Wilsmore (Z. physikal. Chem., 1902, 40, 611) show that r increases at 25° C. from 2.28 to 12.24 when the concentration of the phenol in the water layer is increased from 2.5 to 50 g. per litre. These results indicate that the distribution is very largely dependent on the concentration, unless the quantity of phenol in the aqueous layer is small in comparison with that present in a solution saturated with phenol at the same temperature.

In the experiments described in this paper no attempt has been made to investigate in any detailed manner the distribution of phenol between water and the various extraction media. The requisite information has been obtained by a direct determination of the quantity of phenol extracted by various solvents from an aqueous solution containing 10 g. of phenol and 200 g. of sodium sulphate per litre. In the experiments 50 g. of the extraction solvent was shaken at 25° C. with a litre of this solution, and after the non-aqueous layer had completely separated, it was dried, weighed, and the proportion of phenol in it estimated by appropriate methods.

The liquids used as extraction solvents are indicated in the first column of the following table. The "petrol" was a hydrocarbon fraction boiling at 95° – 100° C.; the solvent naphtha boiled between 145° – 155° C., and the "meta-cresol" was a mixture of *m*-cresol and *p*-cresol containing about 75% of *m*-cresol. The second column gives the quantity of phenol extracted from the aqueous solution by the various solvents, and the sixth gives the percentage of phenol recovered. In the third and fourth columns are shown the concentrations of the phenol, in grams per litre, in the non-aqueous and aqueous solutions respectively, and in the fifth is given the distribution ratio which is derived from these numbers:—

Extraction of phenol from aqueous solutions.

(1)	(2)	(3)	(4)	(5)	(6)
"Petrol" ..	0.47g ..	6.85 ..	9.53 ..	0.72 ..	4.7%
Solvent naphtha ..	3.99g ..	64.6 ..	6.01 ..	10.7 ..	40%
Benzene ..	4.13g ..	67.2 ..	5.87 ..	11.4 ..	41%
Nitrobenzene ..	6.49g ..	138.0 ..	3.51 ..	39.3 ..	65%
Ortho-cresol ..	7.49g ..	134 ..	2.51 ..	53.5 ..	75%
"Meta-cresol" ..	7.47g ..	130.5 ..	2.53 ..	51.5 ..	75%

In reference to the foregoing table it may be pointed out that nitrobenzene was included amongst the extraction solvents submitted to investigation because a knowledge of the influence of the nitro-group on the miscibility of phenol with the corresponding aromatic hydrocarbon was required in another connexion, and not because it could be regarded as a suitable extraction solvent even if the distribution ratio were extremely favourable.

The results obtained with the various solvents show that the cresols are the most effective in removing phenol from its aqueous solutions. The aromatic hydrocarbons are much less efficient, and the paraffin hydrocarbons, as represented by "petrol," are obviously quite unsuitable for use in the recovery of phenol. The relatively high recovery values obtained for the cresols are quite in keeping with the fact that miscibility is generally favoured by similarity in chemical nature. The closeness of the results obtained with *o*-cresol and

"meta-cresol" indicates that the solvent power of the cresols for phenol is not sensibly affected by the relative positions of the CH_3 and OH groups in the molecule.

From the experimental results obtained it may be inferred that a "cresylic acid" of relatively high boiling point could be used advantageously for the removal of phenol from its aqueous solutions. The aqueous liquors need only be agitated with about 5% by weight (or volume) of the "cresylic acid," and if the stripping is carried on in such a way that partially stripped liquors are brought into contact with fresh "cresylic" whilst the original aqueous liquors are agitated with "cresylic" which has already been used, a recovery of 90–95% of the phenol can readily be obtained. From the resulting mixtures of phenol and cresols the phenol can be removed in the usual manner.

DISCUSSION.

Mr. C. P. FINN pointed out that in tar-works practice the separation of phenol from the aqueous liquor tended to occur automatically in that the "carbulates" contained considerable proportions of cresols. He asked whether the author could suggest a suitable treatment for water which separates from cresosote and contains traces of phenolic substances.

Dr. DAWSON agreed that in the "springing" of "cresolates" containing phenolate the phenol would tend to be removed from the aqueous layer in virtue of the relatively high solubility of phenol in the cresols. The "carbulates" which the author had more particularly in mind were, however, those obtained in the production of synthetic phenol. The experiments which had been made with various solid adsorbents had not brought to light any material suitable for continued use in the removal of traces of phenolic substances from aqueous solutions.

Communication.

DETECTION OF MINUTE QUANTITIES OF PETROLEUM SPIRIT IN VEGETABLE OILS.

BY MASAHIRO AIDA.

A number of methods for the detection of petroleum spirit in vegetable oils have been proposed. A careful study of these has shown that they are not satisfactory, but it has been found that Nastjukoff's formolite reaction may be applied successfully for the purpose as follows:—

The oil (50–100 g.) is saponified by means of potassium hydroxide solution. Distilled water and pure calcium chloride solution are added, the liquid distilled by means of steam, and the distillate treated with 40% formaldehyde solution and a few drops of concentrated sulphuric acid. A reddish-brown film coloration on the surface of the liquid, gradually changing to deep yellow, indicates presence of petroleum spirit. If a few drops of the distillate are added to water, a brilliant interference ring of optical waves is produced on the surface of the water; this ring becomes almost invisible after standing for some time and disappears completely on heating. With soya bean oil the ring does not change, even on heating. The above process is capable of detecting traces of petroleum spirit in vegetable oils, and may be made the basis of a quantitative method, the formolite precipitate being weighed after drying at 110° – 115° C.

Birmingham Section.

Meeting held at Birmingham University on
Thursday, March 11, 1920.

MR. L. P. WILSON IN THE CHAIR.

CATALYSIS APPLIED TO THE OXIDATION OF OILS.

BY R. S. MORRELL, M.A., PH.D., F.I.C.

In the linoleum, paint, and varnish industries the problems of the oxidation of oils are of the highest importance. The acceleration of the setting and drying times of oils introduces the study of the action of siccatives.

The vegetable oils used in the industries are glycerides of unsaturated aliphatic acids containing not less than 18 carbon atoms in open chain; the oil from chaulmoogra seeds contains closed ring substances (Barrowcliff and Power, *J. Chem. Soc.*, 1907, 91, 577). The drying oils of animal origin, menhaden and Japanese fish oil, contain the glyceride of clupanodonic acid. Before discussing the catalytic oxidation of oils our knowledge of their complex chemical constitution and the great variety of changes which take place during oxidation will check the tendency to indulge in speculation or to generalise from insufficient data.

This polymerisation is slower than the oxidation. Wolff (*Farben-Zeit.*, 1919, 24, 1119) maintains that in the case of oil varnish on exposure to light of short wave-length oxidation and polymerisation proceed at nearly equal rates, whereas in light of long wave-lengths polymerisation is retarded more than oxidation, with the result that inequality of outer and inner layers occurs, with the production of shrivelled films. Polymerisation is of great importance in consideration of the durability and water-resisting power of oil films. The following table shows the percentage of linoxyn obtained from several forms of drying oils, from which it is evident that the linoxyn produced from a thickened oil is greater in amount and more water-resisting than that obtained from ordinary linseed oil. There is evidence of slight difference between the action of lead and manganese as driers.

With regard to the subsequent changes in the linoxyn there is no consensus of agreement. The possibilities are so many, comprising, in addition to peroxides, acids, aldehyde-acids, substances with definite acetyl value, and volatile products. (See Fahrion, *Chem.-Zeit.*, 1904, 1196; Orloff, *J. Russ. Phys.-Chem. Soc.*, 1910, 42, 658; Fokin, *Z. angew. Chem.*, 1906, 1219; Ingle, *J.*, 1913, 32; 1919, 101; Salway, *J. Chem. Soc.*, 1916, 169, 138; Wilson and Heaven, *J.*, 1912, 565; Waele, *loc. cit.*; Reid, *J.*, 1894, 1020.)

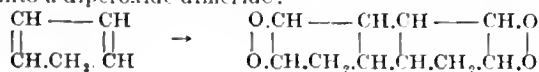
Genthe (*Z. angew. Chem.*, 1906, 19, 2087), in his investigation of the oxidation of linseed oil in

Water absorption of drying oil films after treatment with petroleum spirit and methylated ether.

				Time of drying in days.	Petroleum. 3 Days	Petroleum. 6 Days	34 Days in meth. ether	% Linoxyn.	Water immersion, % gain.			
									1 Day	2 Days	4 Days	8 Days
Linseed oil		169	loss		% loss	60.9	23.8	29.6	33.5	52.9
Pb & Mn present			3.8%	Unchanged	35.3		film white	film white	film white	film white
"	"	"	"	39	—	—	60.5	39.5	—	3 Days		
							62.4	37.6	—	21.6	film white	
										32.5		
Lithographic oil		39	—	—	40.7	59.3	—	3.4		
Pb & Mn present							—	—	film white	
Lithographic oil		39	—	—	46.4	53.5	—	15.2		
Mn present								milky		
Linseed oil		12 hrs.	—	—	68.8	31.2	18.0	5 Days		
Pb & Mn present							cloudy	23		

From the general properties of the glycerides it is to be expected that oxidation will proceed in stages whereby a molecule of oxygen becomes attached at a double linkage; such gradual absorption has been observed in the oxidation of China wood oil (Morrell, *J. Chem. Soc.*, 1918, 113, 111).

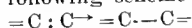
When linseed oil is transformed into the highly-oxidised state of linoleum the glyceride of diperoxylinolenic acid is found to be present (Waele, *J. Ind. Eng. Chem.*, 1917, 9, 1), and in linoxyn there are still unsaturated linkages. Ingle's formula for linolenin (the triperoxylinolenic acid glyceride) does not appear to be realised in practice, although it may represent the autocatalyst formed in the oxidation of linseed oil without the use of driers. Orloff's formula for linolenin (*J. Russ. Phys.-Chem. Soc.*, 1910, 42, 658), representing an oxido and peroxide is possible, but difficult to verify. The author favours a subsequent or, under certain circumstances, a simultaneous polymerisation of the peroxides in view of the results obtained from the study of the cerium salt of tung oil (*loc. cit.*), and from the recent work of Stobbe and Dunnhaupt (*Ber.*, 1919, 52, 1436) on cyclopentadiene, which is transformed on oxidation into a diperoxide dimeride:



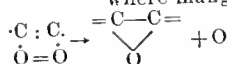
ultra-violet light with and without driers, obtained in the latter case a sinuous form of curve expressing the relationship between the amounts of oxygen absorbed and the time. This form is characteristic of an autocatalytic change (Kullgren, *Z. physik. Chem.*, 1912, 41, 405) and can be expressed by the Ostwald equation for the intervention of an autocatalyst, viz., $\frac{dx}{dt} = k(a-x)(b+x)$, where a is the initial concentration of the oil, b that of the autocatalyst, and x the amount of oil oxidised. It is possible that the autocatalyst is a higher peroxide than that found in linoxyn.

The presence of the siccativ shortens the induction period (*cf.* Rideal, "Catalysis in Theory and Practice," 1919, p. 149). From a comparison of series of metallic driers as arranged by Fokin (*Seifens. Zeit.*, 34, 821) and by Mackey and Ingle (*J.*, 1917, 317), it might appear that the metals which are capable of forming series of oxides prove themselves the best siccatives. Fokin states that polymerisation proceeds parallel with oxidation, and between 0.2 and 20 atm. the oxygen absorption is proportional to the pressure. The velocity is stated to increase with the cube root of the concentration of the catalyst, but it seems not strictly accurate to develop a relationship between the reaction velocity and the concentration of the catalyst added; the superficial concentration ought to be substituted. The metal content of varnishes

stands in no stoichiometric proportion to the mass of the oxidation product, and, according to Weger (Chem. Rev., 1898, 5, 4) the drying power of manganese increases up to 0.2%, further increase in concentration causing no acceleration in drying (cf. Bach, Oxidation of hydriodic acid by hydrogen peroxide in the presence of peroxydase; Ber., 1904, 37, 3798). Lippert (Z. angew. Chem., 1898, 11, 433) states that the combined oxygen is increased by the presence of small quantities of manganese driers, but with increasing amount of drier up to 0.2% the oxygen number falls. It is stated that without driers peroxides tend to be formed, but in the presence of driers atomic oxygen is absorbed. There would appear to be an advantage in the use of mixtures of lead and manganese in the case of the oxidation of linseed oil, and the best proportions are stated to be 5Pb:1Mn up to 0.6%. Ingle (J., 1919, 101 r) states that manganese driers catalyse the transformation of linseed oil into linoxyn four times as rapidly as lead driers. Rochs (Z. angew. Chem., 1911, 34, 80) seeks to explain the difference by the following scheme:—



where manganese acts as a drier, and

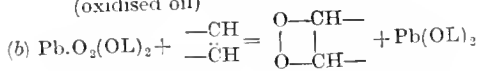
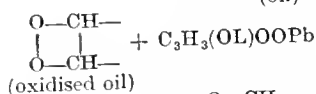
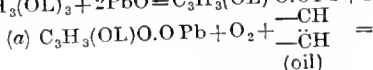
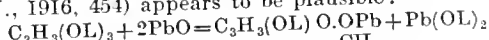


where lead acts as a drier. When there is a combination of the two driers a peroxide-glyceride of the Orloff type referred to above would be obtained. Another view is that the second drier acts as a polymerising agent, causing a combination of unsaturated molecules and preventing the oxidation being carried too far, as in the case of Reid's superoxidised oil. The author considers this view worthy of consideration, especially as aluminium as a drier has been found to give better results than lead (Gardner, Circ. 60, Paint and Var. Manufs. Assoc.); moreover, the advantages of lead as a drier are not so manifest when dealing with polymerised or easily polymerised oils.

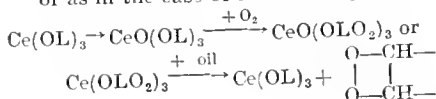
There is no doubt that it is advisable to check excessive formation of peroxide-glycerides, which are overprone to subsequent decomposition, and the combination of metallic glycerides in this respect may be favourable. Excess of a manganese drier in linseed oil mixings causes surface drying with rivelling. It is sometimes stated that a drier may after a time lose its activity. Undoubtedly driers often cause an increase in viscosity of mixings whereby the setting time is retarded, but there is no definite evidence in the case of varnishes provided the mixing remains clear and there is no precipitation.

The theory of the action of siccatives is not as yet satisfactory. There has been much speculation following on the lines of consideration of homogeneous systems, whereas the drying of oils belongs to the class of heterogeneous systems.

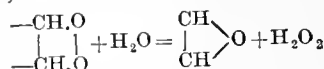
If the theory of intermediate compounds (Engler's) be applied the scheme suggested by Ingle (J., 1916, 454) appears to be plausible:—



or as in the case of cerium tungstate



Lead and cerium here act as direct catalysts to form linoxyn. In autocatalysis without drier it must be assumed that a higher peroxide is formed which is subsequently decomposed (Jorissen, Chem.-Zeit., 1898, 23, 162).



In the presence of water the production of hydrogen peroxide would be accounted for by the formation of Orloff's oxide peroxide glyceride. The influence of moisture on the oxidation of linseed oil and polymerised linseed oil in the presence of driers is discussed by Waele (J., 1920, 49 r), who notes the stability of the peroxides of polymerised linseed oil. He considers that the decomposition of the primarily formed peroxides is initiated by the presence of water and equilibrium conditions, i.e., decomposition *v.* back pressure, are attained by the interpretation of the latter as pressure of water vapour. Gardner (Circular No. 60) states that an atmosphere having a relative humidity of 20°–30° C. gives the best drying conditions. Assuming the intermediate formation of a higher peroxide acting as an intermediate compound (Genthe found that in ultra-violet light as much as 34% oxygen could be absorbed), then Liveing's explanation of the decomposition of hydrogen peroxide in the presence of silver oxide may be applied ("Chemical Equilibrium the Result of the Dissipation of Energy," 1885). It is known that when linseed oil has been boiled without driers it is oxidised very slowly, but its activity is regained when air is blown in. The marked changes of colour in solutions of manganese, cobalt, and cerium driers all indicate a higher degree of oxidation of the metal, but there would seem to be an equilibrium between the state of oxidation of the metal and peroxide of the oil which is only disturbed on exposure to air. It has been observed by the author that cerium varnishes after a time become yellow out of contact with air, marking the presence of ceric salts, and yet there is no loss on drying on exposure to air. Manganese and cobalt liquid driers, which are at first comparatively pale in colour, become permanently dark on being kept even out of contact with the air.

For heterogeneous systems the adsorption view of Faraday (1833) as to catalytic activity being due to adsorption phenomena is most worthy of consideration. In a homogeneous system the reaction is proportional to the concentration of the catalyst, but in a heterogeneous system the reaction velocity will be influenced by the active volume of the catalytic material; moreover the reaction velocity will be governed by the rate of diffusion of the reactants to the surface or the products from the surface as well as by the rate of superficial action; for the mass concentration must be substituted the superficial concentration. Very little is known of the properties of the surfaces of drying oils. Do the metallic driers modify the superficial properties of the drying oils to different degrees according to their drying properties? From the author's observations the interfacial tensions of equivalent strengths of manganese and cobalt linoleates are different from that of lead linoleate dissolved in linseed oil (the strengths of the solutions were 1.3, 1.4, and 4.96% respectively). The interfacial tension of the lead solution to air or nitrogen was lower than that of linseed oil, but manganese and cobalt solutions have a slightly higher surface tension than linseed oil. With solutions of 1/10 concentration the differences are less marked; the diminution in concentration of the manganese and cobalt salts has very little effect on the values of the surface tension, whereas the reduction in the surface tension of lead linoleate is more marked with variation in the concentration. The surface tensions were determined by Morgan's drop method

(J. Amer. Chem. Soc., 1911, 33, 648), and details of the investigation, which is still in hand, will be published later. The drop numbers of the respective oils into water or of water into the oils have been determined, and it was found that the interfacial tension of the lead solution was much greater than that of linseed oil; manganese oil was slightly greater and cobalt oil was less than linseed oil, but the unavoidable presence of alkali in the salt is accountable for the lowering of the interfacial tension. The direct influence of the concentration of the lead salt was again noticeable, likewise the non-influence in the case of manganese (cobalt for the above reason must not be considered without further investigation).^{*} The presence of alkali in the case of the lead salt was proved to be unlikely, because a lead drying oil made without the use of alkali was found to give results of the same order. The above results throw some light on the peculiar behaviour of lead and manganese driers. Lippert (*loc. cit.*) states that above a concentration of 0.2% manganese the oxygen absorbed is reduced with increasing manganese content; the drying power of lead, on the other hand, increases with increasing content. The application of Gibbs' law from the results of the surface tension determinations would favour increasing surface concentration for lead and a slight alteration in surface concentration of manganese or cobalt. The behaviour of a surface containing a lead drying oil to water is well known, and is in keeping with the drop numbers into water. From the brief statement of the results of a preliminary and incomplete investigation, it is evident that the chemical properties of the metals used as driers will not account for their behaviour, and the study of the differences of surface properties is of importance. The diffusion rate of the products of oxidation and polymerisation or of the reactants depends on the viscosity of the layer, so that it is difficult to make a strict comparison of catalysts.

Driers might be considered as promoters adsorbing oxygen selectively at the surface followed by chemical combination.

On an hypothesis of directive and selective adsorption, connexion is made between the intermediate compound theory and the adsorption theory, because the idea of a directive force assumes chemical union between the surface of contact and the molecules of the surrounding medium. The distinctive character of the interfacial molecular condition has been put forward by Langmuir (J. Amer. Chem. Soc., 1918, 40, 1361), who assumes that the solid catalyst becomes coated with a single layer of molecules by adsorption; such condensation is accompanied by a change in the character of the adsorbed molecule involving a dissociation of elementary molecules (*e.g.*, oxygen into atoms), by which the atoms of the condensed substances are definitely associated with certain molecules in the surface layer. The application of Langmuir's theory by Lewis (J. Chem. Soc., 1919, 115, 1821) in terms of the radiation hypothesis involves the consideration of the condensation on the surface of the catalyst in the atomic form.

It is evident that more attention must be paid to the study of the catalytic oxidation of oils from the conditions at the interfacial surface, whereby connexion may be established between the superficial forces and those manifested in the chemical changes which are known to occur during the oxidation and setting of oils.

DISCUSSION.

The CHAIRMAN suggested that the method proposed by Mr. Heaven and himself (J., 1912, 565) might be helpful in regard to the elucidation of the formulæ of Rochs and Ingle; and further, with the reduction of the film to its smallest possible

dimensions, by that method of impregnating kieselguhr possibly some information on the nature of the oxidation by the catalyst might be obtained. The order of activity of the catalysts was very interesting. He had believed that the activity depended upon the fact that they existed in two or more oxide or hydrated forms, and that the activity depended upon the rate of change from one form to the other, this being the rate at which oxygen was transferred. He was doubtful as to the catalytic effect of sodium and calcium.

Dr. E. B. MAXTED said that the surface of separation between the catalyst and the substances which were undergoing reaction on its surface might be regarded from a kinetic standpoint. He thought considerable progress might be made in heterogeneous catalysis generally by considering this surface layer as a zone in which the various possible intermediate compounds and final products were all capable of existence side by side, and in which the actual equilibrium state would be such that there was a definite proportion between these possible intermediate compounds in a kinetic sense only, namely that, while the actual number of molecules of each species in equilibrium remained constant, the individuality of these molecules constantly changed with change in time. It was quite possible to see how cobalt etc. could help the absorption of oxygen by oil, by reason of the fact that among the various possible compounds formed on the surface of separation there would be peroxides in which the oxygen was at a potential sufficient to be transferred to the oil. One other interesting point was the possibility of inhibiting the absorption of oxygen by means of catalyst poisons.

Mr. F. H. ALCOCK asked if the process of oxidation in oils would determine the question of purity and impurity in linseed oil.

Dr. MORRELL, in reply, agreed as to the necessity of considering the question of the oxidation of oil from Dr. Maxted's standpoint, and that a careful examination of what was taking place on the surface was necessary. He had no knowledge of any catalytic poisons in the oxidation. It was stated that certain driers lost their power. In certain cases it had been shown to have been due to the oil or the varnish mixing. There might be deposition of the drier from the solution—such cases were known. The purity of the oil could be decided by the drying oil method. The iodine value determination was always supplemented by a drying oil test.

THE ACCELERATION OF VULCANISATION.

BY D. F. TWISS AND S. A. BRAZIER.

This paper, read by Dr. Twiss, has already been published in the Journal for May 15, p. 125 T.

DISCUSSION.

The CHAIRMAN asked if it was not possible that a certain proportion of the apparent retarding effect of zinc oxide was due to a reaction between the zinc and some of the sulphur at the temperature of vulcanising, which might not occur in the presence of thiocarbanilide?

Dr. SLATER PRICE said that he could not recall having seen any equations for the velocity of a reaction which was auto-catalysed which led to a straight-line curve. It would appear that some other explanations might be given. Straight-line curves were found in various fermentation reactions, for example, in sugar fermentation, and they had been explained by a combination of the sugar and the enzyme, the concentration of this combination remaining practically constant during the reaction.

Dr. MAXTED suggested that if vulcanisation took place between a certain amount of sulphur dissolved in, or associated in some way with the catalyst, it was possible that that might maintain a

^{*} The densities and viscosities of the solutions were determined under the same conditions of temperature.

constant concentration right down to the end of the reaction. Had experiments been made as to the relative vulcanising power of the various solid allotropic forms of sulphur?

The CHAIRMAN pointed out the close parallel in respect of the vulcanisation of rubber and the oxidation of oils. In addition to the combination with sulphur, was not a change in the rubber possible in the direction of polymerisation? In the oxidation of drying oils there was a strong indication of polymerisation occurring at the same time. The degree of polymerisation accompanying combination of the sulphur might probably account for many of the peculiar physical properties of the vulcanised rubber.

Dr. Twiss, in reply, said that he thought the suggestion that the retarding action of zinc oxide might be due to this substance competing with the rubber for the sulphur was improbable. The extent of the combination of zinc oxide and sulphur under the conditions of vulcanisation was not very considerable and as there was only 1% of zinc oxide present in the mixture in question, the actual reduction in the quantity of sulphur through this cause must have been very small. He agreed that zinc oxide itself might exert a very feeble catalytic effect, but the results showed that this substance should then be regarded as a negative rather than as a positive catalyst. As to the suggestion of the intermediate formation of some sulphur compound during the vulcanisation process, analogous to the combination of enzyme and sugar in fermentation, he pointed out that there was evidence of the occurrence of auto-catalysis; this was to be attributed to the influence of some catalytically active body produced by combination of sulphur with some other substance, possibly with one of the natural impurities of the rubber or even part of the rubber itself. Some years ago he had published figures showing that insoluble sulphur had approximately the same vulcanising power as crystalline sulphur. This might be expected in view of the recent evidence of Beckmann that S_8 when heated above the melting point of ordinary sulphur very rapidly became converted into an equilibrium mixture of the same composition as that yielded by S_8 . During the vulcanisation process, therefore, pure sulphur, whatever its original form, would in all probability, be present as the equilibrium mixture corresponding with the temperature. Apart from this, however, the fact that the temperature coefficient was approximately the same at different temperatures indicated that the various allotropic forms present in molten sulphur did not differ greatly in their chemical activity towards rubber. As to the polymerisation of rubber, it was evident that during vulcanisation two processes took place—a chemical process and a physical change induced by the former. The two were not, however, entirely proportional to one another. It had even been suggested that vulcanisation was only a physical process, that the sulphur was practically a catalyst, and that the effect was merely a polymeric change in the rubber itself, but general opinion was contrary to such a view.

other metallic element, and yet, how little we understand concerning the nature of the action of light upon them!

If, as seems most probable, a number of the elements are capable of forming compounds as sensitive to other forms of energy as those of silver are to light, then truly we are but at the beginning of an era of progress which must inevitably lead to undreamt-of additions to our methods of intercommunication and record by means of that wonderful interplay of natural forces of which present-day photography is but one expression.

I do not propose to deal very fully with that part of Hurter's researches for which he and his colleague, V. C. Driffield, are perhaps chiefly remembered by photographers; the avowed primary object of their work was the very practical one of discovering a scientific method of speed determination which would lead to the elimination of waste by incorrect exposure on the part of the user and to scientific control of the industrial preparation of dry plates. This purpose they undoubtedly achieved in so far as any one numerical value can express the light-sensitiveness of a material which is capable of such wide variations of character.

The mere invention of a practical method of determining and expressing the light-sensitiveness of a plate would, however, never have given Dr. Hurter that niche in the Temple of Fame which he justly occupies if it had not been a system which contained within itself the capacity of adaptation to the needs of later workers and different circumstances and had it not been built on a sound scientific foundation.

It is true that the serious, if not insurmountable, difficulties of standardising the conditions of speed testing throughout the industry, the comparatively wide variations in the shape of characteristic curves of commercially useful dry plates, and the proved unsuitability of the true H and D speed number alone as a guide to exposure in certain important branches of photography (not to mention the baneful influence of commercial advertising), have all contributed to cause published speed numbers to be regarded with considerable distrust. Nevertheless it is literally true to say that the Hurter and Driffield characteristic curve is, and will doubtless remain, the most compact and lucid means of expressing quantitatively the qualities of any and every light-sensitive material.

Prior to the work of Dr. Hurter and his colleague it was fairly generally believed that a gelatin dry plate resembled the wet collodion plate in being capable of development to any desired extent if plied with fresh developer, subject only to the practical limitation that general fog supervenes after a more or less short time. Indeed, this view was prevalent for many years, and is probably not yet entirely extinct. Hurter and Driffield showed conclusively, however, that such is not the case, but that for any given plate the limit of possible development is set by the amount of change wrought by the exposure.²

We know now that the reason for this lies in the fact that the silver halide grain behaves as a self-contained unit. These units are by no means all alike³; those grains which have been sufficiently exposed are reducible, while the less exposed ones, which remain unreduced, apparently play little or no part in the normal development process.⁴ Further, we know that the silver halide grains in commercial dry plates vary both in size and in light-sensitiveness.

It is, however, highly desirable that a thorough study should be made of the behaviour of the individual grains in the exposure and development processes to determine:—

(a) The minimum exposures required to make them developable.

(b) The range of sensitiveness to be found among the grains in a few typical emulsions.

Liverpool Section.

Meeting held on May 27, 1920.

DR. E. F. ARMSTRONG IN THE CHAIR.

PHOTOGRAPHIC IMAGES—VISIBLE AND INVISIBLE.

BY F. F. RENWICK, A.C.O.I., F.I.C.

(HURTER MEMORIAL LECTURE.)

The chemistry of silver and its salts has probably been more closely studied than that of almost any

(c) The connexion, if any, between size of grain and light-sensitiveness.

(d) Whether, as seems probable, the rate of reduction of a given quality of grain with a given developer increases to a maximum at a certain exposure before it declines again almost to zero.

Naturally such a research is beset with numerous difficulties, but Kinoshita's⁴ work under Rutherford, at Manchester, on the behaviour of photographic plates to alpha particles shows that they are not insuperable.

At present we merely know from a comparative study of the characteristic curves of different emulsions that the position and shape of the curve of any plate is influenced by the range of sizes and sensitiveness of the grains and their relative proportions⁵ in the emulsion, the extent to which development is carried, as determined by the developing solution employed, its temperature and time of action, and its rate of absorption by and diffusion in and out of the gelatin film⁶; and, lastly, there enter into the final result the extremely complicated optical problems which arise when dealing with the penetration of light through turbid media.⁷

Hurter and Driffeld,⁸ Schaum, and many other investigators have assumed that the amount of developed silver in a negative is a reliable index of the amount of photo-product formed during exposure, but that this is not at all accurately the case has been known for some time, and is strikingly brought out by some experiments carried out last year at the Ilford Laboratories.

A gelatin emulsion in the liquid state was divided into two parts, and one of them was fogged by light while it was thoroughly stirred. Mixtures in known proportions of the light-fogged and of the clean unfogged emulsion were then made and coated equally heavily on a number of test plates. In this way we were able to secure plates having known relative amounts of latent image per unit area. After careful drying, plates of each mixture were developed together, fixed, and dried and their densities measured. Fig. 1 shows the results in graphic form of a typical experiment. Abscissæ represent the relative proportions of fogged emulsion (=latent image \times constant) actually present, the ordinates give the densities developed in various times. If it was not so before, it will already be

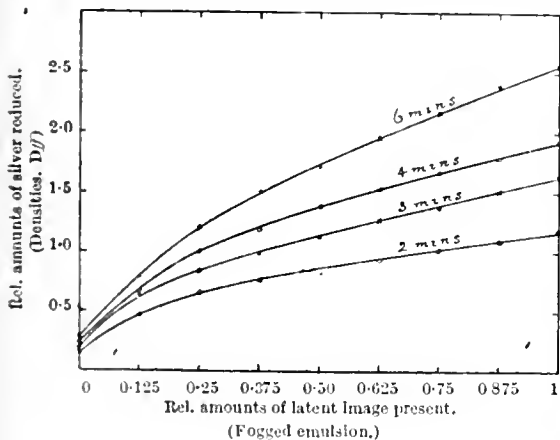


Fig. 1.

evident how complex are the factors which contribute to determine the qualities of a photographic dry plate; fortunately, it is rapidly becoming more clearly recognised that new methods of attack must be devised to enable us to separate one influence from another and so arrive at fundamental truths.

In connexion with the developed plate there is another interesting and important problem—

namely, that of tracing the connexion between the size of the emulsion grain and that of the developed silver grain. It is well known that the latter are, on the average, larger, more irregular in outline than, and somewhat differently distributed from, the original emulsion grains. While emulsions of large-sized grains always give coarser-grained negatives than fine-grained emulsions, there is very little likeness in size or shape between them in the dry negative. Lüppo-Cramer⁹ has, however, shown that the silver deposited in the process of development is in the semi-fluid condition of a colloidal silver-gel, and, as he suggests, it is therefore probable that the cavities and channels in the spongy matrix (caused by solution of the haloid during development and fixation) help to determine the forms of the silver grains and their distribution in the finished negative.

From this brief survey of some of the factors influencing the shape of the Hurter and Driffeld characteristic curve we may turn to a consideration of some of the more chemical aspects of the subject.

Dr. Hurter's consideration¹⁰ of the properties of the latent image led him to believe that the first action of light on silver bromide induced only a physical change and not the formation of a new chemical product. It is interesting to note that in English-speaking countries those aspects of the matter which weighed most strongly with Hurter have usually been prominently borne in mind, while continental photo-chemists have tended in many cases to ignore these difficulties and to seek an explanation of a purely chemical character.

It would take far too long, nor would it be entirely profitable, to examine critically the many explanations of photographic action which have from time to time been offered.¹¹

They divide themselves into two broad classes. Firstly, those which seek to explain all photographic action as chemical change in which one or more new products are formed by the direct action of light. Secondly, those which maintain that, although when *visible* photographic images are formed, chemical change must be admitted, this is not the case in the earlier stages where a development process is essential to make the photographic action apparent.

This division of opinion dates from the early days of photography and still persists as strongly as ever. Meldola credits Fischer with having first suggested a subchloride theory as early as 1814, while direct reduction to metallic silver was offered as the explanation by Arago as early as 1839. On the other hand, Moser, in 1842, and Claudet, in 1848, both held strongly that the latent image was the result of a purely physical change. Subsalt theories were, or are still, advocated by Eder, Schaum, Trivelli, Vogel, Carey Lea, Abney, and many others; reduction to silver is the theory favoured by R. Abegg, R. Luther, W. Reinders, R. Lorenz, R. Zsigmondy, E. Baur, and Lüppo-Cramer.

The only physical change theory which now has many supporters concerns itself with changes in electrical condition brought about by the action of light on the silver halides, though many other more or less vague kinds of physical change have been advanced as explanations in the past and are still advocated by a few.¹²

Electrical theories¹³ of the latent image are relatively new and naturally are fashionable in these days of electrons and photo-electricity. It remains to be seen, however, whether they will prove more helpful in advancing photography than other physical change hypotheses have been. Even if the two chief chemical theories were finally disproved it would still have to be admitted that both have borne valuable fruit by giving rise to much experimental research of the highest value.

The point at issue really lies in the answer to the question: Does the smallest demonstrable light

effect coincide with the formation of a new chemical substance, and, if so, what is its nature?

It will be evident that ultimately everything turns on our interpretation of experiments concerning the persistence or destructibility of the *developable condition*, since this is our only evidence for the existence of something which we call a latent image. Chemical development is a catalytic reaction in which the normal speed of reduction of unexposed silver halides by reducing agents of a certain kind (which is a slow reaction when the silver halide is embedded in a colloid medium) is enormously accelerated by traces of this mysterious photo-product in the light-affected salt. Destruction of the latent image (*i.e.*, loss of the developable condition) does not therefore necessarily imply the removal (by solution or chemical combination) of a chemical substance, any more than the "poisoning" of a sample of platinum black by arsenic means that platinum is no longer present when it fails to activate certain well-known chemical reactions. It should also be borne in mind that while silver readily acts as a nucleus for the deposition of more silver from the developing solution itself in the so-called physical development process, the fact that a photographic image can be built up in silver by means of such a physical developer offers little indication as to the chemical character of the original nucleus which we call the latent image, for such solutions precipitate silver on all kinds of surfaces very readily. When, therefore, I use the term "latent image" it should be understood that I really mean the "developable condition," and I admit that this condition may not always be due to the catalyst originally present which was formed by the action of light.

The present paper will be confined mainly to a consideration of three only of the ascertained properties of the latent image: Firstly, the possibility of physically developing an image on a fixed and washed plate; secondly, the possibility of transferring and subsequently developing (both physically and chemically) latent images from the silver salt in which they are formed to another, by changing the former chemically into a less soluble silver salt; thirdly, the destructibility of the latent image by the further action of light itself under certain conditions.

The possibility of physically developing the latent image on an exposed and fixed collodion plate was discovered in 1858 by Young,¹⁴ and then further investigated by Davanne in the following year. In 1895 Kogelmann¹⁵ discovered that gelatino-bromide of silver emulsions behaved in the same way. Further work on the subject by Sterry,¹⁶ Neuhauss, and Lumière and Seyewetz have improved the technique of the process very materially. Moreover, it has been shown that a number of other fixing agents may replace the usual solution of sodium thiosulphate with similar success.

Such experiments constitute one of the strongest pieces of evidence for the material rather than the electrical character of the latent image.

Unfortunately, while physical development entails very little more exposure in the case of very fine grained slow plates, when plates of high speed, coated with what are known as ripened emulsions of silver bromide in gelatin, are employed, it is usually necessary to give at least ten times the normal exposure necessary for alkaline development to get a complete picture. Hence such experiments fail to give evidence in favour of the existence of a newly formed chemical substance in the least exposed parts of a normally exposed rapid dry plate.

The transference of latent images from one silver salt to another has scarcely received the consideration it deserves in connection with studies of the latent image.

In 1881 Eder and Pizzighelli¹⁷ showed that an exposed silver chloride plate could be converted by

treatment with potassium bromide into silver bromide without any loss of the developable condition.

In 1887 Carey Lea¹⁸ showed similarly that light-affected citrate, benzoate, tartrate, and pyrophosphate of silver gave developable silver chloride or bromide after treatment with dilute hydrochloric or hydrobromic acid, even when followed, after a thorough washing, by treatment with dilute nitric acid. Some advocates of physical change theories of the latent image have sought to account for these results by saying that the physically changed condition produced by light is transferred to the newly formed halide in the course of the reaction.¹⁹ This explanation, in the absence of knowledge as to the nature of the first change, explains nothing.

In attempting to apply the method of image transference to rapid gelatin dry plates we are met with the difficulty that only the iodide and sulphide of silver are less soluble than the bromide, and alkali iodides have long been known to have a powerful effect in preventing development. It has been repeatedly stated that they rapidly destroy the latent image. (Dr. Hurter²⁰ himself makes the statement, and uses it as a powerful argument in favour of a physical change theory of the latent image.) Others have affirmed the contrary.²¹

On considering the matter it appeared probable that the loss of the developable condition observed on treating a dry plate with a solution of an alkali iodide might arise from two causes. Firstly, to the known tendency of the alkali iodides to hydrolyse and behave in certain reactions as if they contained free iodine, and secondly to the great capacity of silver iodide for adsorbing soluble iodides, which would very much increase the difficulty of reducing it to metallic silver by alkaline development. To subdue as far as possible the former tendency it is desirable to add a neutral sulphite to the iodide solution employed in any such experiments.

In some recent experiments I have been able completely to fix out exposed gelatin dry plates in two or more changes of strong solution (20%) of potassium iodide containing 5% of (cryst.) normal sodium sulphite and, after well washing out the iodides with a weak (2%) normal sulphite solution, to develop the fully exposed parts in the Lumière and Seyewetz physical developer (silver sulphite and *p*-phenylenediamine). The loss of the image in the less exposed parts even with slow plates is, however, considerably more than when a neutral thiosulphate solution containing sulphite is employed.

The gelatin coating of most plates will not withstand prolonged treatment with 20% iodide solutions without softening badly or even dissolving, and it soon became clear that part of the loss of developable image was attributable to its mechanical removal with the more soluble constituents of the gelatin itself.

This was confirmed by the observation that a preliminary toughening of the gelatin in a weak formalin bath before fixation was advantageous with all fixing agents, and particularly so with strong iodide solutions. These experiments showed that iodides, even in very strong solutions, certainly do not destroy the latent image completely. Unfortunately they throw practically no further light on the question of the nature of the photo-product, and furnish no evidence whatever as to the effects of minimum exposures.

In 1891 Lainer²² discovered that extremely weak iodine tincture when added to certain of the less energetic alkaline developers considerably augmented the amount of image which could be developed for a given exposure. Interesting studies of this remarkable action of soluble iodides have since been made by v. Hübl,²³ Lüppo-Cramer, and others in which the extremely weak iodide solutions employed were sometimes applied to the plate before development, but no systematic attempt appears

to have been made to study the effect of completely converting all the silver salts present into iodide before development, by means of longer bathing and stronger iodide solutions. The effects produced by extremely dilute solutions are very remarkable, and are undoubtedly important for any student of the latent image problem, quite apart from their practical applications. They appear to me to be best explained by Lüppo-Cramer's suggestion that an appreciable part of the latent image is situated within the crystalline silver halide grain, and is only brought into play by the disruptive processes consequent on the rapid conversion of the surface of the grain into silver iodide or by the use of the most energetic developers. It should also be borne in mind that the alteration of the size and distribution of the substance of the grain which partial conversion to iodide must entail is also likely to affect the bulk, the distribution, and the composition of the reduced silver and unreduced silver iodide²⁴ and sulphide of which the negative grains are usually composed.

When, however, the iodide solutions used convert the whole of the silver bromide into iodide, but are not strong enough to fix the plate, the results hitherto obtained have lent strong support to the supposition that the latent image is to a considerable degree destroyed by iodides.

Five or six minutes' immersion in a 1% solution of sodium or potassium iodide containing 2-3% of a normal sulphite is sufficient to convert all the silver salts into iodide if the solution is kept moving over the face of the plate unless the emulsion coating is unusually heavy. Washing after conversion into iodide should be carried out with a dilute neutral sulphite solution, and development is fairly rapid in a solution containing about 1% of amidol and 10% each of crystallised sodium sulphite and carbonate. Owing to the insensitiveness of silver iodide when precipitated in presence of excess of a soluble iodide, it is possible to develop plates after the iodising process is complete in a strong white light. Of course, should any part of the original bromide remain unconverted to iodide it will be fogged and blacken immediately in the developer, while even in a dark-room any unchanged bromide fogs badly in a few minutes in alkaline amidol, so that unchanged patches, if present, are readily detected.

Some time after I had first succeeded in making negatives in artificial white light in this manner I found that a patent had been taken out in Germany by R. Freund²⁵ for a process of developing plates in weak daylight after treating them with a 4% solution of potassium iodide followed by a rinse in water before development. I can only say that in

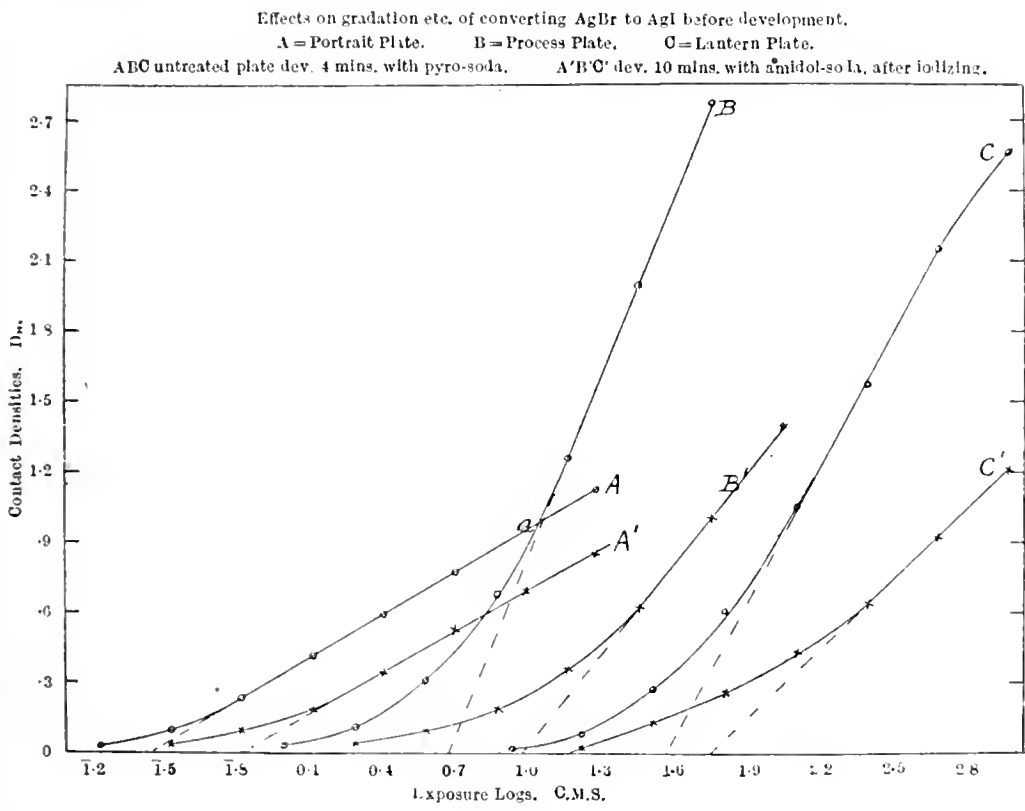


Fig. 2.

Some recent experiments made in the Ilford laboratories have demonstrated, however, that it is possible to "transfer" the latent image from both slow and rapid plates to silver iodide with very little loss indeed, and that such "iodised" plates may be satisfactorily developed either by means of the Lumière and Seyewetz physical developer or chemically by means of solutions of amidol in sulphite and alkali carbonate.

the light of my experiments I am not surprised he did not develop his process to a successful issue. I do not, however, anticipate that the possibility of developing negatives in artificial light will induce photographers to abandon the use of a dark-room for this part of the process of negative-making unless the serious disadvantages apparently inherent in the method can be removed. The chief objections are (1) that some increase of exposure is usually

necessary, though this does not amount to more than two- to three-fold under the best conditions; (2) that the iodising treatment must be carried out in some form of dark chamber; (3) that the highly poisonous cyanide fixing bath is almost essential to dissolve the undeveloped silver iodide in a reasonably short time. There is also a fourth difficulty which I will mention shortly. But for these drawbacks the process would be most useful for developing panchromatic plates.

The diagrams given in Fig. 2 show in the form of characteristic curves some of the results obtained in the Ilford laboratories for a range of sensitive negative materials.

When the silver bromide of a dry plate is converted to iodide the clear, sharp-edged crystals of which the emulsion grains consist are completely changed in character. They lose their crispness of outline, and apparently become a more or less confused aggregate of either amorphous or micro-crystalline silver iodide, as the accompanying photomicrograph shows (Fig. 3). It is therefore not surprising, in view of what I mentioned earlier concerning the influence of grain size on the shape

it is still capable of exercising its powerful catalytic influence in the developing process. That less of the latent image is lost in this process than by fixation followed by physical development is, I believe, due to the partial blocking of the pores of the gelatin by the new halide as it replaces that originally present.

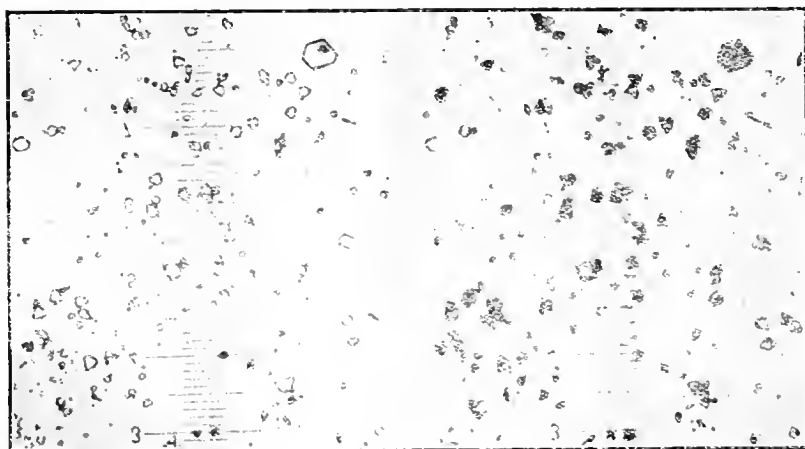
The last reaction of the latent image to which I wish to direct attention is its light-sensitiveness.

The best known and most discussed evidence of this is embodied in numerous very conflicting studies of the phenomena of so-called reversal or solarisation. The most thorough investigation of these phenomena is, I believe, due to Volmer²⁶ and Schaum, but as these effects are notoriously complex and refer to conditions under which chemical change has certainly already begun, I do not propose to discuss them here.

It has, however, long been known that it is easy to demonstrate the light-sensitiveness of the latent image in its early stages under certain conditions.

In 1859 Poitevin²⁷ discovered that if a collodion wet plate be uniformly fogged, then washed to remove the excess of silver nitrate and subsequently

1 small scale division = 1.3μ (thousandths of a millimetre).



Natural emulsion grains.

After conversion to iodide.

Fig. 3

of the characteristic curve, to find that those derived from iodised plates often differ somewhat markedly in character from the characteristic curves of the original dry plates employed. An interesting new field of research is open to exploration in the comparative study of the characteristic curves of "transferred" latent images after development. Such "iodised" plates also respond very well to physical development in Lumière and Seyewetz' silver sulphite and *p*-phenylenediamine developer, giving clear negatives with much more detail than can be obtained from a given exposure after fixation in weak sodium thiosulphate. In all cases the developed plates should be fixed in a weak (2%) solution of potassium or sodium cyanide containing 2% of crystallised normal sodium sulphite, and it is preferable to add a little sulphite to the first few changes of water during the final washing process.

I am of opinion that image transference should be regarded as fixation before development, followed by precipitation *in situ* of another silver salt in place of that dissolved. The photo-product, whether it be modified or not, is not chemically destroyed, but clothed anew in a garment of freshly precipitated and less soluble silver halide on which

immersed in a bath of potassium iodide, then, on exposure in the camera the latent image originally formed is destroyed where the light has fallen on the plate. It therefore becomes possible, after another wash to remove the excess of soluble iodide, to develop a positive picture by means of the usual acid ferrous sulphate (or pyrogallol) and silver nitrate developer. This remarkable reversing action of light on the latent image in the presence of soluble iodides I have recently been able to obtain quite easily with gelatin plates and alkaline development.²⁸ It suffices to give an ordinary negative plate such an exposure to diffuse light as could be developed to a good density in a pyro-soda developer, then to bathe the plate in a weak iodide and sulphite solution till completely converted to silver iodide, and expose the still wet plate behind a transparency. After a brief wash, the plate will develop in alkaline amidol giving a positive copy of the original.

I have also succeeded in making fairly good direct positives in the camera with exposures of 5 to 15 minutes at F8 in a well-lighted studio. (The slide shown is one of the best of them.)

On making an exposure in a spectrograph it was extremely interesting to find that the reversing

rays are restricted almost entirely to a narrow band in the blue of the spectrum lying between 0.43 and 0.48 μ with a sharp maximum at 0.45 μ , as Fig. 4 shows.



Fig. 4.

I particularly wish to emphasize the fact that this reversal will take place perfectly well in the presence of neutral sulphites, thiosulphates, and other halogen-absorbing reagents, conditions under which the liberation of free iodine would appear to be most improbable.

Thiosulphates act as powerful sensitizers in this reversal process, and it is therefore interesting to notice Carey Lea's observation²⁰ that the conversion of gold-coloured allotropic silver to the crystalline intermediate form was accelerated by traces of (hyposulphite?) impurity in the paper it was coated on.

One of the best iodising solutions for reversal effects on negative plates consists of 1 g. sodium iodide, 2 g. cryst. sodium sulphite, 2—4 g. cryst. sodium thiosulphate, 100 c.c. water.

It will at once be seen that the possibility of developing in white light plates which have been iodised after exposure is therefore subject to the further drawback that most of the iodising solution must be removed by washing before light is admitted to the plate, otherwise a serious loss of image may result, though the addition of thio-cyanates to the iodising bath diminishes this tendency. Nor does it appear likely that the direct production of positives in the camera will prove more successful as a practical process on gelatin plates and by chemical development than did Poitevin's original discovery. Both processes, however, are of the greatest scientific interest in their bearing on the problem of the nature of the latent image.

It must, I think, be admitted that many phenomena, and particularly those connected with physical development after fixation and the transference of latent images from one silver salt to another, speak strongly against the view that the latent image is due to a purely physical alteration, electrical or otherwise, of the silver salt. If, then, we must conclude that the latent image is a substance present in excessively minute amount, we are driven to ask what is its nature?

All attempts to prepare true subsalts of silver have failed, except in the case of Guntz' subfluoride,²⁶ and even that is challenged by Vanino and Sachs.²¹ Moreover, the work of a long line of investigators,²² terminating with K. Siehling and W. Reinders, seems to prove conclusively that the photosalts of Carey Lea, which he regarded as lake-like complexes of a subsalt and normal halide, are really solid solutions of colloidal silver in silver halide. Hence it would appear on chemical grounds that the product of light action must be silver rather than a subhalide. One cannot fail to be impressed by the extreme readiness with which silver in the colloidal or even in the finely-divided metallic state goes into solid solution in silver chloride and bromide, and by the great resistance offered by some of these solid solutions to attack by silver solvents and to complete reduction by reducing agents.

All chemical change theories of the latent image have constantly been challenged on the ground that

the amount of energy available in the light required to produce a developable image is totally inadequate to overcome the chemical affinity of the silver for the halogen atom. In its most concrete and recent form this challenge is reiterated by Mees,²³ on the authority of P. G. Nutting.

Consideration of the statement, however, seems to show that it proves too much, for it claims "that it is quite certain what happens when the latent image is produced by a minimum exposure to light, and that the change is really the unit change that can be suffered by any substance when acted on by energy"; in this case, the loss of one electron per silver bromide grain.

It is to be noted, however, that the figures given indicate that the amount of energy capable of producing the first developable impression on a rapid photographic plate must be less than one five hundredth part, and probably nearer one five thousandth of that stated to be required to liberate one electron from a gas molecule.

If, owing to this discrepancy, we must assume that an electron can be so much more easily detached from a solid particle of silver bromide than from a gas molecule, I see no reason why one may not justifiably assume the same to be true for the detachment of one or a few atoms of bromine per particle, especially if the silver bromide grain is embedded in a medium capable of uniting chemically with bromine.

I confess, however, that while such an assumption may be legitimate, I do not consider it entirely satisfactory as an explanation of the beginnings of photographic change in our modern highly-sensitive emulsions. The fact that very sensitive emulsions can be prepared in colloidal silicic acid tells against it, as does also the inability of powerful halogen absorbents to enhance speed when added to an emulsion, although, as is well known, some of them are very effective in inhibiting reversal.

It is much to be regretted that the photographic researches of Carey Lea, and especially his classic papers on the photosalts and the colloidal forms of silver,²⁴ have not been re-published in book form in this country, as they have in Germany, and hence remain largely unknown to the British photographic industry. Much study has been devoted to these extraordinary materials in Germany and Holland, and there can, I think, be no doubt that the work of Reinders²⁵ in particular is the most important contribution of this century towards a solution of the latent image problem.

Carey Lea showed that there is an almost infinite variety of these photosalts, differing in colour, in resistance to oxidising agents, and in light-sensitiveness. He showed that a pale pink form of photochloride containing only a fraction of 1% of dissolved colloidal silver is far more light-sensitive than the normal colourless salt, and that the golden coloured and other forms of colloidal silver ultimately revert to ordinary white (metallic) silver both on exposure to light and on friction.

Reinders has carried the matter still further by preparing solid solutions of colloidal silver in well crystallised specimens of silver chloride, and by showing that some of them are very much more light-sensitive than normal silver chloride, and that when these crystals darken in the light they do so right through the body of the crystal. He also records numerous other interesting observations, such as, that while solid solutions of gelatin in silver chloride raise the light-sensitiveness of the latter very appreciably, a cream coloured solid solution of colloidal silver in silver chloride is still more sensitive; further, that the amount of colloidal silver taken up by silver chloride during crystallisation from ammonia is a maximum in the absence of gelatin, and rapidly decreases in its presence until, with moderate concentrations, quite colourless crystals are obtained even from dark solutions rich in colloidal silver.

It should also be mentioned that R. Lorenz and K. Hiege³⁵ have shown that when clear silver chloride and bromide crystals darken in light, the first effect is the production of a fog or general greying, which later on becomes recognisable under the ultra-microscope as minute particles distributed over the crystal surfaces. These gradually coalesce to form relatively large, dark aggregates of metallic silver with clear interspaces. On these and other earlier observations they advanced the opinion that the latent image is due to colloidal silver in ultra-microscopic form, a view now held by many³⁶ of the supporters of the silver germ theory revived by Abegg³⁷ in 1899.

It is, however, a fact that considerable quantities of colloidal silver solutions (collargol) can be added during the process of making a photographic emulsion without causing any harmful fogging effects, unless the silver is precipitated on the silver bromide grains by addition of acids or other reagent.³⁸

Consideration of these and other aspects of the problem have led me to the conclusion that in our most highly-sensitive photographic plates we are dealing with crystalline silver bromide in which, besides gelatin, some highly unstable form of colloidal silver exists in solid solution, and that it is this dissolved silver which first undergoes change on exposure to light. If this be so, it is clear that it is the function of the ripening process to produce this colloidal silver in the grains of the emulsion in the most sensitive (resonant) form, while avoiding any means which might bring about its precipitation in the gel form—the gel or electrically neutral form of colloidal silver being regarded as the germ or catalyst required to promote development.

J. M. Eder's observations³⁹ on the colour-sensitising action of colloidal silver on silver chloride and bromide serve to remind us of the hitherto unexplained phenomenon that ordinary gelatinbromide emulsions frequently possess a slight sensitiveness to red rays⁴⁰ (sometimes extending through the yellow into the green) at some stage of their preparation. It appears natural to suppose that this colour-sensitiveness is also due to colloidal silver.

Moreover, Carey Lea⁴¹ has shown that some forms of soluble colloidal silver may be restored to the soluble peptised condition by contact with certain salt solutions; it appears possible therefore to offer a reasonable explanation of solarisation by assuming a peptising action on the part of the later-formed chemical products of light action (bromine, hydrobromic acid, etc.) with formation of a photohalide relatively rich in dissolved silver, but almost undevelopable. This suggestion appears to be supported by Carey Lea's observations on the action of nitrites, which are known to inhibit reversal, since he found them to give an insoluble (non-peptisable) form of silver gel. That soluble iodides have a solvent action on silver in the light is fairly obvious from the old direct-positive printing process,⁴² using sun-darkened silver chloride paper after bathing it in a soluble iodide.

Eder's work on the action of nitric⁴³ acid and Sterry's⁴⁴ work on the action of chromic acid on the latent image both seem capable of a complete explanation on the assumption that only true solutions of colloidal silver in silver halide (photosalts) remain after such treatment, and that these are reducible with difficulty by developers. Again, E. Albert⁴⁵ found that if, after giving an exposure to a plate coated with a collodion emulsion, it is treated with a silver solvent (e.g., nitric or chromic acid), then washed and re-exposed to diffused light, a reversed (positive) picture is obtained on development. This experiment also becomes intelligible if we assume that no free silver but only a difficultly reducible and relatively insensitive photosalt remains

in the first exposed areas after treatment with chromic acid.

Such an hypothesis as I have put forward, while it does not throw much light on the why and wherefore of our highly empirical manufacturing processes, seems to offer a more intelligible explanation of the early part of the photographic process than any current theory of the latent image, without calling for any modification of the best supported explanation of the later stages of light action.

It has the merit of reconciling the views of those who demand merely a physical change, those who require the formation of free silver, or a material chemically different from those initially present, to act as a nucleus for development, and it should also meet with the approval of the exponents of the electron theory, since it is apparently well established that colloidal solutions of silver are negatively charged and are precipitated when the charge is lost.

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Manchester Section.

Meeting held at the Grand Hotel on March 5, 1920.

MR. JOHN ALLAN IN THE CHAIR.

THE NEUTRAL HYDROLYSIS OF GUNCOTTON, WITH A NOTE ON THE ALKALINE HYDROLYSIS OF GUNCOTTON.

BY PROF. E. KNECHT AND CAPT. B. R. BOSTOCK.

It is well known that nitrated cotton possesses a pronounced affinity for basic colours, and that it can be dyed with these without the aid of a mordant, but up to now no satisfactory explanation of these phenomena has been advanced. About the year 1902 an attempt was made by one of the authors to ascertain whether a soluble substance could be isolated from guncotton or its decomposition products which would precipitate the basic colours from their aqueous solutions; nothing of the kind could be obtained from the solution of guncotton in boiling caustic soda, but by effecting the hydrolysis of guncotton with cold caustic soda a solution was obtained from which acids threw down a copious white precipitate which on washing and drying yielded a white powder. This substance is somewhat soluble in water, and the solution thus obtained yielded coloured precipitates with basic colours, a property which is shown by one of the products of the alkaline hydrolysis of wool.

Since wool, when heated under pressure with water has been found to yield a product which showed properties in regard to dyestuffs similar to the properties of one of those obtained by its alkali-

line hydrolysis, it was thought that guncotton might behave in an analogous manner. Although a solution was obtained by heating guncotton with water under pressure it was not found possible to isolate from this any substance capable of precipitating basic colours. The primary object of the experiment had thus failed, but it was thought that as guncotton had not been previously completely hydrolysed by water the results obtained might be of some interest.

Silberrad and Farmer (J. Chem. Soc., 1906, 1182) have made an elaborate investigation into the action of water on guncotton at 5-4-4° C. Exposure to the action of water at this temperature was allowed to proceed for 23 weeks, after which a marked deterioration had set in. The decomposition products which had gone into solution were found to consist of nitrous, nitric, formic, butyric, dihydroxybutyric, oxalic, tartaric, isosaccharinic, and hydroxypyruvic acids. Carbohydrates were also found by the fermentation test. The residue consisted of unchanged nitrocellulose.

In our first experiment a small quantity of guncotton was heated for $\frac{1}{2}$ hr. in a sealed tube with water to 200° C. On examining the tube it was found that there was considerable pressure, and that the guncotton had entirely disappeared. The water was of a brownish colour and contained a little charred organic matter, the solution smelling strongly of caramel. Further experiments were conducted on guncotton prepared from pure acids and bleached cotton wool. The product contained 13.05% N. To ascertain the temperature at which complete hydrolysis is effected, sealed tubes, each containing 0.5 g. of guncotton and 25 c.c. of water were heated for $\frac{1}{2}$ hr. in a cannon furnace to 150°, 160°, 170°, 180°, 185°, and 190° C. Only in the last case was any action noticeable, but in this one the guncotton had entirely disappeared, leaving a pale straw-coloured liquid. It was eventually found that a difference of only 2° C. made all the difference between extremely rapid and extremely slow hydrolysis. The rate of hydrolysis and the temperature at which it occurs can be accurately observed by conducting the experiment in a sealed capillary tube containing water, the operation being carried out in practically the same way as the determination of a melting point. It was found that if the temperature was increased even 10°—15° C. beyond 190° a considerable amount of charring took place. Consequently we kept as near to 190° C. as possible in carrying out the hydrolysis for the further work detailed below. The amount of water relative to the amount of guncotton appears to have some influence on the rate of hydrolysis. Two tubes each containing 25 c.c. of water but one charged with 0.5 g. and the other with 1 g. of guncotton, were heated side by side in a cannon furnace to 190° C. In the first case hydrolysis was complete in $\frac{1}{2}$ hr., but in the second over an hour was required before complete solution occurred.

The volume of gas given off from 0.5 g. of guncotton on heating for $\frac{1}{2}$ hr. with 25 c.c. of water to 190° C., collected and measured at atmospheric pressure, was 99 c.c., the same result being obtained in several successive experiments. The amount of guncotton used for collecting the gas for examination was 0.3 g., and in these cases the tubes were completely evacuated before sealing off. After heating in the cannon the gases were drawn off under vacuum. It was found that under these conditions there was not always the same uniformity in volume as in the previous experiments, in which the gases were drawn off under atmospheric pressure, under which circumstances most of the nitrous oxide would remain dissolved in the water.

The gases were analysed in a Bone and Wheeler gas analysis apparatus and gave the following results:—

No. 1 sample of guncotton.—This was carried out

in duplicate. In each case exactly 80 c.c. of gas was obtained having the following composition:—

	(1)	(2)
CO ₂	64.87%	64.9%
N ₂ O	22.37 ..	22.4 ..
CO	3.0 ..	3.0 ..
N ₂	9.75 ..	9.75 ..

No. 2 sample of guncotton.—The gas given off in this case amounted to 87.5 c.c., and contained CO₂ 64.7, N₂O 20.5, CO 5.7, N₂ 9.1%.

There would thus appear to be a considerable discrepancy between the two samples. If, however, the amounts of N₂O and N₂ obtained are calculated on the volumes of the gas evolved it will be seen that they are very nearly the same in both cases. The increased volume obtained from No. 2 sample was made up of CO₂ and CO.

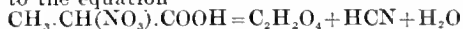
The amount of nitrogen thus found was equal to 10.25% of the weight of the guncotton, and it appeared to be of interest to ascertain the disposition of the remaining 2.8%. An examination of the solutions obtained showed the presence of hydrocyanic acid, ammonia, formic acid, tartaric acid, hydroxypyruvic acid, nitric acid, and traces of nitrous acid.

On heating three separate tubes, each containing 0.5 g. of guncotton and respectively 25 c.c. of (a) dilute caustic soda, (b) dilute sulphuric acid, and (c) water, the amounts of gas formed were: (a) Nil, (b) 126 c.c., and (c) 100 c.c. Hydrocyanic acid was formed only in the last case, viz., by heating with water. The amount of hydrocyanic acid formed was not inconsiderable—in fact, its presence was easily detected by smell and by the formation of a considerable amount of Prussian blue on applying the ordinary test for cyanides. The following estimations were made on the solution obtained in each case from 30 g. of guncotton hydrolysed in an autoclave at 190° C.

	% N as HCN.	
	In distillates.	In residual solutions.
(1)	0.176	—
(2)	0.242	—
(3)	0.211	0.216
(4)	0.216	0.216
Average	0.210	0.216

The total percentage of nitrogen obtained in the form of hydrocyanic acid was thus 0.426%.

The appearance of hydrocyanic acid as one of the products of hydrolysis may seem remarkable at first sight, but in view of L. Henry's statement that nitro-lactic acid decomposes spontaneously according to the equation



into hydrocyanic and oxalic acids (Ber., 12, 1837), the possibility of its formation is by no means ruled out. It is not suggested that in this case nitro-lactic acid is formed as an intermediate product; indeed, the absence of oxalic acid from the decomposition products would indicate that this was not the case. But the formation of some intermediate nitrate which behaves in an analogous manner is not excluded.

The formation of hydrocyanic acid as one of the products of hydrolysis may possibly account for several of our results. In the first place, it is well known that this substance is transformed under favourable conditions into ammonium formate, and this would account for the presence of ammonia in the solution. The presence of ammonia would also account for the observation that nitrous acid could only be detected in traces in the decomposition products. It will also be noticed that the amounts of hydrocyanic acid and ammonia found in the solution varied somewhat in each individual experiment, although the conditions as far as time, temperature, and amount of water to guncotton were concerned were kept as constant as possible.

The estimation of the ammonia was effected in two ways, viz., by the Nessler process and by the

formaldehyde process. Before using the latter process it was first ascertained that none of the constituents remaining in the solution after boiling with a known volume of N/10 sulphuric acid had any effect on the accuracy of the process.

The following results were obtained, a separate hydrolysis of 0.5 g. being carried out for each estimation. The figures represent the ammonia found in percentages of nitrogen: By Nessler process, 1.20%; by formaldehyde process, 1.08, 1.32, 1.19%; average, 1.20%.

The total nitrogen in the solution estimated by the Kjeldahl process was found to be 1.59%. Subtracting from this the nitrogen present as ammonia (1.20%), we have 0.39%, of which 0.12% was found by the nitrometer to consist of nitrogen in the form of nitric acid. The disposition of the nitrogen after neutral hydrolysis was therefore:—

As nitrous oxide and nitrogen	10.24%
As hydrocyanic acid	0.43
As ammonia	1.20
As nitrate	0.12
Otherwise bound and in solution	0.27
	12.26

We were thus not successful in accounting for the whole of the nitrogen. Nearly 12% of the 13% is, however, accounted for by the nitrous oxide and nitrogen and the hydrocyanic acid and ammonia.

The total solids in the solution amounted in one case to 15% and in another to 16% of the weight of the guncotton hydrolysed. The purified residue examined in the polariscope showed a *laevo*-rotation and was found to contain besides ammonia, tartaric acid, formic acid, and hydroxypyruvic acid. It exerted a strong reducing action on Fehling's solution, but the figures obtained, expressed as dextrose, varied so much with different preparations that no conclusions could be drawn from them.

Further work on this reaction carried out in an autoclave fitted for temperature and pressure readings showed that the action commenced at a temperature between 185° and 190° C., and was not explosive but gradual. Unfortunately, the notes, including diagrams of the curves and further analyses had to be left behind by one of the authors on leaving Russia in 1917, and there does not seem to be any prospect of their being recovered.

Some attempts were made to effect the decomposition without the use of pressure, e.g., by raising the boiling point of water by the addition of neutral salts, with the object of obtaining larger quantities of the decomposition products for their more complete examination. These were unsuccessful, but we have recently ascertained that guncotton readily dissolves in hot glycerin, the hydrolysis apparently taking a similar course to that shown with water under pressure. Incidentally it may be mentioned that cordite when heated with glycerin also evolves gases and goes into solution.

Note on the action of caustic soda on guncotton.

It is well known that the nitrocelluloses dissolve more or less readily in caustic alkali. The more highly nitrated products dissolve even in the cold, while if the nitration has not proceeded far an elevated temperature is required. It has been found that even if the nitration has not proceeded far enough to affect the inflammability of the cotton material, the latter may nevertheless be rendered soluble by prolonged boiling in caustic alkali, and advantage was taken of this by one of the authors in 1897 to prepare a groundwork of partially nitrated cotton for lace manufacture (Eng. Pat. Appl., 14,642 of 1897), which would be destroyed in the process of bleaching. The nitration was effected by immersing the cotton fabric for ten minutes in a mixture of 100 pts. nitric acid of sp. gr. 1.415 and 40 pts. concentrated sulphuric acid, care being taken not to allow the temperature to rise above 20° C. A similar result was obtained by denitrating a more

highly nitrated fabric by means of ammonium sulphide to a point at which, while retaining its solubility in caustic alkalis, it has lost its inflammable character.

It is further known that when the nitrocelluloses dissolve in caustic alkalies a complete breakdown of the cellulose molecule ensues, and many researches have been carried out on this question. Probably the most elaborate of these is that of Berl and Smith (J., 1908, 537; and Z. ges. Schiess- u. Sprengstoffwesen), who succeeded in identifying a large number of organic compounds in the products of cold and of hot alkaline hydrolysis.

When a nitrocellulose is dissolved in caustic soda* it is found that the solution contains both nitrate and nitrite, and there are on record several attempts to estimate quantitatively the relative proportions of these two acids. The most recent investigation in this direction is that of Carlson (Ber., 1907, 1192), who gives results obtained on a number of organic nitrates. Of these only one, viz., methyl nitrate, was found to be hydrolysed with caustic alkali without any accompanying reduction of the nitric to nitrous acid. Carlson dissolved a known weight of the nitrocellulose in alcohol and ether and added a solution of caustic soda, the whole being allowed to stand for several hours. The solvent was then distilled off in a vacuum, the residue introduced into an evacuated flask, and after the addition of potassium iodide and hydrochloric acid the liberated iodine titrated with thiosulphate. He found in this way that 82% of the total nitrogen was given off as nitrite.

Carlson's pyroxylin (with 12.5% N), being soluble in alcohol and ether, did not represent the most highly nitrated cellulose, and it seemed of interest to ascertain how such a product would behave when dissolved directly in warm caustic soda.

The mode of procedure was to dissolve a known weight of guncotton in caustic soda on the water-bath and after cooling the solution and making up to a known volume to titrate with this solution into a definite volume of standardised aniline hydrobromide in presence of an excess of hydrobromic acid. It was previously ascertained that the method gave correct results by titrating with a solution of known strength and containing caustic soda into an acid solution of aniline hydrobromide. By adding a known amount of potassium nitrite to the caustic soda solution of a known weight of guncotton it was shown that the organic substances had no effect on the accuracy of the result, the whole of the additional nitrite being exactly accounted for in the titration result. The total amount of nitrogen which had passed into solution as nitrite and nitrate in the hydrolysis was estimated as ammonia by distillation with caustic soda and aluminium borings and was found to be very nearly identical with that found in the guncotton by means of the nitrometer.

The guncotton employed for the experiments was prepared in the same way as that used for the neutral hydrolysis and contained 13.04% N.

The principal results obtained are given in the following table:—

Guncotton used	Nitrogen by nitrometer %	Mean %	Nitrogen as nitrite %	Mean %	Percentage of total nitrogen reduced to nitrite
Sample No. 1	(1) 18.05 (2) 13.08	13.07	(1) 8.49 (2) 8.5	8.49	65%
Sample No. 2	(1) 12.98 (2) 13.05	13.02	(1) 8.32 (2) 8.5	8.4	64.6%

*In spite of the absolute dissimilarity of the two substances it is remarkable that guncotton when being dissolved in hot caustic soda should evolve a smell similar to that produced when wool is being dissolved to the same reagent.

The amounts of nitrogen in solution estimated as ammonia were for sample No. 1 13.2% and for No. 2 13.1%.

It is thus seen that under the conditions of the experiment about 65% of the nitric acid present is reduced in order to provide the oxygen for the organic oxidation products which result from the hydrolysis. This figure is about 17% lower than that found by Carlson, but this may be accounted for by the fact that we used a more highly nitrated cellulose and hydrolysed under different conditions.

It will be evident that the extent to which the nitrogen is reduced in the alkaline hydrolysis is nothing like as great as when the hydrolysis is conducted in water.

College of Technology, Manchester.

DISCUSSION.

Mr. L. G. RADCLIFFE was particularly interested in the fact that hydrocyanic acid was produced by the action of the alkali upon nitrocellulose. He had had a somewhat similar experience during the course of certain experiments in connection with nitrated oils when he had endeavoured to remove the nitro or nitrate group in nitrated ricinoleic acid. The nitrogen was not entirely removed by boiling with alcohol-potash, but some ammonia was evolved.

Mr. D. M. PAUL asked if the laboratory-prepared guncotton samples were actually neutral. It was difficult to remove traces of acid in the laboratory. The purity and stability of guncotton in manufacture depended upon the removal of acid from the fibre.

Mr. J. GRANT asked if the authors had noticed during the hydrolysis of guncotton by water at such high temperatures as 195° C., that as caramelisation had occurred whether furfuroids or any saturated hydrocarbons had been formed. Apparently the whole of the hydrogen had not been accounted for in the organic acids stated to be present.

Capt. BOSTOCK said that every care had been taken in the preparation of the guncotton, and it was washed thoroughly and repeatedly in warm water. In reply to Mr. Grant, he said that when the experiments had been made at the definite temperature at which they had worked caramelisation did not occur. There was no noticeable formation of methane.

Prof. KNECHT said that Mr. Radcliffe's observation regarding the formation of ammonia from nitrated ricinoleic acid was of great interest. It might be explained upon the basis of Henry's results.

Newcastle Section.

Meeting held at Armstrong College on April 21, 1920.

PROF. P. PHILLIPS BEDSON IN THE CHAIR.

THE SETTING OF CALCIUM SULPHATE CEMENTS.

BY C. L. HADDON, M.Sc.

Calcium sulphate exists in three chemical forms, as the dihydrate $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, the hemihydrate with $\frac{1}{2}\text{H}_2\text{O}$, and the anhydrous salt. Anhydrous calcium sulphate occurs naturally as anhydrite in limestone rocks or with deposits of common salt. The hemihydrate, apart from its manufacture, is found in boiler scale. The dihydrate occurs naturally as fibrous gypsum or satinspar, and as crystalline gypsum or alabaster.

On heating gypsum to about 120° C., it loses three-fourths of its combined water, and forms the hemihydrate (plaster of Paris), which is more soluble in water than the dihydrate. If excess of plaster of Paris is shaken with water and rapidly filtered, a clear solution is obtained, but this quickly becomes turbid due to the precipitation of the dihydrate. It is to this property that plaster of Paris owes its setting powers. When mixed with water the hemihydrate dissolves and the solution becomes supersaturated with respect to the dihydrate, and this is deposited. It has been shown by Le Chatelier that the crystals deposited radiate from different centres and interlock, thus giving the final product the usual properties of a set cement. The temperature of the formation of the hemihydrate is 170° C.

On further dehydrating gypsum the anhydrous salt is formed under normal conditions between 160° and 170° C.¹ The setting properties of the anhydrous salt depend on the temperature of dehydration, but several contradictory statements exist on the subject.²

According to Desch, calcium sulphate partially loses its property of setting with water if heated to 200° C., regains it at 500° C. (at which temperature flooring cement is made), and loses it again at higher temperatures. At its best it sets much more slowly than plaster of Paris, although Roscoe and Schorlemmer state that gypsum dehydrated over phosphorus pentoxide between 60° and 70° C. sets even more rapidly than plaster of Paris.

Owing to its rapid setting, plaster of Paris is mixed with excess of water into a thick cream and then poured into the mould. The time of hardening depends on the amount of water present. The larger the percentage of water present the smaller will be the percentage by volume of cement and the further the crystals must grow before they interlock.

In one experiment two lots of 680 g. of plaster of Paris were mixed with 300 c.c. and 400 c.c. of water respectively with the following results:—

Time.	Tensile strength. (lb. per sq. in.)	
	300 c.c.	400 c.c.
15 min.	70	†
30 min.	220	50
1 hr.	300	230
2 hrs.	300	230

† Broke in machine.

680 g. of plaster theoretically requires only 126 c.c. of water.

In the course of time the excess of water evaporates, depositing the dihydrate on the exposed arms of the crystal clusters and thereby increasing the tensile strength. Thus in the second case mentioned above the strength on drying rose to 350 lb. per sq. in., while similar briquettes kept under conditions which prevented drying did not increase in strength after the first hour.

Analysis of two different kinds of anhydrous cement—fine and medium—showed about $\frac{1}{2}$ % of combined water, a trace of iron and alumina, and in the case of the medium $\frac{1}{2}$ % of calcium carbonate.

In the work described below the strength of the cements was measured by making moulds 1 sq. in. in cross section, and pulling them with the ordinary cement-testing machine till they broke. A tensile test gives more concordant results than a crushing test, is easier to carry out, and requires less material. The briquettes were made as follows: The plaster was sifted through a sieve with 2500 meshes per sq. in. to remove coarse particles and then weighed out and mixed intimately with a measured quantity of water until the paste was homogeneous. Some of the paste was then placed in a previously vaselined mould and rammed till all the air holes were pressed out by means of a

cylindrical brass rod $\frac{1}{2}$ in. in diameter and 9 in. long, which was allowed to fall about $\frac{1}{2}$ in. on to the plaster. This operation was repeated till the mould was full, when it was levelled off.

The method of filling the moulds affects the tensile strength of the material, so that for comparative results a definite method of filling must be adhered to.

When the briquettes were hard they were removed from the moulds and then broken as occasion demanded. They were weighed 6 hours after making, and also daily.

Consideration of the appended data obtained by varying the percentage of water and time of setting show that although more water is originally added than is theoretically necessary, yet it evaporates so quickly that all the cement does not become hydrated. Only about 80% of the plaster was saturated when as large an excess of water as possible was used, and even then there was a tendency for the excess of water to separate and be unequally mixed. Even in the samples kept over water for a week, to prevent premature drying, the cement was by no means fully saturated.

From table 3 it will be seen that the briquette takes a week to dry, but that most of the excess of water had dried off by the end of the second day. Comparison of Tables 1 and 4 shows the very great effect on tensile strength of the excess of water present. In view of the mode of formation of the crystals, it appears probable that as the water evaporates it deposits its dissolved gypsum on the crystalline arms, strengthening them, and hence the mechanical strength of the whole briquette is increased. After 24 hours the mass is hard, and after 6 hours the briquettes are sufficiently hard to be removed from the moulds. The maximum strength is reached in about 10 days; when the briquette is put in water it falls to about 400 lb., i.e., the same strength as when the briquette was prevented from drying.

It is well known that the time of setting of flooring cement is affected by the addition of different salts. Rohland³ considers that substances which increase the solubility of calcium sulphate also increase its rate of setting. This seems fairly easily explained, as if the solubility is increased the rate of solution will also be increased and the rate of precipitation of the dihydrate, or setting, should also be increased. Experiments made with calcium chloride confirmed this. A 5% solution of calcium chloride, according to Lunge,⁴ reduces the solubility of calcium sulphate at ordinary temperatures by one-half; table 1 shows its retarding effect on the setting of flooring plaster. Contrary to this theory there is, however, one class of substances which, as far as I can discover, in dilute solutions invariably decrease the solubility of calcium sulphate and have a great accelerating effect on the setting of the plaster.

Sullivan⁵ showed that in dilute solutions ammonium sulphate decreased the solubility of calcium sulphate, although concentrated solutions had the opposite effect. Cameron and Bell⁶ showed that the injurious effect on the solubility of calcium sulphate increases with rising atomic weights in the series hydrogen, magnesium, ammonium, sodium, and potassium, while Bell and Taber⁷ showed that dilute solutions of copper sulphate decrease its solubility, though more concentrated solutions increase it to the same degree as its solubility in water.

To examine the comparative effect of various sulphates tests have been made with the sulphates of hydrogen, Cu, Fe^{II}, ammonium, Fe^{III}, Al, 5% solutions being mixed with the plaster instead of water. The first effect was to increase rapidly the

¹ Le Chatelier Ann. des Mines, 1887, 346-65.

² Z. anorg. Chem., 35 194-200. Glaschnapp, Tonind. Zeit. 32 1148-52, 1197-1202, 1230-5.

³ Z. Elektrochem., 1908, 14 421-2.

⁴ J. 1885, 31.

⁵ J. Amer. Chem. Soc., 1905, 27, 529-39.

⁶ J. Phys. Chem., 1906, 10, 210-215.

⁷ J. Phys. Chem., 1907, 11, 637-8.

rate of setting; in the case of copper sulphate the cement was stronger after 6 hours than with water alone after 24 hours. This very rapid setting also had a secondary effect in that the water was absorbed before it could evaporate, the result being that at least 90% of the cement was fully hydrated, and thus the damping of the cement while it is setting is unnecessary.

The results obtained with medium cement are shown in table 2, from which it will be seen that after the first day the strength decreases, and then increases again. This peculiarity was noticed in every case with 5% solutions, and will be referred to later.

TABLE 1.

Mixture: 300 g. cement, 100 c.c. solution.
Tensile strength in lb. per sq. in.

	6 hrs.	1 day	2 dys.	3 dys.	4 dys.	5 dys.	7 dys
Neat	broke	250	400	440			550
		290	440	500			600
5% ferrous sulphate ..	120	400	400	350	350	400	500
5% zinc		450					
5% copper	350	450		450			600
	400	500					
5% ferric	294	424	418		482		600
5% aluminium	110	400		380			600
5% hydrogen	280	400					500
5% amm. chloride ..		250	300				350
5% calcium			40		180		200
3% ferrous sulphate ..	90	420	440	380			600
1%	80	300	370	410			600
5% glue	br	br	br	br	br	br	600
10% ferrous phate ..	250	400	350				45

	10 days	14 days	21 days
Neat	600-700	600-700	600-700
5% ferrous sulphate ..	550-650	600-700	600-700
5% zinc	550-650	600-700	600-700

Mixture: 300 g. cement,
100 c.c. solution.

TABLE 2.

Percentage of cement forming dihydrate.
3 pts. cement, 1 pt. water by wt.

Neat cement	70.3
.. .. . (over water 1 week)	87.5
1% ferrous sulphate	83.5
3%	91.6
5%	94.5
10%	96.2
5% (over water 1 week)	95.0
5% ferric	92.2
5% zinc	91.3
5% copper	94.3
5% aluminium sulphate	92.8
5% ammonium chloride	82.5
5% calcium chloride	67.6
5% glue	16.7
Neat cement (300 cement: 136 water)	78.0

Very fine cement. (Warm weather).

330 CaSO ₄ .. 120 water	72.2
.. .. 110	64.3
.. .. 100	60.6
.. .. 100 .. (over water 3 days)	79.0
.. .. 100 5% ferrous sulphate	91.4
.. .. 110 5%	96.2

Briquettes made in hot weather with equal parts by volume of plaster and sawdust set when a 5% solution was used, but owing to the rapid evaporation they would not set when water alone was used, i.e., tensile strength failed to reach 50 lb. per sq. in., and they could be crushed with the fingers.

The addition of a soluble sulphate results in the formation of much smaller and finer crystals. As the vapour pressure of a solution is less than that of water the water will take longer to evaporate, and thus the briquette will be longer in drying. This was found to be the case (see table 3); it was also seen that the maximum tensile strength is not attained until the briquette is dry.

Further tests were made to ascertain the effect of varying the strength of the ferrous sulphate

solution. With a 3% solution the strength remained constant for three days and then increased. With a 1% solution the increase was continuous, and with a 10% solution the setting was accelerated more, but the strength did not increase, as in the other case, within seven days of making.

Cohn⁸ has shown that calcium sulphate is more soluble in ammonium acetate solutions than in ammonium sulphate. The effect of 5% solutions of these two salts was examined. The cement used was coarser than on the previous occasions and contained more iron oxide, but the results (table 5) showed conclusively that ammonium acetate retarded the setting, and ammonium sulphate accelerated it, contrary to Rohland's theory.

TABLE 3.

Drying data.

330 parts fine cement: 100 parts solution.			
Time after making	5% ferrous sulphate gms.	Water gms.	
6 hours	139.3	137.8	
1 day	136.9	133.1	
2 days	136.2	127.5	
3	135.9	126.2	
4	135.7	125.7	
5	135.6	125.6	
7	135.50	125.50	
8	135.45	125.48	
9	135.41	125.40	
10	135.37	125.48	
12	135.32	125.49	
14	135.27	125.47	
17	135.23	125.49	
23	135.10	125.48	
28	135.1	125.50	
Actual cement after ignition	108.8	107.8	

TABLE 4.

Effect of varying water content on strength.

Briquettes were placed in a moist atmosphere, and a glass cover placed over, removed after one week and allowed to dry.

	1 day	2 dys.	3 dys.	4 dys.	5 dys.	6 dys.	7 dys.	14 dys
5% ferrous sulphate	420		350		240		240	490
Water	200	360	370	360	360	360	380	510

TABLE 5.

Effect of ammonium acetate and ammonium sulphate.

3 parts of cement: 1 part water.
Tensile strength.

	6 hrs.	1 day	2 days	3 days	6 days	11 dys.
5% amm. sulphate	190	330	370	410		570
7.5%		350		340		
5% amm. acetate	br	br	70	85	105	
Water only		160		350		

It might be thought that an ammonium acetate solution would accelerate the setting, as it would react with the calcium sulphate, forming an equilibrium mixture of four salts, but it must be remembered that calcium acetate would also be formed, and this exercises a retarding effect.

It is difficult to explain satisfactorily the depressing effect of other sulphates in dilute solution on the solubility of calcium sulphate. Davis has brought forward some evidence to show that when plaster of Paris sets orthorhombic crystals are first formed, and subsequently change into normal monoclinic crystals, the customary form for the dihydrate. Obviously, when a change is taking place a fall in tensile strength must occur. Setting

⁸ J. Prakt. Chem., 1887, H. 43-56.

⁹ J., 1907, 727.

and change would go on side by side with neat cement, the drop effect due to change being masked by the increase in tensile strength, due to setting; while with the more rapidly setting sulphate solutions, one more or less follows the other, and the effect is noticed. This seemed a simple explanation, but if it is noticeable with sulphate solutions it should be much more noticeable with plaster of Paris and water. I was, however, unable to find any trace of it on the second or third day. On the other hand, it may be due to the excess of water present, as table 4 appears to show. It is obvious that after the initial set one is dealing with a fairly concentrated added sulphate solution, and that solution may have some deleterious effect, but a full explanation does not seem possible at this juncture.

The time for the complete hydration of calcium sulphate mixed with water is at least a week (see table 3). Table 1, on the other hand, shows that it had mostly set by then and that it will be impossible to ensure 100% saturation unless the particles are infinitely small. The first maximum for sulphate solutions shows that about 24 hours is necessary with a 5% solution.

In conclusion:—

(1) Plaster of Paris forms a weak cement because of the large excess of water, and hence the smaller percentage of cement. The crystals are also much finer grained than with any other calcium sulphate cement.

(2) Neat calcium sulphate cement must be kept damp to ensure fairly complete hydration. This is not necessary if a sulphate solution is added; a 3–5% solution mixed to a thick paste gives the best cement.

(3) Contrary to Rohland's theory, sulphates exert a marked accelerating effect.

(4) With 5% or stronger sulphate solutions a peculiar drop occurs in the tensile strength after the first day, and the cement subsequently becomes stronger on drying.

DISCUSSION.

Mr. S. H. COLLINS referred to the theory that the tensile strength of wood was decreased by the addition of water or oil, which liquid acted as a lubricant to the fibres. That theory could not be ignored when studying plaster.

Mr. G. WEYMAN said that during the purification of oxide of iron in gas works the presence of water vapour prevented the hard setting to a large extent.

Mr. HADDON, in reply, agreed with Mr. Collins that the explanation of lubrication might apply equally well to cements. The increase in tensile strength on drying was very probably due to some such effect. The question of the dehydration of gypsum was very important. Gypsum cements should never be used where they were exposed to heat; that appeared to explain Mr. Weyman's difficulty with iron oxide, in which case the steam heat dehydrated the plaster. The colloidal theory of the setting of calcium sulphate was not supported by experimental evidence so far as he was aware.

Meeting held at Armstrong College on Wednesday, March 3, 1920.

PROFESSOR P. PHILLIPS BEDSON IN THE CHAIR.

A COMPARATIVE METHOD OF DETERMINING THE HEAT OF CARBONISATION OF COAL.

BY GEOFFREY WEYMAN, M.Sc., A.I.C.

During the carbonisation of coal in continuous vertical retorts the coal is continually abstracting heat from the heating surfaces as it passes down

the retort. The rate at which any specified coal can be passed through and effectively carbonised depends on the rate at which heat can be supplied to the heating surfaces, and this depends in its turn on the thickness and conductivity of the material between the heating flues and the retorts, and the temperature difference between each side of this material. The rate at which the coal can be efficiently carbonised in a particular setting depends on the temperature maintained in the heating flues, since it may be assumed that a certain minimum temperature must be obtained on the inside of the retort. The limit of temperature maintained in the heating flue is fixed by the refractory properties of the brickwork.

If we consider the temperature of the heating flue as raised to a maximum and the rate of coal passing through the retort as also regulated to a maximum consistent with the production of a well-burnt off coke, we can see the effect of altering the type of coal passing through, keeping these conditions constant. When a change is made to a more highly bituminous coal of high coking value, the temperature of the heating flue falls, the coke withdrawn begins to show uncarbonised matter, and ultimately raw coal appears. The make of gas per hour also falls off. Either more heat must be supplied or the rate of carbonisation must be reduced. With a coal more of the steam-coal type—a coal less "fat"—the heat supplied to the setting must be reduced or the rate of coal supply increased.

The rate at which coal can be passed through with efficient coking and with the same supply of heat to the heating flues varies up to 30% according to the type of coal used. The make of gas obtained from a setting varies in a similar manner from 25% to 30%. This variation is naturally of very great importance, particularly in the case of continuous vertical retort plants which are capable of easy adjustment. With horizontal retort plants the effect is to a large extent masked by the fixed times of charging.*

The power of continuous vertical retorts to distinguish coals is merely the result of a more skilful and scientific method of carbonisation, and the fact that losses of heat in dealing with different types of coal has hardly been noticed in working with horizontal retorts is very much to the disadvantage of the latter. A coal which can be carbonised quicker in vertical retorts can also be carbonised quicker in horizontal retorts, and by selecting such as give good results in vertical retorts and using them in horizontals the charging of the latter may be made at shorter intervals, and hence the capacity of the plant per day increased.

For the reasons given above an attempt was made to determine this variation experimentally. An endeavour was made to enclose in a calorimeter a known quantity of heat, and then to introduce into it a weighed quantity of coal. The coal subjected to this amount of heat should be carbonised to an extent depending on how far the enclosed heat was sufficient to decompose it. It would be reasonable to suppose that if the heat were not sufficient the evolution of gas would be small and would cease rapidly, as the absorption of heat by the coal reduced the temperature below the minimum necessary. The evolution of gas was taken as the criterion of the rate of carbonisation, and it was thought possible that the heat required to raise the coal to distillation point might be differentiated from that required to carry on the distillation. How far these ideas were achieved will be seen in the results.

The apparatus used was roughly as follows:—A fireclay furnace 8 in. external diam. was enclosed

* Constam & Schl pfer observe that in a small scale horizontal coal-testing plant, the rate of distillation varies with the type of coal. (See Gas J., 1906, 98, 460.)

in a metal vessel from which it was separated at the sides and bottom by asbestos packing. Inside the furnace was placed a crucible 3 in. x 4 in. high in which a known weight (usually 1 kg.) of copper was melted by the blowpipe operating through an aperture in the vessels and lining. When the copper was molten the blowpipe was withdrawn and the hole plugged with asbestos and the lid packed with asbestos placed on. As soon as the first crystals of copper began to form, or when the temperature of the copper fell to some pre-determined temperature as read by a pyrometer, a thin steel tube containing a weighed quantity of the powdered coal was thrust into the middle of the copper. The tube communicated with a washing bottle and meter, which was read every minute until the evolution of gas had practically ceased. The tube was then removed and the pellet of coke examined.

The following are some of the results obtained on powdered air-dried samples containing 8–10% of ash:—

Readings in hundredths of a cubic foot of gas evolved during each minute.

Coal ..	A.	A.	A.	A.	A (2).	B.	B.	B.	B (2).	C.	C.	E.	F.	J.
Wt. of coal, g.	20	20	20	10	10	10	10	5	10	10	5	10	10	10
Wt. of copper, g.	807	836	1000	1000	1000	1000	1000	823	1000	1000	823	990	990	1000
Min.														
1	1.35	0.50	0.65	0.80	0.0	0.60	0.70	0.0	0.40	2.05	0.35	2.5	2.9	5.9
2	1.65	1.30	1.65	1.50	1.75	0.70	0.70	0.65	0.45	0.85	0.75	2.3	3.2	4.1
3	1.25	1.30	2.10	1.20	1.85	0.60	0.60	0.35	0.30	0.50	0.85	1.7	2.6	1.3
4	1.15	1.40	2.30	1.40	1.80	0.50	0.60	0.25	0.20	0.30	0.80	1.25	1.2	0.1
5	1.00	1.30	2.30	1.10	1.70	0.30	0.30	0.20	0.20	0.30	0.75	1.10	0.8	0.0
6	0.95	1.40	1.80	0.90	1.40	0.20	0.20	0.10	0.10	0.15	0.50	0.45	0.25	—
7	0.70	0.90	1.10	0.60	0.40	0.15	0.30	0.05	0.10	0.15	0.25	0.15	0.15	—
8	0.50	0.70	0.70	0.20	0.15	0.15	0.20	0.05	0.10	0.15	0.05	0.10	0.20	—
9	0.35	0.60	0.40	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.10	0.05	0.10	—
10	0.20	0.40	0.30	0.10	0.05	0.05	0.05	0.05	0.05	0.05	0.00	0.05	0.10	—
Total, ft. per ton	5081	5740	7021	9245	10,620	3963	4166	2438	2083	6096	9143	9956	13,110	11,670

The first three results marked (A), using the same coal, show that increasing the amount of copper increased the extent to which the carbonisation is carried. In the fourth result marked (A) the effect is more marked when the weight of coal is reduced. In the two tests marked (B) on another coal the effect is similar. (B2) is another sample of the same coal (B) taken some months later from another consignment. Although the results (B2) are nearly double those (B), both are of the same order and at the lower limit of the scale. (A2) is a similar sample of the coal (A). The difference in the results from different samples of the same coal is such as might reasonably be expected, and is not of an essential nature. In (C) again we see that the weight of coal is an essential factor.

In a general way the method gives consistent results and a true indication of the comparative amounts of heat required to carbonise the coals.

Taking a weight of 10 g. of coal and 1000 g. of copper as a standard conditions, the types of coal used may be arranged in order of the total amount of gas evolved:—(B) 4000, (C) 6000, (A) 10,000, (E) 10,000, (J) 12,000, (F) 13,000. Arranged according to the amount of gas evolved during the first two minutes the following is the order:—(B) 1.4, (A) 2.3, (C) 2.9, (E) 4.8, (F) 6.1, (J) 10.0.

Thus (B) takes by far the most heat, and probably needs raising to a high temperature before giving up any appreciable quantity of its gas. (C) also requires much heat, but starts sooner than (A), which requires a less total quantity. (E) requires still less heat and evolves its gas more readily, while (F) is superior in each respect. (J) is however astonishing in its readiness to give off its gas. These results agree very well with the experience of the coals in large scale practice.

It must be admitted that the method appears somewhat crude. One of the chief troubles was to get the tube free from adherent copper for further use. This trouble was to some extent obviated by coating the tube with black lead.

Why different coals should give such large differences in the rate of carbonisation is not clear. It has been our experience that when efficiently carbonised the yield of gas and tar per ton of any of these coals is very nearly the same, but the gas is lower in carbon content, and the tar is thinner and more paraffinoid in the case of those coals which carbonise more readily. The yield of coke is distinctly greater in the latter case.

On the whole it appears to me that the primary decomposition of coal is exothermic, but that secondary reactions, particularly further decomposition of the tar, will turn the process into a decidedly endothermic one. In the method outlined above secondary reactions will be included. It is probably the further decomposition of the tar

first formed which causes the greater part of the differences observed. (See also Constam and Kolbe, Gas J., 1908, 103, 384; Hollings and Cobb, Gas J., 1914, 126, 917; Lessing, J., 1912, 465.)

Communication.

THE ANALYSIS AND COMPOSITION OF CRESYLIC ACID.

BY J. J. FOX, D.SC., F.I.C., AND M. F. BARKER, D.SC., A.I.C.

The present communication deals with the methods whereby the proportions of the three cresols in cresylic acid may be determined in a comparatively ready manner. Certain statements as to the composition of cresylic acid are on record, but they mostly refer to the proportion of one or two constituents which can be recovered in a more or less pure state and do not indicate the composition of the original mixture. A fairly full account of these results may be found in Lunge's "Coal Tar and Ammonia" (Part 1, 1916, p. 291 etc.).

To determine the composition of cresylic acid it is necessary to be able to estimate the phenol, if present, and at least one of the cresols with a fair degree of accuracy. Phenol can be determined as already described by us (J., 1917, 842) and *m*-cresol by Raschig's process (Z. angew. Chem., 1900, 14, 759).*

* Dawson & Mountford's process (J.Chem.Soc., 1918, 113, 923) may also be used for phenol, and the method suggested for *p*-cresol on page 943 is available as a check on this constituent.

For our present purpose we have restricted the term "cresylic acid" to tar acids distilling up to 210° C. and then refractionated up to 203° or 204° C. with a good column of the Raschig type. Most of the phenol is presumed to have been removed in the works, leaving only cresols in the fraction referred to. This fraction of the tar acids usually contains less than 10% of phenol. When more is present the determination of the phenol is liable to an error of 2–3%, but we are not aware of any process which gives closer results with commercial material. In any case, for the purpose of analysis, the proportion of phenol can always be brought below 10% by adding a suitable amount of *m*-cresol.

Raschig's nitration process for *m*-cresol has been criticised from time to time, and in order to test its validity we prepared pure *m*-cresol in three different ways. As the results are of great importance for this work, we shall describe them in some detail.

(1) *Raschig's sulphonation method of preparation* (Ger. Pat. 114,975 of 1900).—For this process crude *m*-cresol was sulphonated and the sulphonic acid hydrolysed as previously described (J., 1918, 265 T etc.). The degree of purification effected as determined by Raschig's nitration process is shown by the following results: *m*-Cresol as purchased, assay 46.5%; after first sulphonation, 94.8%; after second sulphonation, 99.0%. The last product was fractionated and the middle fraction acetylated. The acetate was then fractionated and the middle fraction hydrolysed. The resulting cresol was separated into three portions by fractional distillation, the middle fraction again being the only one used. This assayed 99.0% and froze at +3.0° C. As Mr. J. M. Weiss, of the Barrett Manufacturing Co., of New York, had some time ago informed us that he had succeeded by repeated sulphonation in raising the freezing point of *m*-cresol to about +10° C., we sulphonated a large quantity of *m*-cresol seven times and obtained a product freezing at +9.6° C. This material, as well as all the products after the third sulphonation, assayed between 99.0 and 100.0% by the Raschig nitration method. The sulphonation process is applicable for the purification of *m*-cresol because the sulphonic acids derived from phenol, *o*- and *p*-cresols are not decomposed at a temperature which suffices for the decomposition of *m*-cresolsulphonic acid. In fact, it is possible to separate as little as 1% of *m*-cresol contained in *p*-cresol by the Raschig sulphonation process. The method is, however, conceivably open to the criticism that some substance is carried through the whole process, and on that account two other methods were employed.

(2) Thymol was treated with phosphoric anhydride as described by Tiemann and Schotten (Ber., 1878, 11, 769) and the product purified by distillation from the other products of the reaction. The yield of *m*-cresol was only 15% of the theoretical quantity; it froze at +2.8° C., and assayed 99.0%, but may, of course, contain a small proportion of some impurity not removed by fractional distillation.

(3) *m*-Toluidine was converted into *m*-cresol by means of nitrous acid. Kahlbaum's *m*-toluidine was used, and, in addition, some was prepared from benzaldehyde, according to the method of Widman and Ehrlich (Ber., 1880, 13, 676; 1882, 15, 2010). The *m*-cresol so obtained was impure, but after removing the tar and higher boiling phenols a product was isolated freezing at +10.4° C. and assaying 99.0% by the nitration process. This *m*-cresol possessed the highest freezing point of all the samples prepared by us.

The *m*-cresol from (2) and (3) darkens more rapidly than that from (1) when exposed to light. All three preparations assay from 99.0 to 100%, and the "crude" trinitro-*m*-cresol obtained during the

assay has m.p. 105°–107° C. We have confirmed Raschig's statement that 17.4 g. of trinitro-*m*-cresol is obtained from 10 g. of pure *m*-cresol. This value also applies to the assay of mixtures of widely different composition, as shown by the following results:—

Composition of mixture.	<i>m</i> -Cresol determined.
<i>m</i> -Cresol 89.8%, phenol 10.2%	89.1%
<i>m</i> -Cresol 89.6%, <i>p</i> -cresol 10.4%	89.6%
<i>m</i> -Cresol 90.0%, <i>p</i> -cresol 10.0%	87.5%
<i>m</i> -Cresol 79.5%, <i>o</i> -cresol 20.5%	78.4%
<i>m</i> -Cresol 53.0%, <i>o</i> -cresol 47.0%	51.5%

As mixtures of the three cresols with less than 10% of phenol all gave equally close results for *m*-cresol, we conclude that Raschig's nitration method is sufficiently accurate for technical purposes.

We have found that, in applying Raschig's process the best results are obtained by the following slightly modified procedure: 10 g. of the cresylic acid is placed in a 1200 c.c. flask with 15 c.c. of 98% sulphuric acid and heated on the water bath until the liquid is viscous when cooled to the room temperature. The liquid is spread in a thin layer over the bottom of the flask by quickly rotating it and then 100 c.c. of nitric acid (sp. gr. 1.4) is poured on to this thin layer. The flask is replaced on the water-bath at once. In a few seconds a violent reaction occurs which is allowed to proceed unchecked. From this point Raschig's procedure is strictly followed. The product obtained should melt between 102° and 107° C. after drying and should be free from tar. When conducted as described the method can be relied upon for all mixtures, the important point of the whole process being the initial sulphonation. It is also an advantage, when the proportion of *m*-cresol is found to be less than 25%, to repeat the assay after adding sufficient pure *m*-cresol to the original cresylic acid to raise the proportion of the *m*-cresol to about 50%.

In addition to Raschig's method for the estimation of *m*-cresol we have worked out another process which is capable of giving accurate results. This method depends on bromination in a dry solvent, such as carbon tetrachloride, and is based on the fact that in such a solution and with excess of dry bromine, *m*-cresol yields a tribromo-derivative, whilst phenol, *o*-cresol, and *p*-cresol yield only di-bromo-derivatives. About 1 g. of the dried cresylic acid is weighed into a tared flask fitted with a small ground-in tap funnel and an outlet having a glass stopcock. Excess of a carbon tetrachloride solution of bromine containing 200 g. of bromine per litre is slowly dropped into the flask by means of the funnel and the mixture allowed to stand for several hours at ordinary temperature. It is then warmed very gently on the water-bath until most of the bromine has been expelled, care being taken that the temperature does not exceed 50° C. The apparatus is connected to a pump and the solvent very slowly distilled off at about 30 cm. pressure, the temperature of the water bath being kept below 50° C. When the flask is dry the tap funnel and the side tube are removed and the increase in weight due to the bromination is determined. To ensure complete bromination the process may be repeated, but in general this is unnecessary. The temperature of heating should be kept as low as possible, because the bromo-derivatives are appreciably volatile above 70° C. The results found with the pure substances are as follows, the figures referring to the weight of bromo-derivative obtained from 1 g. of substance taken.

It is clear that if *m*-cresol contained, say 2% of phenol, *o*- or *p*-cresol, it would be easily detected by this method. All three specimens of *m*-cresol prepared as described gave results which showed them

to be of at least 99.5% purity, and the melting-

		Calculated.	Found.	M p. of bromo-derivative.	
				Crude.	Purified.
Phenol	..	2.681	2.700	36°	36°-40°
<i>o</i> -Cresol	..	2.463	2.449	56°	56°-57°
<i>m</i> -Cresol	..	3.194	3.189	80.5°	81°-82°
<i>p</i> -Cresol	..	2.463	2.481	47.5°	48°-49°

point of the crude tribromo-*m*-cresol was in no case less than 80.5° C. This method is lengthy, but affords a valuable check on the Raschig nitration method. It is also available for mixtures of the three cresols, using the equation,

$$\% \text{ } m\text{-cresol} = (100 W - 246.3) \div 0.731,$$

where *W* is the weight of bromo-derivative obtained from 1 g. of the mixed cresols. The following are a few of the results obtained with synthetic mixtures:—

Weight of bromo-derivative (per gram).	<i>m</i> -Cresol (found).	<i>m</i> -Cresol (actual).
3.027	77.0%	78.0%
2.701	32.6%	33.3%
2.566	14.1%	14.4%

We have also worked out a third method of determining *m*-cresol which consists of a modification of that originally suggested by Ditz and Cedivoda (Z. angew. Chem., 1899, 13, 873; 1900, 14, 1050). The original process has been subjected to adverse criticism by Russig and Fortmann (Z. angew. Chem., 1901, 15, 160), but as it is very rapid we thought it worth while to investigate it carefully with a view to removing the difficulties connected with the process. Ditz and Cedivoda state that under the conditions given by them *o*- and *p*-cresol use up exactly 2 mols. and *m*-cresol 3 mols. of bromine per mol. of cresol. The process is carried out by shaking for exactly one minute an aqueous solution of cresol with bromine obtained from acidified potassium bromide and bromate, and liberating iodine from potassium iodide with the excess of the bromine. We find that on shaking for the prescribed time, *m*-cresol absorbs exactly 3 mols. of bromine, but *o*- and *p*-cresols absorb rather more than 2 mols. of bromine. Our results, after numerous assays, are as follows, the figures referring to the weight of bromine absorbed per mol. wt. of substance:—

	Phenol.	<i>o</i> -Cresol.	<i>m</i> -Cresol.	<i>p</i> -Cresol.
Found ..	482.8	333.3	478.6	331.5
Calculated ..	480.0	320.0	480.0	320.0

In the method as laid down by Ditz and Cedivoda there is considerable difficulty in obtaining an end-point in the titration of the liberated iodine with *p*-cresol, and to a less extent with *m*-cresol, because the iodine is apparently adsorbed by the bromo-derivative and is only slowly given up to the aqueous liquid. A false end-point is thus obtained, which recurs persistently. As much as 7 to 10 c.c. of *N*/10 thiosulphate must be sometimes added over a period of about 30 min. after the titration is apparently finished. We have therefore substantially altered the process and adopted the following procedure. About 1 g. of the cresol is weighed into a 200 c.c. graduated flask; 5 c.c. of 2*N* sodium hydroxide solution is added and the liquid diluted to the mark with distilled water. 10 c.c. of this solution is placed in a well-stoppered flask of about 300 c.c. capacity. To this is added an excess

(100 c.c.) of a mixture of equal parts of *N*/20 potassium bromide and *N*/100 potassium bromate, followed by 10 c.c. of hydrochloric acid (sp. gr. 1.08). The flask is closed and sealed by running a little water round the stopper and shaken for exactly one minute. The stopper is then removed only far enough to allow the addition of 20 c.c. of a 10% potassium iodide solution, the latter being introduced in such a manner that throughout the addition the liquid always forms a seal round the stopper. These precautions prevent loss of bromine vapour, and thus remove a serious source of error in the assay. The flask is placed aside for one hour and the liberated iodine is titrated. Towards the end of the titration 10 c.c. of carbon tetrachloride is introduced. This has been found desirable in order to produce a sharp end-point, and is due to the solution of the mixture of bromocresol and iodine in the carbon tetrachloride. As a result of numerous tests we find that *m*-cresol takes up 480 g. of bromine per 108 g. of cresol, this being nearly the theoretical quantity. On the other hand, *o*- and *p*-cresols require 333 and 332 g. of bromine respectively per 108 g. of cresol. Calculating to bromine absorbed per gram, 1 g. of *m*-cresol is equivalent to 4.444 g. of bromine, and 1 g. of *o*- and *p*-cresol are respectively equivalent to 3.080 g. of bromine very nearly. If *X* = % *m*-cresol and *Br* = bromine actually absorbed, the percentage of cresol is given by

$$X = (100 \text{ Br} - 308) \div 1.364.$$

The results obtained with synthetic mixtures containing *m*-cresol up to 50% are within 2% of the actual amounts present. With larger percentages of *m*-cresol (80% or so) the results are from 3 to 4% too low, and in such cases it is better to add *o*-cresol to the original cresylic acid in order to bring the percentage of *m*-cresol down to about 50%. With the modifications given above this bromine method is very rapid and sufficiently accurate for general work. The bromine factors for *o*- and *p*-cresols are empirical, but it was found that they were valid over a large range of synthetic mixtures, whilst the theoretical factors gave serious discrepancies, except with cresylic acids in which *m*-cresol is present to the extent of 80% or more.

We are now in a position to utilise these three methods for determining *m*-cresol in cresylic acid, and by ascertaining the sp. gr. accurately at 15.5°/15.5° C. we have all the data necessary for determining the *o*-cresol and the *p*-cresol actually present. The only assumption made is that the sp. gr. of a mixture of the three cresols is additive to the fourth decimal place, and this will be found to be true, or very nearly so, in all cases. Thus sp. gr. 1.0433 was found instead of 1.0434 calculated, for a mixture of cresols containing 5.4% phenol and 54.0% *m*-cresol, and 1.0434 was found for 1.0434 calculated for cresols containing no phenol and 31.5% *m*-cresol. The values for the sp. gr. of the constituents used by us for this purpose are 1.0774 for phenol, 1.0516 for *o*-cresol, 1.0388 for *m*-cresol and *p*-cresol; and the formula connecting specific gravity and composition is

$$G = [1.0774 \times P + 1.0388 \times M + 1.0516 \times O + 1.0388 (100 - P - M - O)] \div 100.$$

In this equation, *G* is the observed specific gravity at 15.5°/15.5° C.; *P*, *M*, *O*, and (100 - *P* - *M* - *O*) are the percentages of phenol, *m*-cresol, *o*-cresol, *p*-cresol respectively.

This expression simplifies to 0.0386 *P* + 0.0128 *O* = 100 (*G* - 1.0388), in which *O*, the proportion of *o*-cresol, is the only unknown quantity. If no phenol is present, the equation is simply 0.0128 *O* = 100 (*G* - 1.0388).

The last equation applies to all mixtures of the three cresols, such as "tri-cresol" and commercial *m*-cresol. The following table gives a few results

that we have obtained by the utilisation of the aforementioned equations:—

	1	2	3	4	5	6	7	8
Phenol	9.7 (10.0)	4.6 (5.1)	1.1	11.0	20.0	16.6	nil	3.4
<i>o</i> -Cresol	54.6 (55.4)	35.2 (33.4)	nil	11.3	32.1	31.2	37.8	28.0
<i>m</i> -Cresol	15.1 (17.1)	30.1 (30.8)	72.0	44.3	11.7	20.2	16.7	33.5
<i>p</i> -Cresol	20.6 (17.5)	30.1 (30.7)	24.4	18.5	29.4	17.3	45.5	35.1
Xylenols	—	—	—	14.9	6.8	14.7	nil	nil

(1) and (2) are synthetic mixtures, the actual composition being given in brackets. (3) was purchased as "80% meta-cresol," (4) is a "mixed" cresylic acid consisting of a mixture of about two hundred samples of cresylic acid from which the phenol had been stated to have been removed by the manufacturers, (5) and (6) are samples of cresylic acid containing comparatively large amounts of phenol. (7) is a sample of "tri-cresol" and (8) a colourless cresol free from xylenols. The deficiency in (3) is due to water. The "xylenols" consisted of the fraction which, on re-distillation with a Raschig column, did not distil below 205° C. In general the xylenols so separated had sp. gr. 1.0326 at 15.5°/15.5° C. The cresylic acids from which (4) was derived were collected about four years ago, and were all supposed to contain less than 10% phenol. So far as could be traced they included no tar acids of blast furnace origin. We have found that the *o*-cresol result may be as much as 3% low when much phenol is present, and within 2% of the actual quantity in the absence of phenol. This, of course, tends to make the *p*-cresol result too high, since it is taken by difference.

We have applied the method to ascertain the composition of distillates from cresylic acid fractionated out up to 195° C., some of the results being the following:—

	Phenol.	<i>o</i> -Cresol.	<i>m</i> -Cresol.	<i>p</i> -Cresol.	Sp. gr., 15.5° 15.5° C.
1	24.8	39.1	17.9	18.2	1.0534
2	—	64.1	16.5	19.4	1.0470
3	—	68.0	15.1	16.9	1.0475
4	—	72.1	14.1	13.8	1.0480

This table refers to distillates obtained by adding ortho-cresol to cresylic acid and distilling the mixture to 195° C. preparatory to determining phenol (J., 1917, 842). From these and other results we have found that this fraction of cresylic acid nearly always contains from 65% to 75% of *o*-cresol and, if phenol is present also, the total proportion of phenol and *o*-cresol in the distillate is also from 65% to 75%.

Approximate results for the *o*- and *p*-cresols can be obtained by observing the refractive index of the mixture of cresols at 50° C. by means of the Abbe refractometer. The refractive indices used by us for this purpose are $n_D = 1.5309$ for *o*-cresol, 1.5266 for *m*-cresol, and 1.5260 for *p*-cresol. The formula connecting n_D and the percentage is given by $O = (100 n_D - 0.0006 M - 152.6) \div 0.0049$, in which $O = \%$ *o*-cresol, $M = \%$ *m*-cresol, and n_D is the observed refractive index at 50° C. The latter can be determined at any two convenient temperatures and the necessary correction for 50° C. applied. Thus 36.3% *o*-cresol, 31.5% *m*-cresol and 32.2% *p*-cresol gave a reading of 1.5277, the calculated reading being 1.5279. Taken in conjunction with the third method for assaying *m*-cresol previously described, this procedure is very useful when only approximate results are required, owing to its rapidity. It cannot, however, be employed with advantage unless a large proportion of the mixture consists of *o*-cresol, because the refractive indices of *m*-cresol and *p*-cresol are too close to each other. This condition

can always be secured by adding 40% of *o*-cresol to the original cresylic acid.

We take this opportunity of commenting upon the criticism of our curves by Dawson and Mountford made in the footnote to their paper (J. Chem. Soc., 1918, 113, 923. See also our paper, J., 1918, 265 r). The statement that our *m*-cresol contains 15% of *p*-cresol is without foundation, as it assayed 99.0% by Raschig's process, 100.3% by the first bromination process, and only slightly under 100% by the second bromination process, but it froze at a lower temperature than the *m*-cresol employed by Dawson and Mountford. With *m*-cresol freezing at +10.4° C. we have obtained curves higher than our original curves for *m*-cresol and the other phenols. We also found a eutectic point on our *m*-cresol and *p*-cresol curve with 12% concentration of *p*-cresol and at +0.15° C., which we failed to observe with *m*-cresol of lower freezing point. Our original curves must be corrected for *m*-cresol of freezing point +10.4° C., but the results for the lower freezing *m*-cresol still hold good. We have also repeated our observations for the pairs, phenol-*p*-cresol and *o*-cresol-*p*-cresol, attention being paid more particularly to the eutectic points shown in our previous paper. These observations confirm our previous results. As our method of determining "freezing" points, or to be more precise, depression of freezing point, is a cryoscopic one, it cannot be expected to give the same results as those obtained by Dawson and Mountford, who determine the temperature at which complete solution occurs. It is well known (e.g., the determination of the "titre" of fatty acid mixtures) that the melting point or temperature of disappearance of all solid matter in a mixture of closely related substances is higher than that of the freezing point or "titre." As a matter of fact, pure cresol mixtures are even more complicated than the curves indicate, for at least one other set of curves can be obtained in the following manner. The mixture of the cresols is cooled, and the lowering of the freezing point is determined in the usual way (a); the mixture is then melted in the same apparatus and the temperature adjusted till it is just above that of (a). A crystal of the main constituent is then introduced, and the mixture well stirred, when the temperature will be found to rise to a second maximum (b) and then fall to the temperature of the external cooling bath. The solids separating in (a) and (b) are different; in (a) the liquid is filled with a heavy deposit or shower of crystals, whilst in (b) only a few crystals separate. The phenomenon recalls that described by Miers and Isaac (Proc. Royal Soc., A, 1907, 79, 322) for mixtures of betol and salol. More than 4° C. difference between the two curves may sometimes be observed, but we have determined only a few points on this second curve and have not followed it further.

It should be noted that we have confined our investigations to such cresylic acid as can be obtained commercially, and have not made any special examination of cresylic acid from different kinds of tar. We have, however, as far as possible excluded tar acids of blast furnace origin.

We desire to express our indebtedness to Sir J. J. Dobbie, F.R.S., for permission to publish the results in this paper.

Government Laboratory.

10th May, 1920.

THE VISCOSITY OF SOLUTIONS OF NITRO-CELLULOSE IN ETHER ALCOHOL.

BY WILLIAM HOWIESON GIBSON AND ROBERT McCALL.

In the manufacture of cordite R.D.B., which was extensively employed as a propellant during the war, the gelatinisation of the nitrocellulose is effected by means of a mixed solvent of ether and

alcohol. Owing to difficulties which were experienced in the early stages of manufacture, an investigation was made into the viscosity relationships of cellulose and nitrocellulose solutions and standardised conditions were arrived at.

The present paper deals with the observations made on the viscosity of nitrocellulose in ether-alcohol and the influences affecting the viscosity of such solutions. It has been demonstrated that when suitable conditions are chosen the viscosity of an ether-alcohol solution is characteristic of the nitrocellulose used, and is therefore a useful criterion in the examination of nitrocellulose. All the viscosity determinations referred to in this paper were made by means of the Falling Sphere viscosimeter, as described by Gibson and Jacobs (*Chem. Soc. Trans.*, 1920, 117, 473). This method is very suitable for use with viscous solutions such as are obtained with fairly strong concentrations of nitrocellulose.*

Initially solutions of nitrocellulose in ether-alcohol for viscosity determinations were obtained by dissolving 4 g. of dry nitrocellulose in 100 c.c. of the mixed solvent ether and alcohol, solution being facilitated by mechanical shaking. At the Research Department, Woolwich, the ratio of ether to alcohol used in the mixed solvent was 3:2 by volume, and in factories the ratio used was 3:3.75. In both cases the specific gravity of the solvent constituents was the same, being, ether 0.72 and alcohol 0.817 (92% by wt., approx.) at 15°/15° C.

Usually it was found that solutions of nitrocellulose in the factory solvent gave viscosity values about 70% of those obtained in the Woolwich solvent. This, however, was not invariably the case, for sometimes the factory method yielded values much higher than the Woolwich method, while in other cases the values were almost identical by the two methods. The following are a few typical examples:—

TABLE I.

Source of nitrocellulose.	Viscosity, Woolwich.	Viscosity, Factory.
Queensferry	3.33	3.61
Gretna blend 1076 ..	87.32	75.2
do. 1094 ..	79.6	94.1

Such variation in the values for viscosity evidently called for some explanation, as did also the peculiar results obtained in the determination of the viscosity of several nitrocelluloses of high nitrogen content. Thus it was observed that the viscosity of some of these did not show a constant value after the solution had assumed the temperature of the bath. The time of fall of the ball increased, and in some cases the solutions eventually became so viscous that the ball would not fall through them. The solution at this stage had set to a stiff jelly.

Other solutions which had shown a rise had a gelatinous appearance. Further, it was always those solutions which had a jelly-like appearance that gave this rise in viscosity on standing. In some cases the solutions were already quite gelatinous when removed from the shaker, and no viscosity determinations were possible.

To arrive at a better understanding of these facts, a study was made of the effects of varying the proportion of ether to alcohol in the solvent.

Viscosity determinations with varying proportions of ether-alcohol.

The nitrocotton used was a soluble nitrocellulose with a nitrogen content of 12.2%, as used for R.D.B. manufacture at Waltham Abbey. The nitrocotton was dried in a steam oven for 1½ hrs. at 95° C. and cooled for 15 mins. in a vacuum desiccator before weighing, an operation which was performed within a period of two minutes on account of the hygroscopicity of the nitrocellulose.

The weighed nitrocotton was placed in a stout bottle of about 250 c.c. capacity and the alcohol added; the whole was well shaken and allowed to stand for about 15 mins., and then the ether was added. The bottle, with its contents, was then vigorously shaken for a few minutes and placed in a shaking machine overnight. The viscosity was determined by the falling sphere method at 20° C., about an hour being allowed for the solution to attain equilibrium. The following table gives the times of fall of the sphere with these solutions:—

TABLE II.

Viscosity of 4 g. nitrocellulose (12.2% N) in 100 c.c. of ether-alcohol.

Ratio ether : alcohol. (vol.).	Viscosity, (time in secs.).
80 : 20	160.7
75 : 25	59.6
70 : 30	39.9
60 : 40	28.8
50 : 50	23.1
45 : 55	26.1
40 : 60	30.5
30 : 70	90.4

These results are shown on Graph I. (B). The curve is a striking one. It will be seen that a minimum viscosity value is obtained when the ratio of ether to alcohol is approximately 55:45 by volume or about 1:1:1 by weight. As the ratio of ether to alcohol is increased or decreased the viscosity rises until it reaches a maximum at 80:20 on the one hand and 30:70 on the other. With ratios outside these values the nitrocotton was not dissolved.

The range of mixtures of ether-alcohol, 100 c.c. of which would dissolve 4 g. of this nitrocotton, is thus between 80:20 and 30:70. The optimum solvent for the nitrocotton was that containing the ether and alcohol in the proportion 55:45 by volume, gel formation taking place to a greater or less extent according as the solvent used was removed from, or approached that composition. The solutions obtained with the 80:20 and the 30:70 solvents had a distinctly jellied appearance. The time of fall of the ball increased in both cases on standing, and eventually the solution became so viscous that the ball would not fall. The values given are those obtained from the first ball dropped after the solution shaken over-night had stood for 1 hour at 20° C.

It is important to note that the rise in viscosity occurred at both ends of the curve, excluding the possibility that the rise was due to loss of ether during the experiment. The effect of a slight loss of ether might account for a rise at the alcohol-rich end of the curve, but at the ether-rich end would produce a fall.

In one case, with a solvent rich in alcohol, the time of fall of the ball changed in the manner shown in Table III.

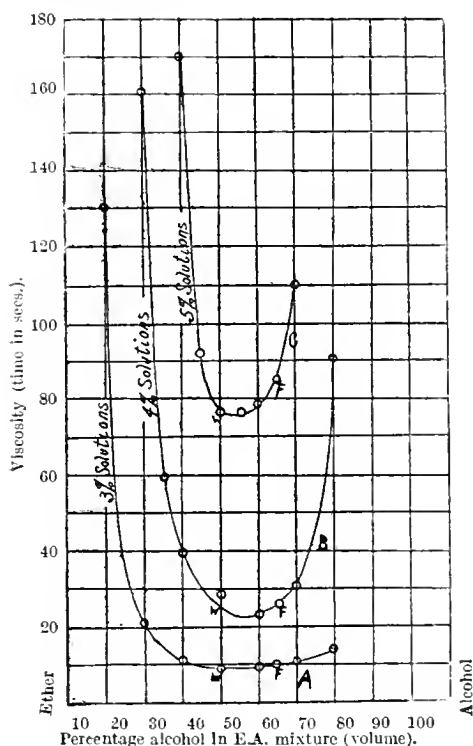
The initial period was taken as 20 mins. after the solution had been transferred to the viscosimeter tube after having been shaken over-night for 14 hrs.

* In the cases in which the absolute viscosity has not been calculated, the time of fall in seconds of a $\frac{1}{16}$ -inch ball through 15 cm. has been given; this is almost proportional to the viscosity, as the density changes are small.

TABLE III.
Alteration of viscosity with time.

Period. Mins.	Viscosity, time of fall (secs.).
15	102.5
18	112.2
21	117.0
26	123.7
70	167.5
101	230.0
370	801.0
550	very long.

In another case, with solvent rich in ether, the time of fall of the ball increased from an initial value of 557 secs. to a value of 619 secs. in a period of 8 hrs. at 20° C. A determination of the amount of solvent lost in that time (by weighing) gave a loss of only 0.02 g., showing that the concentration had not appreciably altered. These experimental facts are evidence of actual gelation having taken place in the solution.



GRAPH 1.

Solutions of nitrocellulose. Waltham Abbey R.D.B. in ether—alcohol. The values W and F are those obtained from the Woolwich and factory solvent respectively.

The experiments just described indicated that the proportion of ether to alcohol played an important part in the determination of the viscosity, and also that with excess of either constituent variable results are obtained. Both of these subjects were therefore further investigated.

Effect of concentration of nitrocellulose.

In order to ascertain to what extent the viscosity ether-alcohol curve would be influenced by changes in concentration, the viscosity curves of 3%, 5%, and 6% solutions were determined, with the results given in Table IV. The procedure in

making up the solutions was the same as in the case of the 4% solutions.

These results are represented in Graph I., curves A, C, and D.

The curves obtained were all similar in form to that of the 4% solutions. The chief point of interest lies in the fact that the minimum viscosity value was always obtained when the ratio of ether to alcohol was approximately 55:45, showing that concentration has little or no influence on the minimum point.

Further, as the concentration increased the range of ether-alcohol mixtures which would dissolve the nitrocellulose diminished, and formation of the gel took place more abundantly and readily.

TABLE IV.

Ratio. Ether:alcohol (vol.).	Viscosity (time in seconds).		
	3 g.*	5 g.*	6 g.*
90 : 10	130.2	—	—
80 : 20	21.1	—	—
70 : 30	10.8	170	—
65 : 35	—	92.0	—
60 : 40	9.2	76.4	260.4
55 : 45	—	76.5	250
50 : 50	9.3	78.5	260
45 : 55	—	85.0	299
40 : 60	10.6	110	—
30 : 70	13.8	—	—

* Weight of nitrocellulose per 100 c.c. of ether-alcohol.

Table V. gives the viscosity values for each concentration and optimum solvent. The concentration by weight was calculated in each case.

TABLE V.

Relation between concentration of nitrocellulose and optimum solvent.

Concentration of nitrocellulose solutions.		Viscosity (secs.).	Log T.
g. per 100 c.c.	g. per 100 g.		
3	3.93	9	0.9542
4	5.22	24	1.3802
5	6.55	76	1.8808
6	7.86	250	2.3979

On plotting the logarithm of the time against the concentration by weight the curve obtained is a straight line.

Arrhenius (Biochem. J., Aug., 1917) obtained a very similar relationship from Baker's measurements of the viscosities of nitrocellulose in acetone solution (J. Chem. Soc., 1913, 1653). Arrhenius' formula, which has some theoretical foundation, may be expressed: $\log \eta = \theta C - \log \eta_0$, where η and η_0 are the viscosities of solution and solvent respectively and C is the concentration of the nitrocellulose; θ is a number which proved to be approximately constant for a narrow range of concentrations.

Influence of the nitrogen content of the nitrocellulose on the ether-alcohol viscosity curve

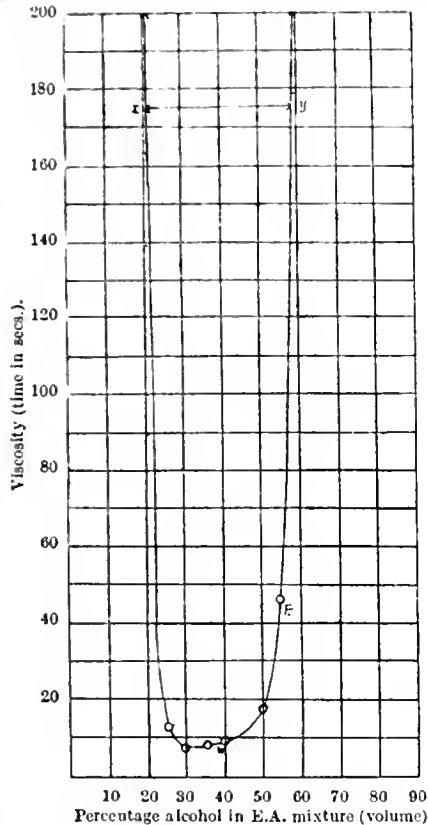
Having established that the minimum point of the viscosity ether-alcohol ratio curve was not affected by changes in concentration of the nitrocellulose, it was necessary to determine to what extent it might be influenced by a change in the nitrogen content of the nitrocellulose. Determinations were according made on nitrocelluloses containing 12.55% and 11.8% nitrogen respectively with the following results. Solutions containing

4 g. of nitrocellulose in 100 c.c. of solvent were used in each case:—

TABLE VI.
Nitrocellulose of 12.55% nitrogen content.

Ratio. Ether: alcohol (vol.).	Viscosity (secs.) of nitrocellulose with	
	12.55% N.	11.8% N.
90 : 10	—	Very long.
80 : 20	Very long.	198
75 : 25	12.9	—
70 : 30	7.6	45
65 : 35	8.5	—
60 : 40	9.0	34.6
50 : 50	17.2	32.2
40 : 60	Very long.	33.2
30 : 70	—	37.6

Some of the above results are represented in Graph II.



GRAPH II.
Nitrocellulose of nitrogen content 12.55%.
4 g. N.C. per 100 c.c. E.A.

The minimum viscosity and optimum ether-alcohol ratio of the solvent for each nitrocellulose, as read from the curves, are as follows:—

TABLE VII.
Optimum solvent with varying nitrogen content of nitrocellulose.

Nitrogen content, %	Min. viscosity (secs.).	Optimum ether-alcohol ratio.	
		By vol.	By wt. (approx.)
11.8	31.8	50 : 50	0.9 : 1
12.2	23.1	53 : 47	1.0 : 1
12.55	7.6	70 : 30	2.1 : 1

It is established from these results that the optimum solvent for a nitrocellulose depends on its

nitrogen content, and that the higher the nitrogen content, the more ether the optimum solvent requires.

It will be seen that in the case of the nitrocellulose containing 12.55% N, the range of ether-alcohol mixtures which dissolve it is much smaller than that of the nitrocellulose with 12.2% N, and this range again is less than with 11.8% N. It might be expressed as a value, namely, the range of composition for the ether-alcohol mixture which will give measurable viscosity at given concentration and temperature. For one concentration it would be represented by the line XY on Graph II.

Conclusions.—With a given nitrocellulose, the viscosity of its solution in ether-alcohol is dependent upon the ratio of ether to alcohol, and reaches a minimum value at a definite ratio. On either side of this "optimum" ratio the viscosity rises with increasing steepness; it was owing to this steepness that the former Woolwich method and the "factory" method, in both of which solvents diverging considerably from the optimum compositions were used, gave such irregular results.

Within the limits studied the optimum ratio of ether to alcohol does not depend upon the concentration of nitrocellulose in solution; on the other hand it is a characteristic property of the particular nitrocellulose, and its determination can be made repeatedly with concordant results. As far as can be seen at present the governing factor is the nitrogen content.

The minimum viscosity of a given sample of nitrocellulose reached in the solvent of optimum composition is again a definite and characteristic property, and repetitions of its measurement can be made with very satisfactory agreement. The value of this minimum viscosity is affected greatly by the origin and treatment of the nitrocellulose, and so the determination of this minimum value affords a guide to uniformity in manufacture.

ADDENDUM.

The influence of nitroglycerin on the viscosity of solutions of nitrocellulose in ether-alcohol.

It seemed possible that the presence of nitroglycerin in cordite might modify the action of ether-alcohol solvents in such a way as to alter the relationships which have been described above, and consequently experiments were carried out to test this question.

The viscosity values have in most cases been expressed in absolute units; the times of fall of a $\frac{1}{16}$ -in. steel ball through 15 cm. of the solutions in a 2 cm. tube have also been quoted.

The nitrocellulose used was made at Waltham Abbey for cordite R.D.B., and the ether and alcohol were of the same sp. gr. as before.

The nitroglycerin was extracted from dynamite. Its density at 20° C. was 1.60. Its viscosity, as determined with the Ostwald viscosimeter, was 0.3774 C.G.S. unit at 20° C.; this value is nearly 40 times the viscosity of water, and about 1/22 that of glycerin at the same temperature.

In the first place the nitroglycerin and ether-alcohol were taken in the proportion in which they occur in the manufacture of cordite R.D.B.

Three ether-alcohol mixtures were used, namely, 70:30, 50:50, and 30:70 by volume. The composition of the solutions was therefore as follows:—

TABLE VIII.

Effect of nitroglycerin in large proportion.

Solution.	Ratio ether-alcohol (vol.).	Wt. of ether-alcohol mixture.	Wt. of nitro-glycerin.	Wt. of nitro-cotton.
1	70 : 30	g. 57.3	g. 42.7	g. 5
2	50 : 50	57.3	42.7	5
3	30 : 70	57.3	42.7	5

In the case of solution 3 the nitrocellulose was not dissolved. Solutions 1 and 2 were too viscous for any measurement to be made, although qualitatively it was obvious that solution 1 was more viscous than 2. Both solutions were very much more viscous than 5% solutions of nitrocotton in ether-alcohol alone.

The following were the values obtained with 5% solutions by weight of the nitrocellulose in ether-alcohol.

TABLE IX.

Viscosity of the nitrocellulose for nitroglycerin experiments.

Ratio ether-alcohol (vol.).	Time of fall (secs.)	Density.	Viscosity (absolute).
70 : 30	23.8	0.78	12.59
60 : 40	19.2	0.79	10.14
55 : 45	20.6	0.80	10.86
50 : 50	21.4	0.80	11.29
40 : 60	36.3	0.81	14.11
30 : 70	78.2	0.82	41.44

The curve obtained on plotting the above results is similar in form to those in fig. 1, and has a minimum point when the ratio of ether-alcohol is 57.5:42.5.

The effect of replacing part of the ether-alcohol in 5% solutions of nitrocellulose by varying amounts of nitroglycerin was examined. In preparing the solutions the nitroglycerin was added to the ether, which was then poured on to the alcohol-wet nitrocotton. Otherwise the procedure was the same as in the case of nitrocellulose.

The following were the values obtained, together with values for solutions in which solvent was substituted to the extent of 10, 12.5, and 20% of nitroglycerin.

TABLE X.

Effect on nitroglycerin.

	Ratio of ether-alcohol (vol.).	Time of fall (secs.).	Density.	Viscosity.
Nitro-glycerin, 5.33 g., ether-alcohol, 94.67 g.	70 : 30	39.4	0.80	20.78
	60 : 40	30.4	0.81	16.02
	55 : 45	30.6	0.82	16.09
	50 : 50	34.3	0.82	18.04
Nitro-glycerin, 10 g., ether-alcohol, 90 g.	40 : 60	58.0	0.83	30.46
	70 : 30	42.1	0.82	22.14
	60 : 40	32.8	0.83	17.23
	55 : 45	34.1	0.84	17.88
Nitro-glycerin, 12.5 g., ether-alcohol, 87.5 g.	50 : 50	39.9	0.84	20.92
	40 : 60	72.0	0.85	37.70
	65 : 35	52.0	0.84	27.79
	60 : 40	49.4	0.85	25.86
Nitro-glycerin, 15 g., et.-alc., 85 g.	55 : 45	51.4	0.85	26.92
	50 : 50	55.8	0.86	29.22
	60 : 40	66.8	0.86	34.93
	55 : 45	68.4	0.86	35.77
Nitro-glycerin, 20 g., et.-alc., 80 g.	50 : 50	83.8	0.87	43.64
	70 : 30	47.0	0.87	24.53
	60 : 40	181	0.88	83.92
	55 : 45	153	0.88	79.77
	50 : 50	166.6	0.89	86.72

The viscosity-ether-alcohol curves are normal. The minimum viscosity occurred when the ratio of ether to alcohol was 57.5:42.5, as in the case of 5%

solutions of nitrocotton alone. The curves, however, lie at a higher level, and the range of solubility is diminished.

In the case of the results with 20% of nitroglycerin the time of fall of the ball was greater after standing in every case. The times chosen are those of the first fall of the ball after the solution had stood for 2 hours at 20° C.

The chief point of interest lies in the fact that that ratio of ether to alcohol which gives the minimum was not altered by increasing the amount of nitroglycerin. As the amount of substituted nitroglycerin was increased the curves ascended to a higher level and the range of solubility was diminished. The effect was reminiscent of, but greater than, that produced by increasing the proportion of nitrocellulose present in ether-alcohol solution.

A further experiment was carried out in which varying quantities of nitroglycerin were added to 5% solutions (by weight) of the same nitrocellulose in ether-alcohol. The ratio of ether to alcohol was 57.5:42.5, this being the optimum solvent for that nitrocellulose. As in the previous experiment, the additions of nitroglycerin were made to the ether before it was added to the alcohol-wet nitrocotton.

Table XI. shows the results obtained.

TABLE XI.

Solutions of 5 g. nitrocotton in 100 g. ether-alcohol used.

Nitro-glycerin added.	Time of fall (secs.).	Density.	Viscosity.
g.			
0	23.2	0.788	12.26
2.5	23.3	0.802	12.29
5.0	23.6	0.808	12.42
7.5	23.2	0.816	12.20
10.0	24.0	0.829	12.60
12.5	24.9	0.839	13.05
15.0	25.5	0.844	13.37
20.0	25.8	0.865	13.49

After an addition of 10 g. of nitroglycerin a slight rise takes place.

Conclusions.—The optimum composition of an ether-alcohol solvent for a nitrocellulose is not affected by the addition of nitroglycerin.

Substitution by nitroglycerin of a proportion of the ether-alcohol solvent in a solution of nitrocellulose results in a solution of viscosity approximately equal to that of a similar solution in which the equivalent volume of solvent is absent. This holds only for small substitutions.

Nitroglycerin does not act as a solvent when added to solutions of nitrocellulose in ether-alcohol, the viscosity of the solution tending to be increased rather than decreased by such addition.

This work was carried out at the Research Dept., Royal Arsenal, Woolwich, and is published by permission of the Director of Artillery, War Office, to whom our thanks are due. We also desire to express our thanks to Lieut.-Col. R. A. Craig, C.M.G., the Superintendent of Research, and to Sir Robert Robertson, K.B.E., F.R.S., the Superintending Chemist, for the interest they have taken in this work.

Research Dept.
Royal Arsenal.

Birmingham Section.

Meeting held on March 11, 1920.

MR. L. P. WILSON IN THE CHAIR.

CATALYTIC ACTION IN THE OXIDATION OF CELLULOSE.

BY L. P. WILSON, F.C.C.I., F.I.C.

In the manufacture of artificial cellulose fibres from cellulose, the most commonly employed method is the viscose process, in which cellulose sodium xanthate is produced and dissolved in dilute alkali to form a solution. This solution is projected through fine apertures into a coagulating medium, and so is formed into filaments.

The first stage in the production of a solution, whether the starting material be wood or cotton is the formation of alkali cellulose by immersion in a solution of caustic soda of over 15% strength; this causes a partial gelatinisation of the fibres and the formation of a definite chemical compound which, after a process of oxidation and subsequent reaction with carbon bisulphide, forms sodium cellulose xanthate, a brownish plastic solid, which is soluble in water, forming a liquid of high viscosity.

Whether or not the cellulose xanthate will form a solution which can be used for the production of threads depends largely on the exact amount of oxidising action to which the alkali cellulose is submitted; this is in any case very small, but it causes an alteration in the molecular arrangement of the cellulose, and determines whether the xanthate when mixed with water is a stiff gel or a liquid less viscous than glycerin; the chemical difference between these two conditions cannot be stated, but the physical differences are most marked.

This oxidation has been generally effected by exposure to the atmosphere, and at the ordinary temperature—say 20° C.—the process may take four days. If oxygen is introduced by other means—such as by treatment with peroxides or hypochlorite—this time may be reduced in proportion to the activity of the oxidising agent employed. For instance, 1% of sodium peroxide added to the caustic soda solution used to produce the alkali cellulose results in a sufficient oxidation taking place during a steeping period of about 5 hrs.

Whichever method of oxidation is employed—exposing to air after the formation of alkali cellulose, or oxidising while still in the caustic soda solution—the process can be greatly accelerated by the use of suitable catalysts. These catalysts are generally oxides or hydroxides of metals which exist in two states of oxidation, e.g., iron, nickel, cobalt, cerium, vanadium, manganese. The catalyst is applied either by first impregnating the cellulose with a salt of the metal, or by suspending the hydrated oxide in a fine state of division in the caustic soda solution; as an example, sheets of wood pulp may be soaked in a solution of nickel sulphate containing 0.33% nickel oxide and then pressed to such a weight that they contain 0.25% of nickel oxide and dried. The sheets of pulp are then steeped in caustic soda of the usual strength, containing in addition about 0.8% of sodium peroxide. Immersion for $\frac{1}{2}$ hr. under these circumstances will produce the required change in the cellulose, which would have taken four or five hours without the catalyst to accelerate the oxidation. The same sort of effect is obtained by using iron solutions of suitable strength and exposing the alkali cellulose to atmospheric oxidation. In this case 0.1% of FeO will accelerate the process, so that it takes about one-fourth of the normal time. The amount of change which takes place, and which is indicated by the viscosity of the xanthate, is proportional to the

amount of oxygen absorbed, and does not depend on the state of the oxygen, i.e., whether it be free atmospheric oxygen or combined, for example, either as hypochlorite or peroxide. As far as is known, these variations only affect the rate of reaction, which is, of course, greatly dependent on the presence, nature, and quantity of catalyst, if any, employed.

The effect of the catalyst in a heterogeneous system of this type depends, to a large extent, on its distribution and its fineness of division, so that it is difficult to give accurate relative values for different metals. The following figures, however, indicate the comparative effects of a number of metals—applied under as nearly as possible equal conditions—in modifying the molecular state of the cellulose:—Without catalyst, 100; nickel, 9; vanadium, 22; cobalt, 28; iron, 33; cerium, 42; manganese, 60; chromium, 77; lead, 200; manganese and lead, 45. It will be observed from these figures that lead has a negative value, giving a figure greater than the blank. When, however, it is used in conjunction with manganese, the effect of the two together is greater than that of manganese alone. Apparently, therefore, lead, although not an oxidising catalyst when used by itself, acts as a catalytic accelerator in conjunction with manganese.

DISCUSSION.

Dr. E. B. MAXTED observed that in the hydrogenation of oils it was easier to measure the absorption of hydrogen than to ascertain the iodine value of the oil. With the rate of the shaker constant the curves were quite smooth. The method might be applied to the oxidation of cellulose.

Dr. D. F. TWISS enquired concerning the evidence as to the relationship between viscosity and oxidation; with rubber there was no such simple connection. On what experiments was the assumption as to the proportionality between the extent of chemical action and that of the physical change in cellulose actually based?

Mr. WILSON pointed out that the difference in viscosity was the measure, more or less direct, of the molecular change of the cellulose, and that change had been correlated with the actual absorption of oxygen, and the corresponding viscosities had been obtained in a parallel set of experiments on a larger scale. The figures, while not the actual direct measure of the activity, were fair in a comparative sense, and placed the metals approximately in the right order. The fineness of division of the metallic compounds had considerable effect. The method employed was similar to that suggested by Dr. Maxted. It was not necessary in the case of cellulose to have shaking, because the material was very open and exposed a large surface to the action of the oxygen.

London Section.

Meeting held at Burlington House on April 12, 1920.

MR. JULIAN L. BAKER IN THE CHAIR.

THE FERTILISING VALUE OF SEWAGE SLUDGES.

BY WINIFRED E. BRENCHELEY AND E. HANNAFORD RICHARDS.

(Rothamsted Experiment Station.)

The sewage sludges produced by the usual methods of tank treatment have never been highly valued as manure. As a rule farmers within easy reach of the works have been willing to take the sludge when they could get it without payment,

but in many places the local authorities have had to pay for its disposal by burial, burning, or other means. In a few cases where the sludge is prepared specially for sale as a fertiliser and is put on the market in a convenient form, no difficulty is experienced in disposing of it at a price which at any rate pays the cost of manufacture.

That the farmers are right in their low estimate of the manurial value of sewage sludge in the forms generally available is shown by the results of many field trials designed to test the fertilising constituents of sludge in comparison with equivalent amounts of artificial manures. The most complete series of trials was made under the auspices of the Royal Commission on Sewage Disposal about twelve years ago.*

Seven types of sludges produced by various methods of settlement or chemical precipitation showed practically no manurial value when applied to mangolds, turnips, and hay crops on farms in England and Wales. Further, pot culture experiments with wheat carried out by Dr. Voelcker at the Woburn Station of the Royal Agricultural Society gave a maximum increase over the unmanured pot of only 15%. Some of the sludges gave negative results. The samples giving the best yields contained large amounts of lime added to facilitate pressing into cake. Dr. Voelcker reported that none of the sludges was worth 10s. per ton to the farmer.

Although there is no doubt that the nitrogen, by far the most valuable constituent in sewage sludge, is mostly present in a form very difficult of digestion by the soil organisms, it may be argued that the field trials of the kind just mentioned did not give the sewage sludges a fair chance. It is unreasonable to compare the effect of nitrogen in a substance like sewage sludge, to be obtained for the asking, with equal amounts of nitrogen in the form of ammonium sulphate, or sodium nitrate costing £21 per ton. A much better comparison can be made with farmyard manure if both sludge and dung are applied at the same rate of dry matter per acre. Thus a dressing of 10 tons of farmyard manure (20% dry matter) should be compared with 4 tons of sludge (50% dry matter). Tested in this way over a complete rotation the sewage sludge would probably show a small increase over the unmanured plots, but far below that given by the dung. Giving every possible chance to the sludge it must be admitted that it is of little value to the farmer.

Within the last ten years at least two new methods of sewage treatment have been introduced, viz., slate beds and activated sludge. Both these processes yield sludges of definite manurial value, as is clearly shown by the experiments described below.

As regards activated sludge the process as a practical method of sewage purification is hardly yet out of the experimental stage, but the claims made for its fertilising value appear to be thoroughly justified, and the present results merely confirm tests made elsewhere.†

In the case of the sludge from slate beds, although this has been recognised as superior to the ordinary tank sludges,‡ we are not aware that any quantitative tests of its manurial value have been published. This material is decidedly inferior to activated sludge as a fertiliser, but, since it is produced without the cost of air compression or agitation, and can be easily dried, it is by no means to be despised. On the other hand, it must be remembered that the slate bed effluent requires further treatment on filters in order to reach a degree

of purity comparable with that attained by the activated sludge process in one operation.

It has long been known that the suspended matter washed out from percolating filters—so-called humus—is an excellent manure. It resembles activated sludge in many ways and forms a useful "starter" for the more rapid production of this material from the suspended matter in raw sewage. One feature common to all three types of solid residue, viz., slate-bed sludge, filter "humus," and activated sludge, is that they are produced under aerobic conditions of increasing thoroughness in the order given. On the other hand, the old tank sludges tested by the Sewage Commission were all essentially anaerobic sludges, varying only in the time they had been allowed to remain on the tank bottoms from which air is practically excluded. It seems probable that the access of air combined with the presence of suitable surfaces for the deposition of colloids—another feature common to the slate bed, percolating filter, and activated sludge methods—are the factors which produce the marked increase in manurial value of these sludges compared with the older tank deposits.

Chemical analyses of sludges.

The samples of sludge used in these experiments were (1) activated sludge (Withington) and (2) slate bed sludge (Harpenden). The activated sludge was supplied by Dr. Arden from the continuous flow plant at Withington, Manchester. It was fairly well divided and in good condition for applying to the soil. The Harpenden sample came from a heap of air-dried sludge taken from the settling tanks which intercept the suspended solids washed out from the slate beds, the effluent passing on to the percolating filters. An analysis of pressed cake from precipitation tanks at Derking is added for comparison.

Sludge.	Activated. %	Slate bed%,	Precipita- tion, %.
Moisture	7.26 ..	55.65 ..	37.67 ..
Organic matter	76.72 ..	20.76 ..	24.81 ..
Inorganic matter (grit)	16.02 ..	23.59 ..	37.52 ..
Calculated on the sludges dried at 100°C.			
Nitrogen	7.09 ..	2.83 ..	0.89 ..
Phosphoric acid	3.82 ..	0.34 ..	0.66 ..
Potash	1.12 ..	0.08 ..	0.07 ..

The nitrogen is by far the most valuable constituent in the sludges. Six of the usual tank sludges tested by the Sewage Commission for manurial value contained an average of 1.22% N. The slate bed and activated sludges are from 2 to 6 times as rich.

To estimate the probable manurial effect of the nitrogen, its quality must be considered as well as its total amount. In ordinary tank sludges the bulk of the nitrogen is of an insoluble type, quite useless to plants and very difficult of digestion by the soil organisms, whose function it is to convert the insoluble residues into soluble plant food. The best way to determine the quality as distinct from the quantity of nitrogen is to see how much of it is converted into nitrate when the manure is allowed to decompose in the soil.

Nitrification experiments were made with both sludges in comparison with dried blood, the manures being added at the rate of 60 parts of nitrogen to 1,000,000 of soil.

Nitrogen as nitrates: Pts. per million of dry soil.

	At start	After 32 days	After 106 days	After 200 days
Soil only	7.3 ..	10.7 ..	17.5 ..	16.8 ..
Soil+slate-bed sludge	7.3 ..	18.0 ..	33.1 ..	33.2 ..
Soil+activated sludge	7.3 ..	32.3 ..	56.0 ..	60.9 ..
Soil+dried blood	7.3 ..	34.0 ..	77.6 ..	75.2 ..

Both the sewage sludges thus contained nitrogen in a form capable of nitrification, but there is a great difference in the proportion of the total

(1) Royal Commission on Sewage Disposal. 5th Report. Appendix VIII. (1908).

(2) Arden and Lockett. J., 1914, 523, 1122; 1915, 937. Bartow and Hatfield. J. Ind. Eng. Chem. 1916, 8, 17; Nasmyth and McKay. *ibid.*, 1918, 10, 339.

(3) Mumford, J. Bd. Agric., 1916, 23 (2).

nitrogen available in the two samples. In 15 weeks 26% of the nitrogen in Harpenden slate-bed sludge is converted into nitrate. In the same time 66% of the Withington activated sludge has been nitrified, and the action continues slowly up to 200 days. The dried blood was taken as a check on the nitrifying power of the particular soil used. It behaved normally as the whole of its nitrogen was nitrified in 15 weeks.

Evidently both kinds of sludge have manurial value. The pot culture experiments described below show that the crop increases induced by the sludges are roughly proportional to the availability of the contained nitrogen as found in the nitrification experiments.

Pot experiments were carried out in triplicate in glazed earthenware pots. The soil chosen for use was poor, in order that the response to the manures applied should be sharply marked, and, as it was rather heavy and inclined to clog, an admixture of 10% of sand was made. Each pot contained 35.4 lb. of soil, to which was added in every case 16 g. superphosphate, 8 g. potassium sulphate, and 160 g. calcium carbonate. Nitrogenous manures were then added as follows:—

Pots 37-39.—No nitrogenous manures added. (Control.)

Pots 40-42.—16 g. sodium nitrate.

Pots 43-45.—41.6 g. activated sludge, containing the same amount of nitrogen as 16 g. sodium nitrate.

Pots 46-48.—219.6 g. activated sludge, containing six times the amount of nitrogen in 16 g. sodium nitrate.

Pots 49-51.—116 g. slate-bed sludge, containing half the amount of nitrogen in 16 g. sodium nitrate.

Pots 52-54.—232 g. slate-bed sludge, containing the same amount of nitrogen as 16 g. sodium nitrate.

(Owing to the low nitrogen content of Harpenden sludge it was not possible to apply it in the proportion of 6:1 of nitrogen added as sodium nitrate.)

First crop.

Barley was sown on April 24, 1918, with seeds graded between 0.04 and 0.05 g. Ten seeds were sown per pot, but as soon as the seedlings were about 2 inches high they were thinned out, leaving the five strongest in each case. Observations were made throughout the growing period, and at the close of the experiment (August 15) the plants were cut, the ears separated from the straw, and the green weights of both recorded. The crop was then placed in a drying-room at about 90°, and, when thoroughly dry, the weights were again taken.

Growth noted.

37-39. *Control.* The plants grew well from the first, the colour of the leaves being a yellowish-green. After two months' growth they began to look unhappy as if they were starving, for the lower leaves were dying and the tips of the upper leaves were shrivelling. This phase passed in about a week, though the colour remained very pale, and very soon after this the ears began to emerge from their sheaths, earlier than in any of the other pots, all of which had received nitrogenous manure. At the time of cutting the plants were very tall and erect.

40-42. *Sodium nitrate.* Within a month from sowing these plants were stronger and darker than the controls, and before long they began to tiller strongly. The growth became very strong, the leaves were particularly broad and dark green, but the plants were succulent and rather flabby, giving the impression that they would lodge easily. As time went on they became markedly shorter than the controls and the ears were much later in emerging from their sheaths, as the nitrogenous

1st Crop—Barley.

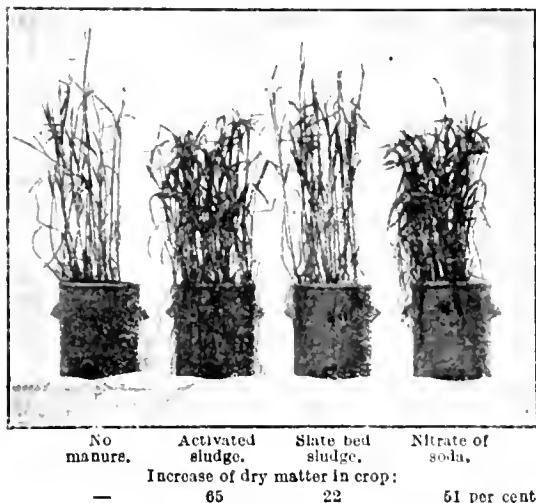


Fig. 1.

2nd Crop—Mustard.

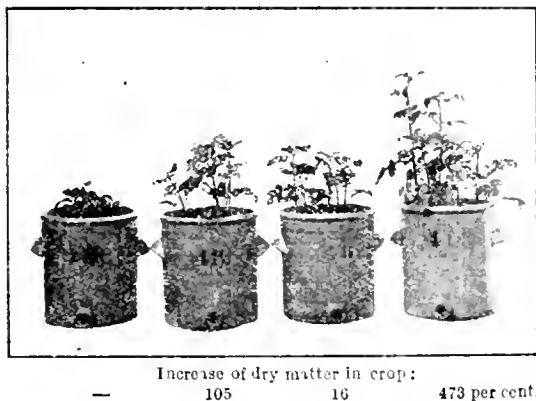


Fig. 2.

2nd Crop—Mustard.

Heavy dressings

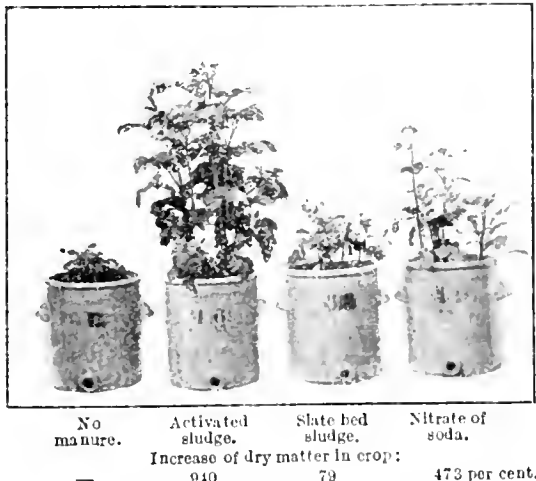


Fig. 3.

manuring tends to lengthen the period of growth and to delay maturity.

43-48. *Activated sludge.* The plants receiving an equivalent dressing of nitrogen (pots 43-45) resembled the sodium nitrate plants very closely from the beginning to the end of the experiment—so closely that it would have been difficult to draw a distinction. Those with a six-times dressing of nitrogen were also very similar, but at times during growth they gave the impression of being heavier than either those with sodium nitrate or a single dressing of sludge, an impression that was borne out by the weights after cutting.

49-54. *Slate-bed sludge.* The plants all through were in striking contrast to those grown with activated sludge. The type of growth resembled that of the controls (37-39), being upright, with few tillers and pale yellowish-green leaves, maturing much earlier than the plants with sodium nitrate or activated sludge. The Harpenden sludge plants certainly looked better than the controls, and at the time of harvesting those with the full dressing of sludge (pots 52-54) were a little shorter and darker than those with the half-dressing.

still growing vigorously, and it is certain that some considerable amount of extra dry matter would have entered into the grain if it had been possible to carry the plants on longer. The unmanured plants and the slate-bed sludge plants seemed to have reached much the same stage of development, so that while the sludge did not affect the rate of maturing or the type of growth, it caused a very considerable increase in crop, amounting to 22% of the fresh weight and 31% of the dry weight, an appreciable difference outside the limits of experimental error. From this point of view, therefore, the slate-bed sludge proved to be quite a useful source of nitrogen, although unit for unit the increase was considerably less than with sodium nitrate or activated sludge. The half dressing of sludge (pots 49-51) gave plants that were very similar to those with the full dressing, except that the proportion of dry matter was somewhat lower in both straw and ears.

Second crop (for residual effect of sludge.)

The day after the barley crop had been removed the soil in the pots was thoroughly stirred up and mustard was sown. The seeds soon germinated and

Average weights of barley plants from three pots.

	Pots.	Green weight.			Dry weight.			% increase over control dry matter.	% Dry in green.		
		Straw. g.	Ears. g.	Total g.	Straw. g.	Ears. g.	Total. g.		Straw.	Ears.	Total.
Control	37-39	56.80	20.20	77.00	24.05	11.27	35.32	—	42.44	55.78	45.86
Sodium nitrate ..	40-42	97.67	30.32	127.99	38.43	15.03	53.46	51.12	39.35	49.58	41.77
Activated sludge ..	43-45	88.88	34.82	123.70	38.30	19.90	58.20	64.77	43.09	57.15	47.04
(1 equiv.)											
Activated sludge ..	46-48	119.48	34.05	153.53	48.45	19.28	67.73	91.74	40.53	56.63	44.13
(6 equiv.)											
Slate bed sludge ..	49-51	65.18	27.18	92.36	26.95	16.05	43.00	21.75	41.35	59.04	46.44
(4 equiv.)											
Slate-bed sludge ..	52-54	69.05	25.48	94.53	30.45	16.10	46.55	31.80	44.11	63.19	49.26
(1 equiv.)											

A comparison of the average weights of plants grown with similar amounts of nitrogen applied as sodium nitrate, activated sludge, and slate-bed sludge respectively shows a remarkable agreement between the results of the first two treatments and a marked falling off in the case of the slate-bed sludge. With sodium nitrate and activated sludge the total green weights of the plants were practically the same, but the sludge plants had the heavier ears and the lighter straw. The dry weights show that a larger proportion of water is present when the nitrogen is applied as sodium nitrate, the percentage of dry matter in green being 47.0% with activated sludge and only 41.8% with nitrate. So far, then, as bulk of crop and percentage of dry matter are concerned, the activated sludge compares most favourably with the more usual nitrogenous dressing, giving what was practically an equal return for each unit of nitrogen applied. The use of six times the amount of sludge gave a further increase of crop, both in green and dry weight, but this increase was by no means commensurate with the extra expense of the very heavy dressing.

Slate-bed sludge, on the other hand, gives a less marked result, though the increase over the unmanured plots is considerable. The amount of dry matter in the ears, moreover, is higher than that in the sodium nitrate plots, but this may be partly due to the inevitable difference in maturity at the time of harvesting. The slate bed sludge plants were fully developed, and it is probable that the passing of food material into the grain had come to an end, so that the dry weight would not increase further. The ears of the sodium nitrate plants were not nearly so far advanced, the plants were

in a fortnight were thinned out to five per pot, no differences between the pots being then noticeable. The plants were grown for about three months before cutting.

37-39. *Control.* The plants did not make much growth but remained short and stocky and soon showed signs of starvation, while the lower leaves turned yellow and died off. These were the smallest plants all through the experiment.

40-42. *Sodium nitrate.* The plants soon ran ahead and became tall and sappy, outdistancing all others except those with a six-times dressing of activated sludge. Nevertheless, a fortnight before cutting the lowest leaves were turning yellow, and at the close many of these leaves had died off and a good many more just above were quite yellow. In spite of this all the upper leaves were very dark, and the plants were the only ones of the series that

43-48. *Activated sludge.* The plants with the single dressing resembled those with the single dressing of slate-bed sludge. They were rather better than controls, but the lower leaves had died off and growth was much behind that with sodium nitrate or the heavier dressing of activated sludge. In the latter case the plants were exceedingly strong from the beginning, and at the end they were hefty plants with very dark green leaves throughout, as even the lowest leaves showed hardly any trace of dying off. This set stood out markedly from all the rest.

49-54. *Slate-bed sludge.* As stated above, with the single dressing the plants resembled those with a single dressing of activated sludge, but the residual effect of the half-dressing was much less marked, and the plants did not look very much ahead of the controls.

Average weight of mustard plants from three pots.

	Pots.	Green wt. g.	Dry wt. g.	% dry in green	% In- crease in dry wt. over control
Control ...	37-39	20.53	3.23	15.64	
Sodium nitrate ..	40-42	117.07	18.52	15.64	473.4
Activated sludge (1 equiv.) ..	43-45	44.7	6.63	14.92	105.3
Activated sludge (6 equiv.) ..	46-48	200.57	33.58	11.34	939.7
Slate-bed sludge (1 equiv.) ..	49-51	28.73	3.75	12.91	16.1
Slate-bed sludge (1 equiv.) ..	52-54	42.17	5.78	13.77	79.0

From the above table and from the comparison of the growing plants it is clear that, while single dressings of slate-bed and activated sludges had a certain residual manurial value in these experiments, the actual increase of crop was not of any great significance. Sodium nitrate, however, in similar dressing left a considerable amount of available plant food, which was more than doubled with the heavy dressing of activated sludge. In the latter case the water content of the mustard plants was very high, much higher than with sodium nitrate or the lighter dressing of sludge.

From the results of the foregoing experiments it is clear that activated sludge has a very real manurial value by reason of its relatively high content of nitrogen in a readily available form. The practical difficulty of drying the sludge containing 98% of water as it comes from the settling tanks has yet to be overcome, but if an economic drying process can be found activated sewage sludge promises to become a valuable manure for the farmer and market-gardener.

There are comparatively few sewage works of any size where slate beds are in use. However, it may be worth noting that sludge from these beds has a definite manurial value and deserves the attention of farmers and gardeners wherever it is available.

DISCUSSION.

Dr. S. RIDEAL said that the most remarkable feature in the results given was the phosphorus and potash content of the Withington activated sludge. Calculated on the dry matter it contained no less than 1.12% of potash, whereas the other sludges referred to contained only 0.03%. How could the presence of this high percentage of potash be accounted for? The activated sludge also contained 3% of phosphoric acid, whilst the others contained a very small quantity. These figures might account for the fact that activated sludge was a better fertiliser than the others, quite apart from its nitrogen content. On the other hand, it was known that with all these sludges the difficulty in getting the fertilising value in an available form was largely due to the presence of oils and fats in the sludge. Perhaps distillation of the sludge would be resorted to, especially when it could be got with such a low water content as 7% as had been shown in the paper. During the war he had suggested that the soluble nitrogen of sewage might be a useful source of nitrate for explosives purposes, and he had made some calculations which showed that it would be possible to recover the soluble nitrate from filter beds if they were allowed naturally to evaporate in large covered glass areas protected from the rain—in fact, by imitating the conditions that obtained in Chili, India, and elsewhere. If it were possible to convert the whole of the nitrogen in sewage into nitrate and then evaporate the water from the filtrate, this would produce a most useful stimulant and fertiliser for plant growth.

Mr. JOHN HUGHES said that sewage sludge could be completely dried by spreading it in thin layers

and protecting it from the rain; it could then easily be ground.

Mr. E. H. SAMS said that the fertilising value of the residue from the distillation of sludge was very small; it only contained 0.7% of ammonia, and the organic matter had been destroyed. The trials carried out at Rothamsted might have been supplemented by including a sewage sludge as pumped from settling tanks on to the land.

Mr. W. T. LOCKETT agreed that it might be advantageous to subject activated sludge to some process of distillation to obtain oils, ammonium sulphate, and other compounds.

Dr. H. T. CALVERT (Ministry of Health) said that sewage sludges could be divided into half a dozen or more classes. There were chemical or precipitated sludges, sludges with trade waste, sludges with a high percentage of grease, and those with little or none. A process was in successful operation at Huddersfield in which the grease was extracted from sewage sludge by means of a solvent, and the resulting sludge was then sold as a fertiliser. This involved drying by heat, thus destroying plant seeds in the sludge, and he was hopeful that the activated sludge could be subjected to a similar process. He rather looked to that solution than to distillation. A feature of the experiments described in the paper was the large amount of activated sludge used in one pot experiment; that suggested to him that we were, perhaps, not proceeding on quite the right lines in speaking of the sludge being dependent entirely on its nitrogen content for its fertilising value. The nature of the soil ought to be taken into account, and experiments should be made in altering the nature of the soil.

Mr. RICHARDS, in reply, said that the high content of potash in the Withington sludge was certainly very striking. The figures of analyses made at Rothamsted agreed very closely with those given by Dr. Ardern as typical for this sludge. Other samples had been analysed at Withington, and the results showed that the sludge there did contain a high percentage of potash. At the same time, he was at a loss to understand why it was so high compared with other sludges. On the other hand, he thought there was some possibility of an explanation being forthcoming of the high content of phosphorus. There was a sulphur cycle and a phosphorus cycle as well as a nitrogen cycle, but very little attention had been paid to the two former, and it was possible that the phosphorus, passing from the inorganic to the organic form, might contribute in the same way as nitrogen probably did, to increase the phosphorus content of the sludge. The pot culture experiments described in the paper were only a preliminary to a fairly large piece of work which had been planned out at Rothamsted under Dr. Russell's direction. They were trying not merely to find out the fertilising value of samples of activated and other sludges, but they were producing activated sludge in sufficient quantities to test it on the Rothamsted experimental farm. Laboratory experiments were also in hand from which they hoped to get information as to where the large amount of nitrogen in activated sludge came from; was it due to the fixation of atmospheric nitrogen, as had been suggested in some quarters, or was it derived from the ammonia in the sewage? With regard to the possibilities of distillation, that was largely an engineering proposition with which he was not competent to deal. The de-watering problem had not yet been solved, and it was a very serious one. In the case of the little plant at Harpenden, the works were excavated out of the solid chalk. By making a lagoon in the chalk and pumping the sludge into it under a waterproof cover he had been able to get the sludge into a spadeable condition in about a fortnight, i.e., 15% of dry matter. In that state it could be carried on a farm cart, but beyond that it did not yield its water very readily. With regard to the

residual value of the sewage sludges when applied in heavy dressings, the pot-culture experiments were not comparable with actual operations on the farm. The pots were kept in a glass-house protected from rain, and leaching did not take place. The only water was that given when it was required, so that the results were rather better than would be obtained, probably, on the farm. Generally speaking, he thought that the fertilising constituents of activated sludge which were not taken up by the first crop had a reasonable prospect of being taken up by the succeeding crops.

Nottingham Section.

Meeting held on May 19, 1920.

MR. J. WHITE IN THE CHAIR.

THE DELIQUESCENT AND DRYING OF AMMONIUM AND ALKALI NITRATES AND A THEORY OF THE ABSORPTION OF WATER VAPOUR BY MIXED SALTS.

BY E. B. R. PRIDEAUX, M.A., D.SC., F.I.C.

The application of scientific control to the processes of drying and grinding salts will require a more complete knowledge than is at present available of the vapour pressures of the various systems which can be formed by salt hydrates, solutions, etc. In the case of anhydrous salts, which alone have been considered in this paper, the tendency of the saturated solution to lose water to an atmosphere in which the pressure of water vapour is p_1 will be a function of the ratio p_1/p , p being the pressure of water vapour in equilibrium with the univariant system, salt, saturated solution, vapour at a given temperature. The application of high drying temperatures in the hope of thereby increasing p_1 so that it is unquestionably above p , while usually successful in this object and in securing rapidity of drying, may, on the other hand, be undesirable on account of the existence of transition points or possibility of decomposition and on account of the cost of maintaining these higher temperatures. For each salt there is probably an optimum temperature in determining which the equilibrium vapour pressures must be taken into account. These pressures, p_1 , as well as those p of the atmosphere, may conveniently be referred to that of saturated water vapour p_s at the same temperature, and the dehydration tendency of the salt expressed as its relative humidity, p_1/p_s , which is comparable to the relative humidity p/p_s of the atmosphere. The greater is p_1/p_s the more favourable will be the conditions for drying the salt at ordinary temperatures. If the ratio relative humidity of salt to relative humidity of air (or p_1/p) is greater than 1, drying is possible, and a suitable figure by which to express the speeds of drying would probably be the Van't Hoff maximum work expression $4.57 \tau \log p_1/p$ for 18 g. of water. Similarly, the tendency to deliquesce under atmospheric conditions is a function of the ratio p_s/p_1 .

In the determination of the vapour pressures which will just produce deliquescence in the pure nitrates and in a mixture at various temperatures the kind co-operation of Dr. R. M. Caven is gratefully acknowledged.

Experimental.—The pressures of saturated solutions were measured by two distinct methods, i.e., by the direct and the differential tensimeter. The former (the Johnston tensimeter) was similar to that used in determining the pressures of saturated ammonium nitrate above 40° C. (J., 1919, 353 r).

On the whole, this method is preferable for measurements at higher temperatures or when only a few measurements are required for each of a series of solutions. The differential tensimeter is independent of the barometric pressure and is particularly well suited for measuring small differences of pressure at lower temperatures at which a long time may be required for the attainment of equilibrium.

The Johnston tensimeters may be in one plane, although they are often bent round for convenience of use. The differential tensimeters were of the usual type. One hull contained the saturated solution and solid salt, the other, water or another saturated solution. In all measurements the solutions were maintained at the temperatures until the pressures, or pressure differences, were constant, and at least for a quarter of an hour. When the manometer liquid was mercury the readings were corrected to mercury at 0° C. by the approximate formula $h = h_0 (1 - 0.0001733t)$. When it was bromonaphthalene the heights, h , in cm. of bromonaphthalene were multiplied by the factor $10D + 13.596$, D being the previously ascertained density of bromonaphthalene at the temperature in question. The factor varies from 1.108 at 10° to 1.090 at 35° C., and the result is in mm. of mercury at 0°.

Ammonium nitrate.

TABLE I.

Pressures of saturated solutions of ammonium nitrate.

Temperature	Difference water minus solution.	Water.	Solution.	Measured directly.
11.6	3.00	10.21	7.21	—
12.2	—	—	—	7.1
15.0	4.25	12.78	8.53	—
15.9	—	—	—	10.5
17.3	4.89	14.72	9.83	—
17.6	4.99	15.00	10.01	—
19.0	5.7	16.46	10.76	—
20.4	6.5	17.84	11.34	—
20.7	—	—	—	12.3
22.0	7.11	19.60	12.55	—
23.9	—	—	—	14.8
24.1	8.57	22.31	13.74	—
25.7	9.95	24.55	14.60	15.5
27.0	10.45	26.65	16.20	—
27.3	—	—	—	16.8
28.0	11.70	28.10	16.40	—
29.3	12.90	30.31	17.41	—
30.1	—	—	—	19.8
30.3	14.10	31.92	17.82	—
31.15	14.70	33.70	19.00	—
31.60	15.00	34.58	19.58	—
31.70	—	—	—	20.8
32.00	—	—	—	21.6

In column 5 are given the pressures determined in the Johnston tensimeter with mercury. In column 2 are the differences of pressure between water and saturated ammonium nitrate in the differential tensimeter with mercury. By subtracting these from the corresponding pressures of saturated water vapour the pressures of ammonium nitrate are obtained (column 4). They are, on the whole, about 1 mm. lower than those obtained by the direct method. A formula has been calculated which reproduces the pressure of saturated ammonium nitrate up to 32° C. $\log p = 7.425 - 1869/T$.

TABLE II.

Temperature.	Pressure of solution from formula.	Relative humidity.
10	6.7	0.76
15	8.7	0.70
20	11.2	0.66
25	14.5	0.61
30	18.3	0.57
35	23.2	0.56
38	29.9	—

The vapour pressures do not rise very sharply with rise of temperature, the increase between 11° and 32° being only about 13 mm. as against 25 in the case of pure water. Consequently the increase in speed of drying resulting from rise of temperature

is not so great as in the case of less soluble salts like potassium nitrate. Deliquescence will not take place and the salt can be dried in air of a relative humidity below 0.7 at 15°, whereas at 32° the relative humidity must be below 0.6.

Potassium nitrate.—This salt has been taken as a standard of deliquescence in Marshall's "Explosives," not only for other nitrates but also for hygroscopic mixtures such as black powder. It seemed desirable to obtain its direct relation to ammonium nitrate by means of the differential tensimeter using bromonaphthalene. The differential pressures so obtained are given in Table III.

TABLE III.

Pressures of saturated potassium nitrate solutions.

SERIES I.			
t°C	Difference KNO ₃ —NH ₄ NO ₃	Pressure of NH ₄ NO ₃	Pressure of KNO ₃
11.0	1.80	7.2	9.0
17.8	3.59	10.3	13.9
23.1	5.08	13.15	18.8
24.8	6.61	14.1	20.7
26.0	7.25	14.6	21.75
26.9	7.89	16.2	23.9
28.0	8.41	16.5	25.0
29.05	9.20	17.2	26.4
29.65	9.56	17.5	27.1
	water minus KNO ₃	water	KNO ₃
27.4	1.12*	27.2	26.08
28.8	1.45*	29.6	28.15
26.0	8.1	15.3	23.4
26.68	8.35	15.7	24.05
27.60	9.0	16.4	25.4
28.61	9.57	17.2	26.77
29.50	10.20	17.9	28.1
30.55	11.02	18.8	29.82
31.60	11.60	19.5	31.1
31.22	11.35	19.2	30.55
31.79	11.80	20.3	32.1
32.1	12.1	20.1	32.2

The two measurements marked with asterisks are those of saturated water vapour against saturated KNO₃. Two series of results in different tensimeters give results which differ by about 0.6 mm. in the overlapping part of the curve. On adding the pressures of ammonium nitrate, there is obtained the pressure curve of saturated potassium nitrate, which lies very close below that of water at these temperatures. The relative humidities p_1/p found thus are given in column 2 and those given by Marshall in column 5.

TABLE IV.

Relative humidity of potassium nitrate in terms of saturated water vapour.

t°C	$p(\text{KNO}_3)$	$p(\text{H}_2\text{O})$	Relative humidity	Relative humidity (Marshall)
0°	—	—	—	0.97
10	8.5	9.17	0.93	0.965
15	11.6	12.70	0.915	—
20	16.0	17.39	0.92	0.947
25	21.2	23.55	0.90	0.938
	or 22.0		or 0.93	
30	29.2	31.55	0.925	0.925
32.0	33.0	35.36	0.93	—

The vapour pressures of mixed salts.—Commercial salts usually contain small quantities of others, i.e., in the case of ammonium nitrate, sodium nitrate, ammonium sulphate, etc. These affect the relative humidity, and the most important in this respect will be the more soluble salts such as sodium nitrate. A theory of the mechanism of deliquescence in such a mixture has been propounded (below) and has been tested on experimental results.

The vapour pressures of mixtures of ammonium nitrate and sodium nitrate were measured against those of pure ammonium nitrate. There are two extreme solutions possible with solid ammonium nitrate in excess, (1) that containing the two salts in the same ratio as in the mixture; and (2) that saturated with both salts. Case (1) was first investigated by means of a solution made from ammonium nitrate and 2% of sodium nitrate. The vapour

pressure differences between this and pure ammonium nitrate were somewhat irregular, but the latter was higher. The difference at the highest temperature employed (42°) was only 1 cm. of bromonaphthalene or 1.1 mm. of mercury.

Case (2) gives the most important measure of the effect of other salts, according to the opinion of T. M. Lowry, who states that when the ammonium nitrate contains 2% of sodium nitrate the deliquescence is determined by the vapour pressure of the solution saturated with both salts.

TABLE V.

Differences of pressure between saturated NH₄NO₃ and NH₄NO₃+NaNO₃.

t°	p in cm. of bromonaphthalene	p in mm. of mercury.
11.60	1.26	1.40
14.15	1.40	1.60
16.05	1.65	1.82
17.95	1.81	1.99
20.35	2.02	2.22
22.70	2.34	2.575
25.20	2.68	2.945
27.63	2.99	3.275
29.80	3.37	3.78*
30.31	3.46	3.78†
32.20	3.79	4.14
34.00	4.11	4.50
36.05	4.65	5.06
39.10	5.44	5.91
37.84	5.15	5.60†
36.72	4.85	5.28†

*Upwards.

†Downwards.

The vapour pressures of a solution saturated with respect to both ammonium nitrate and sodium nitrate were measured against one saturated with the former alone, in the differential tensimeter, using bromonaphthalene and a milk-glass scale reading in $\frac{1}{4}$ -mm. The readings were converted into mm. of mercury at 0° in the manner already described and are given in Table V.; also on the graph. It will be seen that the differences in pressure are considerable—from 2.55 mm. at 10° to 11.25 at 38°. At a temperature of 30° a sample of ammonium nitrate containing sodium nitrate will absorb moisture or be impossible to dry at the low relative humidity of $14.3 \div 31.6 = 45\%$, whereas in the case of pure ammonium nitrate this limiting relative humidity is $18.5 \div 33$ or 56%. This shows the added difficulty of drying an impure salt and also, if such has to be dried, that raising the temperature is not necessarily and in all cases advantageous. (See Table VI.)

TABLE VI.

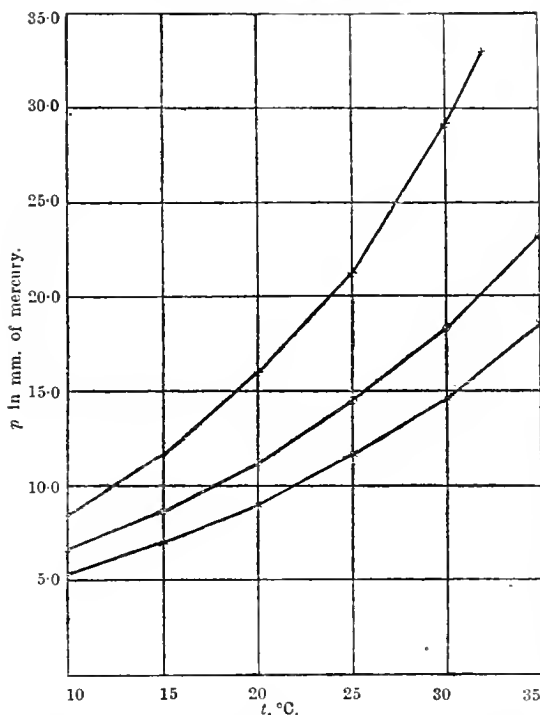
Pressures and relative humidities of saturated NH₄NO₃+NaNO₃.

t°	Pressure NH ₄ NO ₃ minus NaNO ₃	Pressure NH ₄ NO ₃ + NaNO ₃	Relative humidity.
10°	1.27	5.4	0.50
15°	1.69	7.0	0.55
20°	2.22	9.0	0.51
25°	2.90	11.6	0.49
30°	3.73	14.6	0.46
35°	4.76	18.5	0.445
38°	5.62	21.4	0.43

Much valuable evidence on the course of deliquescence or desiccation is afforded by the experiments of Lieut.-Col. J. A. Hall, M.Sc., D.Sc., to whose kindness the author is indebted for permission to use them in the present paper. The salt investigated was sodium nitrate, which is deliquescent under ordinary atmospheric conditions corresponding to its high solubility, 87.5 at 18° (ammonium nitrate, 182 at 18°) and vapour pressure of a saturated solution, 25 mm. at 31.8°, obtained from an extrapolation of the pressure concentration isotherm given in Landolt and Börnstein's tables. The effects of the addition of varying quantities of sodium chloride to sodium nitrate are qualitatively comparable to those of the addition of sodium nitrate to ammonium nitrate.

The method of J. A. Hall briefly consists in exposing the pure or mixed salts in an enclosed space to atmospheres of constant and definite humidity from a large excess of water, or aqueous sulphuric acid, kept at constant temperatures. The samples, of approximately the same weight, surface, and depth, were exposed in weighing bottles and weighed at intervals. It was found by preliminary experiments that these variables, and likewise the fineness of grinding, had effects on the gain of water which were only of secondary magnitude compared with those of the variables under examination: pressure of water vapour and the composition of the sample. The salts were dried to constant weight at 120° — 130° C. and ground so as to pass a 40-mesh

Pressures of saturated solutions.
Highest curve KNO_3 .
Middle curve NH_4NO_3 .
Lowest curve $\text{NH}_4\text{NO}_3 + \text{NaNO}_3$.



sieve but be retained by a 60-mesh sieve. The rate of absorption of water, determined by weighing at regular intervals, shows fluctuations which are due no doubt to the effect of varying convection currents on the vapour transference and of varying rates of diffusion in the solution. These fluctuations are effectively smoothed out by continuing the comparison over long periods, and thus the differences in the rate of gain per unit weight of salt and unit time for different mixtures are clearly revealed. The absorption method is admirably adapted for investigating the behaviour of pure and commercial salts exposed to varying atmospheres for long periods. It gives little or no information, however, about the "first deliquescence" of mixtures of two salts as determined above by the tensimeter method. The final deliquescence or continued deliquescence (see below) is, however, definitely determined.

Theory of deliquescence of mixtures.

A soluble salt, A, is considered, which is mixed with a few per cent. of another soluble salt, B. A fundamental assumption, justified by general experience and a few definite measurements, is that all solutions containing both salts and saturated with respect to one (in these cases A is the more

soluble) have a lower vapour pressure than that saturated with A alone. It is applicable therefore chiefly, and in its simplest form, to salts which crystallise anhydrous and which do not form double salts under the conditions. When the mixture is exposed to water vapour, this will dissolve the surface and, no doubt, in time make its way round the grains in the interior of the mass. It will attack in the first place the particles of B which are distributed over the surface of the grains and with them form the solution, saturated with respect to both A and B, which has the lowest vapour pressure, p_1 , i.e., that which is determined by tensimeter measurements with large amounts of both A and B and their solution. On the addition of more water more solution will be formed, which is still saturated with respect to A but unsaturated to B, since there is no longer sufficient B to give the required fixed ratio of B to A. Later the whole surface of each grain will be covered with a film of solution which, given uniform distribution of B in A, finally has the same ratio of B to A as that in the total mixture. The vapour pressure rises to p_2 . The relative humidity, p_2/p_1 , which is characteristic of this long deliquescence is higher than that, p_1/p_s , of the first deliquescence. If the mixture contains 1% or less of B, p_2 may be much less than p_1 , as has already been noted in the case of ammonium and sodium nitrates. What happens afterwards depends upon the magnitude of the pressure p of the atmosphere. If this lies between p_1 and p_2 , the absorption of moisture will stop before the second deliquescence is reached, the impure salt will only take up a certain amount of moisture (cf. remark quoted from J. A. Hall's paper after Table III). In the case of pure salts, of course there is no such stabilising influence; they cannot attain an equilibrium condition of partial deliquescence, but if p is ever so little greater than p_1 , the whole will dissolve in time.

If the mixture of salts is not moved, only a small amount of solution will form on the surface. But with stirring and grinding in this atmosphere, more solution will be formed, i.e., so much as contains the whole of B and so much of A as will give the ratio B/A of a solution which at the same time has a pressure p_1 and is saturated with A. If p is higher than p_2 , the surface film will extend inwards, giving finally a solution in which the salts are present in the ratio of the original mixture.

Series I.—Saturated atmosphere 23° C. Vapour pressure of water 21 mm.

Gains in weight of NaNO_3 + varying amounts of NaCl in atmospheres of different humidities.

		Weight of samples = 5g. Time in hours.					
		NaCl %					
From start	Interval	None	0.03	0.5	2.0	3.0	
22.5	22.5	0.055	0.064	0.085	0.096	0.148	
45.5	23	0.055	0.060	0.080	0.083	0.111	
69	23.5	0.049	0.062	0.067	0.103	0.110	
90	21	0.060	0.054	0.064	0.075	0.099	
116.5	26.5	0.060	0.104	0.105	0.086	0.130	
138	21.5	0.065	0.064	0.090	0.104	0.106	
Totals		0.354	0.408	0.491	0.552	0.704	
Mg. per g. per hr.		0.51	0.59	0.71	0.80	1.0	

In this series the difference between the pressure p_s of saturated water vapour and of all possible solutions was considerable. The first deliquescence of the mixture samples was rapidly completed and not recorded, the subsequent gains recorded represent the rates of transfer due to the difference between 21 mm. (p_s) and the final pressures p_2 corresponding to solutions saturated with sodium nitrate and containing sodium chloride to potassium nitrate in the proportions of 0.08%, 0.5%, etc.

The mean rates of gain over several periods show a steady increase with increasing percentage of sodium chloride. Had the deliquescence pressure been due to solution saturated with respect to both salts the rates of absorption would have been con-

stant for all the mixtures. This was probably the case in the earliest stages of deliquescence.

Series II. Atmosphere over 37.7% sulphuric acid at 23° C. Vapour pressure 13 mm. Relative humidity 62%.

Time Intervals	None	NaCl			
		0.08	0.5	2.0	3.6
22.5	0.001	0.006	0.019	0.018	0.077
23	0.000	0.002	0.010	0.010	0.034
23.5	0.000	0.001	0.005	0.005	0.027
21	0.000	0.000	0.002	0.004	0.018
Totals	0.001	0.009	0.036	0.037	0.150

It would appear that in such an atmosphere there is a percentage of water capable of being held by sodium nitrate containing salt which depends among other things upon the percentage of salt. Series I, in the saturated atmosphere showed no slacking off of the rate of absorption, which would imply that in such an atmosphere the end condition would be when all the sample was in solution in the absorbed water.

In Series II the aqueous vapour pressure of the sulphuric acid used was nearly or quite equal to that of the solution of the pure sodium nitrate. With 0.08% of sodium chloride the solutions formed in the early stages with a fairly high ratio of chloride to nitrate have pressures which are lower than *p*. They absorb water, but the rate of absorption falls to zero in 69 hours, the solution formed by that time having a pressure nearly equal to *p*. With 0.5% of chloride the initial rate of absorption is greater, but again falls in time to a low value. The first solution which absorbs and deliquesces continuously will be that which, having a ratio of B/A equal to that of the original mixture and saturated with A, has at the same time a pressure below *p* (i.e., 13 mm. at 23° C.). This mixture will be recognised by the fact that the rate of absorption becomes constant, which had evidently not taken place even in the last experiment with 3.6% of chloride.

Series IV.—Atmosphere over 43.7% sulphuric acid. Vapour pressure 10 mm. Relative humidity 48%. Salt wet.*

Time Intervals	None	NaCl%			
		0.08	0.5	2.0	3.6
		Weight of water at start.			
19.5	0.128	0.165	0.216	0.208	0.285
27.5	0.086	0.145	0.182	0.197	0.286
25	0.042	0.072	0.143	0.188	0.288
25	0.014	0.040	0.121	0.181	0.291
25	0.000	0.019	0.091	0.183	0.294
10	0.008	0.015	0.080	0.183	0.206

*The numbers are those selected from the original paper, and hence are not consecutive. Those not selected are quite similar.

These results show the reversal of deliquescence. The wet salt was placed in an atmosphere of lower vapour pressure than its own. The loss should have been uniform in the case of the pure salt, but probably the slowness of diffusion in the solution interferes with regularity of loss. It is evident, however, that with about 2% of chloride there is very little loss, and with 3.6% a gain. Such a mixture could not be dried at this temperature unless the percentage of saturation of the atmosphere was reduced below 48.

Series V.—Saturated atmosphere at 13° C. Vapour pressure 11 mm.

Time Intervals	None	Percentage of salt.			
		0.08	0.5	2.0	3.6
24	0.047	0.051	0.070	0.080	0.077
23	0.047	0.043	0.054	0.055	0.070
25	0.047	0.052	0.065	0.064	0.072
Totals	0.141	0.151	0.189	0.199	0.219
Mg. per g. per hour	0.392	0.42	0.525	0.55	0.61

These results are similar to Series I, and show the constant gain of water from a saturated atmos-

phere at a lower temperature, at rates which vary also with the percentage of chloride. The method is particularly valuable at these lower temperatures, at which it is sometimes difficult to obtain concordant results with tensimeters.

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DISCUSSION.

Mr. RICHMOND referred to the practical use which might be made of such measurements in the grinding and drying of salts and in laboratory drying processes. Specimens of the same salt often differed considerably in their tendency to deliquesce, strikingly illustrated by the behaviour of the common salt of the household. He would be glad of any information as to the best conditions for drying salts and for analysis.

Mr. WILKIE said that tetrahydrated potassium sodium tartrate presented some manufacturing difficulties. One sample might give a dry powder—another apparently similar a coherent mass. During grinding the chemically combined water was liberated, and the substance then probably crystallised again. In drying aqueous and hydro-alcoholic liquors to constant weight in water ovens did a higher temperature possess any advantages?

Mr. DUNFORD called attention to the fact that the drying of glue was much influenced by the state of the atmosphere, and that it was possible by suitable plant to reduce the time very much—an important factor in commercial success.

The AUTHOR, in reply to Mr. Richmond, said that cases differed so much that it was impossible to make general rules, but it seemed to him better where possible to dry at a relatively low temperature and to decrease the relative humidity of the atmosphere. The speed of drying in a vacuum was probably much greater than even in a rapid current of dry air. In answer to Mr. Wilkie, he did not desire to minimise the undoubted advantages of drying at higher temperatures, especially the gain in speed, but merely to point out possible danger of an unnecessarily high temperature. The drying of glue, not being a reversible process, could not be susceptible to exact measurements similar to those on salts, yet measurements would no doubt have some application. One application of a knowledge of the pressures of salt solutions to this industry might be in making atmospheres of controlled humidity.

Communication.

THE EFFECT OF THE ADDITION OF CERTAIN FATTY ACIDS ON THE INTERFACIAL TENSION BETWEEN B.P. PARAFFIN OIL AND MERCURY.

BY SHANTI SWARUPA BHATNAGAR AND WILLIAM EDWARD GARNER.

In a recent communication on "The theory and practice of lubrication" (J., 1920, 51 T), Wells and Southcombe have mentioned that Professor Lewis has measured the interfacial tension between oil and mercury.

The following measurements on the interfacial tension between B.P. paraffin oil and mercury and the effects of the addition of the organic acids on the metal-oil interface, began in October, 1919, at the suggestion of Professor F. G. Donnan, who definitely considered it as a problem of lubrication. Although the complete series of organic fatty acids has not been examined, on account of the difficulties in procuring some of them, the interest

created by the communication of Wells and Southcombe demands that our results should be published without any more delay.

Since there is no method yet known for measuring the solid-liquid interfacial tension, the liquid metal mercury was chosen. B.P. paraffin was selected on account of its definite composition and its high viscosity and high boiling point—properties which are good characteristics of an oil for lubrication purposes.

The fatty acids which were tried were pure except in the case of linolic acid, which was a technical product. The stearic and palmitic acids were purified by Kahlbaum, and the oleic acid was a pure sample obtained from the oil department of the Imperial College of Science.

The interfacial tension between mercury and the solutions of fatty acid in paraffin oil was determined by the dropping pipette method, already used by Donnan (*Z. physik. Chem.*, 1899, 31, 42), and later by his pupils. The bulk of the pipette was filled with carefully cleaned mercury up to a definite mark, and the head of mercury was always kept constant by an adjustable side-reservoir of mercury (fig. 1). This kept the rate of the falling drop quite constant. The end of the capillary dipping in the oil-solutions was made smooth and as regular as possible by grinding it very carefully with fine carborundum, the inner capillary being protected by sealing wax. The capillary was narrowed so that only two drops of mercury were formed in about a minute. It is necessary that the rate of formation of the drop should be very slow, otherwise the size and shape of the falling drop will not be regular, as pointed out by Rayleigh (*Proc. Roy. Inst.*, 1890—1892, 13, 857).

All necessary precautions were taken to prevent the entrance of dust into the apparatus.

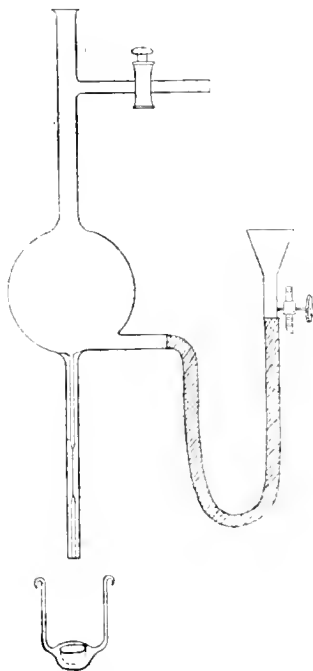
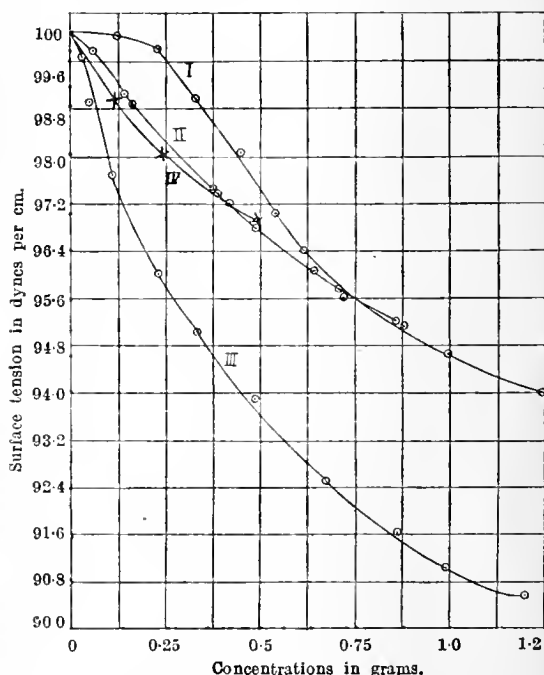


Fig. 1.

The solutions of the fatty acids in B.P. paraffin oil were made up by weight in resistance glass bottles previously carefully cleaned. The acids formed a slight emulsion before they dissolved completely. In all cases the observations were taken only when a clear solution was obtained. Palmitic acid was not very soluble, and so only a few concentrations could be tried.

According to the theory of the instrument (Rayleigh, *Phil. Mag.*, 1899 (5), 48, 321), the surface tension is directly proportional to the weight of a drop. A large number of drops, never less than 20, was allowed to fall into a glass spoon (fig. 1), placed in the various solutions, and weighed after dissolving away the oil with carbon tetrachloride. The results so obtained were finally confirmed in each case by allowing about 50 drops to fall into a weighed quantity of the solution and noting the increase in weight due to the addition of mercury. The deviation between the values of the weight of the drop so obtained was never greater than 0.1%, the slightly variable results in the latter case being due to the drop of oil which is left on the end of the capillary after the experiment. This error was minimised as much as possible by wetting the end of the capillary with the same solution to the same level previous to the experiment.

The average weight of a drop has been calculated from at least 100 drops in each case. The results are shown in curves I., II., III. and IV. The surface tension of B.P. paraffin oil has been assumed to be 100 dynes per cm.



Curve I. represents oleic acid, II. stearic, III. linolic, and IV. palmitic acid. The curves for stearic, palmitic, and oleic acids are very similar in nature, especially at higher molar concentrations. The linolic acid employed was not pure, but in this case the diminution in the weight of the drop of mercury is very much greater than in the case of stearic and palmitic acids. The curve for oleic acid shows an inflexion point at a concentration of about 0.25%. Similar abnormalities have been observed by Donnan and also by Forch in case of nonylic acid.

The resemblance of these curves to those for fatty acids obtained by Langmuir (*J. Amer. Chem. Soc.*, 1919, 41, 1879), by plotting force in dynes per c.c. as ordinates against areas per molecule in sq. cm. as abscissae, is rather striking.

On the strength of this resemblance it might be possible to apply the views of Langmuir (*J. Amer. Chem. Soc.*, 1917, 39, 1865) in explaining the abnormal behaviour of oleic acid in dilute concentrations. Langmuir finds that an oleic acid film in

maximum extension is only half as thick as that of stearic or palmitic acid, so that when doubled in thickness by increasing the concentration (corresponding to the placing of a globule of oleic acid on the surface as tried by Langmuir), the film becomes of the same thickness as that of stearic and palmitic acids, and the cross-section of the molecules is also the same, with the result that the lowering in tension at higher concentration is also the same. It thus appears that the increased efficiency of a mineral oil as a lubricant to which an organic acid is added runs parallel with a lowering of tension at the metal-oil interface.

The authors are indebted to Professor F. G. Donnan, F.R.S., for suggesting this research and for the interest he has shown in the work.

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THE RECTIFIED PETROLEUM SPIRIT FROM THE TOLUENE-PETROL FRACTION OF BORNEO PETROLEUM, WITH A NOTE ON LABORATORY FRACTIONATING COLUMNS.

BY W. A. SILVESTER.

In a recent paper on the above subject (H. E. Evans, this J., 1919, 38, 401 T) it is stated that "it was found impossible to separate . . . further" by distillation the two main fractions which this petrol yields, notwithstanding that, *e.g.*, the "lighter" one ("distilling at 102°–103°") is apparently simply a mixture of heptane, bp. 98° and methylcyclohexane, b.p. 101°. The figures given below, obtained in working along similar lines, do not confirm this conclusion.

Although the experiments now reported afford incomplete support to the assertion, other experience leads the writer to state that the principles of fractional distillation are, as far as laboratory work is concerned, still imperfectly apprehended. The above citation appears to illustrate this, and the point will be considered further later.

The following are the relevant results of one series of experiments:—

(1) 4000 c.c. of the toluene-free petrol, sp. gr. at 15°/15° 0.748, was distilled up a "Hempel" column, 2 ft. high and 2 in. diameter (a lagged copper cylinder $\frac{1}{2}$ -filled with $\frac{1}{4}$ -in. lengths of glass tubing) at the rate of two drops per second, giving from 104°–108°, 1740 c.c., sp. gr. (15°/15°) 0.753 108°–112°, 1205 c.c., sp. gr. 0.746; above 112°, 950 c.c., sp. gr. 0.742 (bar. 766 mm.).

In a second series 4375 g. of the petrol (sp. gr. 15°/15° 0.7485) was more carefully distilled, through a screened all-glass 8-section Young "evaporator" still-head, 3 ft. high and about 1 in. average diameter, giving, again on a single fractionation:—

Range.	Weight, g.	Sp. gr. 15°/15°.
90° to 102°	59	0.752
" 104°	69	0.752
" 106°	948	0.750
" 108°	992	0.748
" 110°	790	0.746
" 112°	429	0.742
" 114°	342	0.739
" 116°	290	0.736
" 118°	191	0.736
" 120°	234	0.737
" 121-6°	146	0.743
Residue	41	(bar.: 762 mm.).
Total	4355	loss therefore 20 g. or 0.5%

In this and the following experiments the temperatures were registered by a carefully calibrated thermometer with 0.2° divisions and corrected for stem exposure. All specific gravities were taken with a specially calibrated small hydrometer and were from time to time checked pycnometrically.

The above fractionations compare as follows (percentages in round numbers):—

Range.	Hempel 2-foot, by wt.	Young evaporator 3-foot, by wt.
104°–108°	44%	47%
108°–112°	30%	26%
Above 112°	25%	26.5%

This is a good result for the much shorter Hempel column.

Working on a somewhat heavier sample of petrol (sp. gr. 15°/15°: 0.7483), Evans's first distillation (the only one available for comparison) was made through a 12-pear Young column. The results compare thus with the second series above:—

Range.	12-pear. by vol.	8-section "evaporator," by vol.
90°–108°	35%	47%
108°–115°	38%	35%
115°–120°	25%	18%

Thermometric differences probably account for some of this disagreement.

All the above material, from both fractionations, save small quantities used meanwhile for other tests, was again twice *cumulatively* fractionated up the "evaporator" still-head, giving products showing the following boiling-point-specific-gravity relationships:—

Range.	Sp. gr.	Range. (Bar.: 760 mm.).	Sp. gr.
100° to 101°	0.755	111°–112°	0.743
" 102°	0.756	" 113°	0.742
" 103°	0.757	" 114°	0.741
" 104°	0.7565	" 115°	0.740
" 105°	0.755	" 116°	0.738
" 106°	0.754	" 117°	0.737
" 107°	0.752	" 118°	0.737
" 108°	0.750	" 119°	0.7365
" 109°	0.748	" 120°	0.737
" 110°	0.747	" 121°	0.740
" 111°	0.745	" 122°	0.741
		" 123°	0.741
		Residue	0.755

* Max. volume per degree.

One point is clearly indicated by the above figures and could only have been brought out by checking continuously the distilling range against the specific gravity—that is that repeated cumulative fractionation would have further resolved the mixture and separated the respective paraffinic and naphthenic components of the two main fractions. This is only to be expected in spite of the small differences in boiling-point. A still further separation of methylcyclohexane (hexahydrotoluene) from the 102°–103° fraction was, in fact, achieved, but the data obtained are not now at hand. Some evidence, however, is yielded by the following figures, which also demonstrate the absence of any noteworthy constituent of boiling-point between 105° and 115°. These are the results of tests made to give a definite index of the homogeneity of the different fractions. 100 c.c. of each fraction was distilled in the standard Engler apparatus for petrol tests, and the volume percentage distilling up to, between, and above the original limits of the fraction registered:—

Fraction.	Vol. % distilling between original limits, <i>i.e.</i> , in original temperature range.
102°–103°	56
103°–104°	504
104°–105°	424
105°–106°	384
106°–107°	31
107°–108°	26
108°–109°	26
109°–110°	23½
110°–111°	18
111°–112°	26
112°–113°	26½
113°–114°	26½
114°–115°	23½

The lack of homogeneity in even the best fraction—that boiling from 103° to 104°—is here very apparent, and the present writer is at a loss to understand how Mr. Evans came to his conclusion.

The presence of methylcyclohexane in the petrol was proved by isolation of its chloro- and nitro-derivatives.

To return to the question of fractionation. In this branch academic and laboratory practice is still far behind that of the works. Separation of the components of a boiling liquid mixture in a fractionating column takes place (1) by fractional condensation by loss of heat through the walls of the column, and (2) by interchange of heat between the rising vapour and the returning condensate whereby the "heavy" constituents of the vapour condense out whilst the "lighter" parts of the condensate revolatilise. On the works scale the two processes are kept separate; fractional condensation is regulated, and kept under control, by the "dephlegmator"—a small reflux condenser at the top of the column. The condensate thus provided is brought into intimate contact with the rising vapour by a bubbling device. The whole process therefore is of great efficiency. In the laboratory, on the other hand, such apparatus as is commonly used, except the columns of Hempel and Linnemann and their modifications, mainly relies on fractional condensation to effect its object. Such contrivances as those of Glinsky, Young ("pear" and "rod-and-disc"), Dufton (J., 1919, 45 T), and many others simply cause the vapour to take a tortuous passage and depend almost entirely on external—uncontrolled—cooling to bring about the desired separation. Naturally, some heat-interchange between vapour and condensate also takes place, but to nothing like the degree needed for efficient fractionation.

A simple and easily constructed laboratory-size column, in which there is proper provision for heat-interchange is that devised long ago by Hempel. The value of a bead packing in small gas-washing devices is well recognised, and the only reason the writer can assign for the unpopularity of the Hempel still-head is its tendency to choke and "puke." This defect is overcome by proper attention to the construction. If such a column is lagged and surmounted by a constant temperature dephlegmator, so as completely to reproduce works practice in miniature, most efficient results are obtained (Washburn, J. Ind. Eng. Chem., 1920, 19, 73).

The superiority of the Hempel column, even as commonly used, is demonstrated both by the writer's and Mr. Evans's results and also by comparative tests reported from time to time (e.g. Friedrichs, Z. angew. Chem., 1919, 32, 340; Rittman and Dean, U.S. Bureau of Mines Bulletin on Analytical Distillation of Petroleum; Young, Chem. Soc. Trans., 1899, 679). In other respects these comparative tests are of little value. The efficiency of "fractional-condensation" devices obviously depends largely on the thickness and area of the walls, as well as on the height; this is usually overlooked in

reporting comparisons. It is to be regretted that any standing is given to the fantastic arrangements of bulbs and bends, such as are sold as Le Bel-Henninger etc. still-heads.

Finally, in the Hempel device, as ordinarily used, it is evident that a not inconsiderable proportion of the fractional condensation results from absorption of heat by the packing material. It would be expected, therefore, that, *ceteris paribus*, a packing of high would be more efficient than one of low specific heat. This appears to be illustrated by Mr. Evans's results (*loc. cit.*) A steel (sp. ht. 0.1158, heat capacity for 1 c.c. 0.915 cal per 1° C.) packing proved more efficient than a lead one (sp. ht. 0.0314, heat capacity for 1 c.c. 0.358 cal per 1°). The decrease in size of the balls—and therefore increase in surface—and the relative increase in mass per c.c. would also take part in the effect.

THE DETECTION OF NATURAL BARYTES IN LITHOPONE ETC.

BY S. STEWART, F.I.C.

Lithopone, Orr's zinc white, and other pigments composed essentially of zinc sulphide and barium sulphate should contain the latter only in the precipitated form. Inferior qualities sometimes contain natural barytes, to the detriment of their covering power, owing to the large size of the particles of barytes as contrasted with those of the precipitated sulphate. Its opacity is less, and although, when used as an ingredient in ordinary paints, this is perhaps of secondary importance, it becomes a matter of serious consideration when used for some other purposes; for example, in making white inlaid linoleum the use of lithopone containing natural barytes leads to the production of a yellowish white. The microscope affords a convenient means for differentiating between natural and precipitated barium sulphate. The test is carried out as follows: A minute portion of the sample is spread on a microscope slide with a drop of water, dried, and examined with a $\frac{1}{2}$ in. or $\frac{3}{4}$ in. objective, the diaphragm being closed so as to give a dark background. If only precipitated barium sulphate be present, it appears as a very fine powder composed of minute crystals of uniform size, whereas if there is an admixture of natural barytes, even when very finely ground, transparent irregular pieces of greater size will appear. The certainty of the method is enhanced if the zinc sulphide present in the lithopone be first removed by treatment with dilute hydrochloric acid and potassium chlorate and the insoluble residue examined as above.

This method has been in use for 22 years in the laboratory of Messrs. Michael Nairn and Co., Ltd., Kirkcaldy, to whom the author's thanks are due for permission to publish it.

PROCEEDINGS OF THE

THIRTY-NINTH ANNUAL GENERAL MEETING,

Newcastle, July 13th, 1920.

The thirty-ninth annual general meeting of the Society was held in the Chemical Lecture Theatre of Armstrong College on July 13.

THE LORD MAYOR OF NEWCASTLE (Councillor Walter Lee, J.P.), in extending to the Society a hearty welcome to the city, assured them that they in Newcastle fully realised the importance of the chemical industry and he hoped from the scientific, practical, and social points of view that their meeting would be a great success. He looked to the industry they represented to take a great part in the work of reconstruction now proceeding. The country was now beginning to settle down and there was a feeling that the boom was ending. There would be the question of providing employment for many people, and he looked to science to do a great deal for industry in that direction.

SIR THEODORE MORISON, Principal of Armstrong College, in welcoming the Society to Armstrong College, said that it was a source of satisfaction to him that they had chosen the College as their meeting-place. The association of the Universities with industry was one of the outstanding features of the present time. Industry was becoming more and more alive to the importance of education, and particularly university education, and the universities were also becoming alive to the importance of industry. At one time the universities had rather held aloof from the practical side of life, and it was due particularly to the chemical faculty that the division between universities and industries had been broken down. In every faculty of the universities the professors were now bringing forward their contributions to industry; perhaps the most remarkable thing of all was to see the faculty of philosophy, recently discovering that one of its branches—psychology—might be of use to industry.

PROF. LOUIS, in thanking the Lord Mayor and Sir Theodore Morison, said:—In the regretted absence through illness of the President, it devolves upon me to thank you in the name of the Society of Chemical Industry for the welcome that you have accorded us on the occasion of our visit to Newcastle-on-Tyne. I do this with the utmost pleasure, more particularly because I feel that such a two-fold Civic and Academic reception is one that peculiarly befits the aims and objects of our Society, a Society whose principal object has always been to foster the applications of science to industry. You, my Lord Mayor, have welcomed us as the chief Civic representative of a great city renowned for its industry and enterprise, the capital of a district second to none in the United Kingdom for industrial development, and you, Sir Theodore Morison, have been good enough to welcome us as the head of the principal educational and scientific institution of this city. We are grateful for the manner in which you have thus united to bid us welcome here, but I may be allowed to say that I see in this joint reception of the Society of Chemical Industry something more than a manifestation of the traditional North-country hospitality; I hail it as an omen of the new era that is developing around us and which, properly understood, will certainly make for the continued prosperity of Tyneside. As one who has lived here for a quarter of a century in touch with a certain number of leading industries of the district, I think that I am justified in voicing my conviction that the local industries have never,

until perhaps quite recently, duly appreciated the advantages that they could derive from a more intimate contact with the modern applications of science. North-country manufacturers have indeed not availed themselves as much as they might have done of the resources of scientific technology. There are, of course, various reasons that have contributed to this result, and I venture to suggest that one cause, generally overlooked, is to be found in the many admirable qualities of the Tyneside working man. I have been engaged in work and in the management of men in many quarters of the globe and can truthfully say that for skill, intelligence, and resourcefulness the North-country workman has no superior anywhere. It is this fact that has given rule-of-thumb methods a longer lease of life than in most other places and has enabled the managers of North-country works to dispense with scientific methods and scientific control of operations for a longer time than could have been the case were these operations conducted less skilfully. The severe industrial stress due to war has, however, forced forward a change of view in this as in so many other respects, and the need for intensive production under altered conditions has brought home the importance of applying the methods of modern science, and manufacturers are coming to perceive that the sphere of the chemist and the physicist may be largely extended. I could give a long list of Tyneside firms, which half a dozen years or so ago took but little heed of these sciences, but which to-day recognise the chemical laboratory as an essential part of their establishments, and will readily admit that they have made no better investment than the money spent on this portion of their equipment. It is becoming daily more evident that the after-war struggle will be in its way as strenuous as was that of actual warfare, and that we can hold our own only by developing and extending the scientific and technical methods that brought us victoriously through the war. That you, my Lord Mayor, and you, Sir Theodore Morison, have combined to welcome to Tyneside a Society that stands pre-eminently for the applications of high scientific methods to industry is evidence that you appreciate the importance of these principles in this district, and we sincerely hope that the visit here of our Society will contribute its share towards stimulating interest in a more intimate union between science and industry in all its branches, particularly in the highly diversified series of industries which we group together under the head of "Chemical Industry," with which we are here most closely identified.

THE LORD MAYOR briefly responded to a vote of thanks accorded by acclamation. PROF. LOUIS then took the chair and the formal business of the meeting was proceeded with.

The minutes of the last annual meeting were taken as read.

It was unanimously agreed, at the suggestion of the Chairman, to ask the Secretary to send to the President, in the name of the Society, a telegram expressing their sympathy with him in his illness and their hope that he would have a speedy and complete recovery.

Dr. G. S. Weyman and Mr. A. Short were appointed scrutineers of the ballot for the election of Council, and the ballot was declared closed.

REPORT OF COUNCIL.

During the year the Council has held 10 ordinary meetings, and the following Committee meetings have been held: Finance Committee, 18; Emergency Committee, 4; Publications Committee, 8; Transactions and Abstracts Sub-Committee, 24; Review Sub-Committee, 24; Annual Reports, 5; Sub-Committee on Weekly Journal, 1; Literary and Libraries Committee, 2; General Purposes Committee, 5; Technical Research and Allied Societies Committee, 9.

The number of members on the Register is now 5612, as compared with 5236 last year. Since the last Annual Meeting 562 members have been elected, 71 former members have been restored to membership, and the losses have been 257.

The Council regrets to record the deaths of 46 Members (of whom 13 were Original Members), viz.:—

Joseph Barrow, Harry Broadbent, John R. Brooke, Prof. Adrian J. Brown, Arthur H. M. Brown, Thomas Buggy, Paul Butler, F. J. R. Carulla, Herbert S. Coleman, Percy W. Copeland, W. J. Cousins, W. J. Cowan, Wm. B. Cowie, Joe Frost, John C. Galletly, Dr. Frank B. Gallivan, Holbrook Gaskell, George H. Graves, Dr. Harold C. Greenwood, Dr. J. Grossmann, C. E. Groves, Edwin Haviland, Robert F. Jenkins, Wm. A. Johnston, Walter R. King, F. E. Lott, Dr. Edward G. Love, J. S. McArthur, Dr. Rudolph Messel, John Metcalf, Dr. B. More, Thos. Pierre Morson, Wm. Letts Oliver, Prof. L. T. O'Shea, T. W. Pilley, W. Cobden Samuel, Robert C. Scafe, Kenneth W. Smith, Watson Smith, William G. Smith, J. Fleming Stark, I. Frank Stone, Herbert S. Sutton, John C. Umney, Dr. Elwyn Waller, Leonard E. Weldon.

Dr. R. Messel, F.R.S., was President in 1911–12, and again in 1914, and held the office of Honorary Foreign Secretary from 1913.

Mr. Watson Smith was Editor of the Journal from its commencement until 1914.

Mr. John Gray's year of office as President of the Society expires after the Annual General Meeting, and as he was unable to accept nomination for a second year the Council has nominated Sir William J. Pope, K.B.E., F.R.S., as his successor in office.

Mr. D. Lloyd Howard found it necessary, owing to pressure of business engagements, to resign the post of Honorary Treasurer to which he was elected in 1918, and Mr. E. V. Evans, one of the Vice-Presidents, has been appointed his successor.

Professor Henry Louis, a Vice-President and former President, has been elected Honorary Foreign Secretary in succession to Dr. R. Messel, deceased.

Dr. E. F. Armstrong resigned the office of Vice-President, retaining a seat on the Council in virtue of his being Chairman of the Liverpool Section, and Mr. S. R. Trotman was elected to fill the vacancy. Mr. L. P. Wilson was elected a Vice-President in room of Mr. Evans.

Mr. John Gray has been nominated a Vice-President, and to the other vacancies caused by the retirement of four Vice-Presidents Mr. D. Lloyd Howard, Dr. Stephen Miall, and Mr. Edwin Thompson have been nominated.

Four ordinary members retire from the Council, and to fill the vacancies thus created six nominations have been received; a ballot will therefore be taken.

The following chairmen of local sections retire: Mr. C. E. Sholes (American), Mr. Q. Moore (Glasgow), Prof. P. P. Bedson (Newcastle), Mr. F. H. Carr (Nottingham), and Mr. W. McD. Mackey (Yorkshire). The following have been elected to succeed them respectively: Mr. S. R. Church, Mr. J. H. Young, Dr. J. H. Paterson, Mr. J. H. Dunford, and Mr. S. H. Davies.

Prof. J. W. Hinchley retires from the chairmanship of the Chemical Engineering Group, and Mr. J. Arthur Reavell has been elected his successor.

Dr. Stephen Miall has resigned the Honorary Secretaryship of the London Section, and has been succeeded by Dr. G. W. Monier-Williams.

The Council desires to express its cordial thanks to the retiring officers for their services to the Society, and has conveyed to the relatives of the late Dr. Messel and of the late Mr. Watson Smith its high appreciation of the valuable work done by them respectively.

The President during his year of office has visited the following Local Sections of the Society and held informal meetings with the Committees:—Birmingham, Edinburgh, Glasgow, London, Newcastle, and Yorkshire. He has also met the Committee of the Chemical Engineering Group. On each occasion he was accompanied by the General Secretary. The Council is very pleased to know that these visits have given much satisfaction to the local committees and feels sure they will be productive of great benefit to the Society.

An invitation was received from the Newcastle-on-Tyne Section to hold the Annual General Meeting (1919) in Newcastle. For the reasons stated in last year's Report of Council this invitation was not accepted, but it gave the Council much pleasure to receive a renewal of the invitation for 1920, which has been unanimously accepted.

A cordial invitation has been received from the Canadian Section to hold the Annual Meeting for 1921 in Montreal, and the Council recommends its acceptance.

It was decided in November that for the further period of twelve months no enemy aliens should be admitted to membership of the Society.

The Committee of the London Section has promised to give a donation of 200 guineas to the Society out of funds remaining over from the Annual General Meeting last year. The Council much appreciates this generous gift.

The Audited Balance Sheet and Statement of Income and Expenditure for the year ending December 31, 1919, which have already appeared in the Journal for June 30, will be laid before the meeting.

The Amended By-Laws recommended by the Council for adoption, and which have been circulated among the members, will be submitted at the meeting.

The Journal for 1919 contained 1866 pages of text (Review 486, Transactions 412, Abstracts 968), compared with 1612 in 1918. Advertisements: 1298 pages in 1919; 1464 pages in 1918.

Volume IV. of the Society's Annual Reports on the Progress of Applied Chemistry was published this year, and the purchase of the new volume has been accompanied by a considerable number of orders for Vols. II. and III. Volume I. has been out of print for more than a year.

The preparation and publication of a collective index to the Journal from 1906 to 1919 inclusive has been under consideration, but, having regard to the probable cost involved, it was decided that at the present time it is impossible for the Society to undertake the work without substantial financial assistance.

A proposal that the Journal should be published weekly has been discussed, but in view of the present financial position of the Society it was resolved that the whole question be deferred for the present.

Representations have been received that the present system of charging authors the cost price for reprints of papers published in the Journal is objectionable, and that it acts as a deterrent to would-be contributors. The Council is in full agreement with these views and has decided that the former plan of giving a certain number of

free copies should be reverted to as soon as the Society's financial position permits.

Arrangements have been made to supply the press with items of news of general interest relating to chemical industry. It is hoped by this means to make the Society's activities more widely known.

The Council earnestly invites the co-operation of all members of the Society in maintaining and extending the supremacy of the Journal as the premier non-trade British publication devoted to chemical industry.

Mr. G. H. Ford, M.Sc. (Manchester) has been appointed Assistant Editor of the "Review" Section of the Journal.

A scheme drawn up by the President, Mr. John Gray, for the setting up of a number of standing committees has been adopted with the object of facilitating the work of the Council and of providing an appropriate series of committees to which matters requiring special consideration can be at once remitted. A list of these committees, with the names of the chairman and members in each case, is given in the Journal, vol. 39, p. 85 B.

In the absence, through ill-health, of Dr. Carpenter, Chairman of the Finance Committee, the Council has appointed Dr. E. F. Armstrong interim Chairman.

Professor Henry Louis has been elected a member of the Finance Committee in place of Dr. Messel, deceased, and Dr. Lovinstein has resigned his membership of it.

Mr. J. Arthur Reavell was co-opted to the Transactions and Abstracts Sub-Committee of the Publications Committee as representative of the Chemical Engineering Group in the absence, through ill-health, of Prof. J. W. Hinchley.

Prolonged and serious consideration has been given to the financial position of the Society. The Income and Expenditure accounts for the years 1918 and 1919 show deficiencies of £3145 and £2353 respectively. In order to wipe off the former, several of the Society's investments had to be realised, and a similar course will have to be adopted for the purpose of clearing off the latter. The deficiencies have been due to the enormous increase in the cost of printing and paper, and the Council, realising the seriousness of encroaching further on the Society's reserve funds, has, after much deliberation, decided that the Annual Subscription must be increased from January 1 next. A resolution regarding this will be placed before the meeting. To provide for the case of young persons applying for membership a scheme has been devised under which such persons will be admitted at a lower subscription than that payable by ordinary members. This scheme will be laid before the meeting for adoption.

Realising the urgent importance of deriving as large an income as possible from the advertisements in the Journal the Council decided to take this business into its own hands and gave Messrs. T. G. Scott and Son the required six months' notice, which expired on May 31. Mr. P. Le Good has been appointed Advertisement Manager, and he, along with his staff, are accommodated in the Society's offices.

A resolution was unanimously passed at an Extraordinary General Meeting of the Society, held on October 23, declaring that the Chairman and Honorary Secretary of a Subject-Group shall be *ex officio* members of the Council; and this was unanimously confirmed at a similar meeting held on November 20. (See Report of Council, 1919.)

The Council has awarded the Society's Medal for 1920 to Monsieur Paul Kestner in recognition of his distinguished services to chemical industry. (See Journal, vol. 39, p. 157 A.)

Through the generosity of Messrs. Cross and Bevan, and of Sir T. P. Latham, Bart., of Weybridge, a prize is again offered for an essay on "The Interconnexion of Economic Botany and

Chemical Industry"; also a fellowship endowment. (For full particulars see Journal, vol. 38, p. 471 B.)

It is gratifying to know that members of the Society are taking advantage of the facilities offered them by the Chemical Society for using its Library and borrowing books from it. The Council has renewed for 1920 its donation towards the expenses incurred in connexion with the Chemical Society's Library Extension Scheme.

The London Section appointed a Committee, of which Professor J. C. Philip was Chairman, to report on the steps which should be taken in order to standardise the application of the Refractometer to industrial and scientific purposes, and its report was published in the issue of the Journal for December 15.

The Council has received from the Chemical Engineering Group of the Society a resolution requesting it to take up consideration of Standardisation on a wide and comprehensive basis in connexion with apparatus and materials in the Chemical Industry to which standardisation can properly be applied. The Council considers that this subject is one of great importance, and it is securing the co-operation of the Association of British Chemical Manufacturers and of the British Engineering Standards Association in dealing with it.

In the interests of the members of the Society who are Chemical Engineers the Council has taken an active interest in opposing the Civil Engineers (Registration) Bill promoted by the Institution of Civil Engineers. Having regard to the admitted impossibility of the Bill passing in the present session of Parliament, it has been decided to take no further action at present.

The compilation of a Card-Index of the Catalogue of Publications referred to in Chemical Literature, and the Libraries of the U.K. in which they are to be found, has now been brought up to date by Dr. Holt, and has been deposited in the offices of the Society. Further particulars regarding this will be given in an article which it is hoped that Dr. Holt will shortly contribute to the "Review." The Council appreciates very highly this valuable work which Dr. Holt initiated and has brought up to date.

Substantial progress has been made in arranging and cataloguing the stock of books and journals belonging to the Society, and an anonymous donor has contributed the sum of £25 towards the expense of the work.

The Report of the Empire Sugar Supply (Technical) Committee, and the Report of the Committee on the Standardisation of Laboratory Glassware were published in the issue of the Journal for August 15. (See Report of Council, 1919.) Each of these Reports has been reprinted.

The draft rules of the Federal Council for Pure and Applied Chemistry provide that the constituent bodies shall contribute to the expenses of the Federal Council (including the agreed contribution to the International Union of Pure and Applied Chemistry) on a membership basis. The Society's contribution will be £100 per annum, and this has been agreed to. (See Report of Council, 1919.)

The Grasselli Chemical Co. presented to the Society a framed copy of the medal which they have founded in connexion with the American Section. (See Report of Council, 1919.) The gift was accepted with thanks to the donors.

The Imperial Mineral Resources Bureau submitted a scheme to establish a complete system of abstracting publications dealing with the entire subject of mineral products, and suggested that a committee should be formed to consider the best means for carrying this into effect; it was also their intention to set up other committees which should direct their attention to special subjects and classes of mineral products. The Council, on the invita-

tion of the Bureau, nominated two representatives to the "Abstracts" Committee and one representative to each of certain other committees.

A communication was received from the University of London requesting the Council to suggest names of members to form a committee representative of the Chemical Trades to examine its Scheme for Degrees in Commerce. It was considered desirable that such a committee should comprise representatives both of the Society and of the Association of British Chemical Manufacturers, and this suggestion was approved by the University authorities.

A list of the Society's representatives on outside bodies is given in the Journal, vol. 39, p. 85 R. To this list the following should be added:—

Imperial College of Science and Technology (Board of Governors):—Dr. Charles Carpenter.

London University Degrees in Commerce.—*Chemical Trades Committee*:—The President, Dr. Carpenter, Mr. D. Lloyd Howard, and Dr. C. A. Keane, representing the Society; Sir John Brunner, Bart., and Mr. C. A. Hill representing the Association of British Chemical Manufacturers.

Mr. W. F. Reid was appointed the Society's representative on the Committee of the Department of Import Restrictions. The work of the Committee has since been taken over by the permanent officials of the Board.

A communication was received from the President of the American Chemical Society inviting the Council to appoint a Committee on Nomenclature, Spelling, and Pronunciation to co-operate with the corresponding Committee of the American Society. Prof. G. T. Morgan, Mr. T. F. Burton, and Dr. E. H. Tripp were appointed such a Committee, to co-operate with the similar Committee of the Chemical Society of London.

Consideration has been given to a communication from the Conjoint Board of Scientific Societies pointing out the serious handicap from which the nation suffered in the early years of the war in respect to the application of science to war problems and the desirability of approaching the Admiralty and War Office with the object of establishing a more satisfactory and permanent basis for the application of science to the problems concerned in view of future wars. The Board regards it as essential that continuous research by co-operation between the Services and individuals engaged in scientific research should be established with a view to the application as soon as possible of new discoveries and principles to war problems, and that facilities for the mobilisation of a scientific staff without delay and in the most efficient manner should be provided. The Council is fully in accord with the views and proposals of the Board, and has submitted certain suggestions bearing on the scheme.

A communication was received from the National Union of Scientific Workers regarding the question of making representations to the Lords of the Treasury for the purpose of obtaining concessions in respect of the assessment for income tax of scientific workers, along with a suggested form of petition, and the Council has expressed its sympathy with the proposals submitted.

The above report, which was taken as read, was adopted on the motion of the Chairman, seconded by Dr. J. T. Dunn.

HON. TREASURER'S REPORT.

MR. D. LLOYD HOWARD, the retiring treasurer, moved the adoption of the statement of account and balance-sheet (see Journal of June 30, 1920). He had again to report a deficit, though not so large a deficit as in 1918. At the last general meeting he was able to announce that the income from advertisements was coming in at the rate of £10,000 a year, and he was now glad to say that the income for the whole year had exceeded £10,000. That

income they hoped would be increased when the new arrangements made with regard to advertising came into full operation. As to the general position, they had again been compelled to borrow money from the bankers; they were able to borrow sufficient to cover the year's expenditure without realising further securities, and though borrowing was bad, selling was worse. Later they would have resolutions put to the meeting dealing with the question of raising the subscription. It was not for him to go deeply into that subject except to say that while the financial situation was serious he did not consider it to be by any means desperate, and he believed that if the meeting adopted the recommendations of the Council the Society ought to have every prospect of winning through and being again on a sound financial basis. As retiring Treasurer he wished to explain why he did not seek re-election. He had taken office on the death of the late Hon. Treasurer in 1918, and he took the opportunity of thanking his colleagues on the Finance Committee and the Council for the great help they had always given him and for the unvarying kindness and consideration always shown him. They had made his term of office extremely enjoyable in an anxious time. Those not on the Council ought to know the enormous debt they owed to all the officers, especially to their last three Presidents, Dr. Carpenter, Prof. Louis, and Mr. Gray. The initiation and successful working of the Finance Committee and the drafting of the scheme of committees now in operation was due to those three Presidents. His reason for resigning his position as Hon. Treasurer was exactly as stated in the report, namely, that the demands of his business did not give him sufficient leisure to devote that amount of time to the treasurership that he felt he ought to give. He was entirely in accord with the policy of the Finance Committee in every respect, and deeply regretted that his engagements would not permit him to continue in office. He formally moved the adoption of the report.

MR. E. V. EVANS, the Treasurer-elect, in seconding the adoption of the report, said he was sure that he would be voicing the feeling of the members in assuring Mr. Howard of their appreciation of the services he had rendered to the Society. He had set his successor a very high standard to live up to. The main object of the Society of Chemical Industry was to supply its members with a periodical account of the progress of the industry and to keep industrial chemists in touch with the march of applied chemistry. The Journal was the life-blood of the Society, and the Council was therefore most anxious that the committees responsible for the work should keep it at the highest possible scientific standard. The income of the Society was largely dependent upon the popularity of the Journal, and upon its popularity depended the membership and its use to advertisers. They received criticisms of the Journal from time to time, and he assured them that criticism was always highly appreciated. The policy of the Society was to attempt to cater for the chemical manufacturer and commercial chemist as well as for the academic chemist, and all their efforts must be towards making the Journal increasingly interesting and useful to chemists and chemical manufacturers. Since their expenditure had exceeded their income, it was necessary to increase the subscriptions. The only alternative to that would be to decrease the effectiveness of the Society, but that would be disastrous policy. He could not be a party to such a procedure, and would, personally, rather see the Society go down in honourable bankruptcy—which, by the way, was impossible—than its value be detracted. He had much pleasure in seconding the adoption of the report.

The report was unanimously adopted, and a vote of thanks was passed to the retiring Treasurer by acclamation.

THE ANNUAL SUBSCRIPTION.

THE CHAIRMAN said, that the next item on the agenda, the proposed increase of subscriptions, was perhaps the most important of all. In a sense the meeting had already agreed to it by adopting the Report of the Council, but he hoped the matter would be taken independently and discussed on its own merits. The position was, simply put, that they were spending more than they were getting. The Society was in a flourishing condition so far as concerned the influx of new members, but every new member cost more than he paid. Obviously such a state of affairs could not continue. The Finance Committee and the Council had carefully studied the whole position, and had come to the conclusion that the proposed increased subscriptions would just about cover the year's expenditure by rigid economy and care, though it would not leave any surplus. He formally moved the following resolutions:

- (1) That the annual subscription payable by members be £2 10s. from January 1, 1921.
- (2) That the Life Composition Fees be increased from £30 and £25 to £40 and £35 respectively.
- (3) That a member joining under the age of 25 shall be entitled to pay a subscription of £2 per annum until attaining the age of 25, or for three years from the date of election, whichever is the longer period; but subject to the provisions that his application for membership must be accompanied by (1) satisfactory evidence of age, and (2) a declaration that he is not joining the Society as a nominee of any firm or corporation, but solely in his own interests as an individual member.

DR. E. F. ARMSTRONG, in seconding the resolution, said that the Council had not put them forward lightly. The question had been considered for over twelve months. A year ago they had deferred bringing it forward because, like other people, they had been optimistic that things would improve. That optimism had not been justified, and the consequence was that the Council had found it necessary to put forward these resolutions. The Council had taken up a firm position in refusing to allow the standard of the Journal to fall, and he hoped they would all support the Council. He did not think a reduction of expenditure was possible to any appreciable extent. The increase in expenditure was caused by matters over which they had no control—paper and printing costs. The only cutting down of that expenditure was by reducing the size of the Journal—a policy which he felt sure they would agree was a shortsighted one. The decision to increase the subscription had not been come to without much consultation with the Local Sections.

DR. L. F. GOODWIN suggested that more members might be obtained in Canada and in the United States by allowing members there to come in at a lower rate of subscription in order to some extent to meet the competition of the American Chemical Society.

THE CHAIRMAN pointed out that Canadian members already were costing more than the Society received from them. If they only charged the Canadian members the net cost of the Journal the Society would be out of pocket. The ordinary expenses had to be borne by Canadian members as by others. Any representations of the kind suggested by Dr. Goodwin would, however, receive careful consideration by the Council.

ALTERATION OF BY-LAWS.

The proposed alterations to By-laws, as circulated in the Journal, were moved by the Chairman,

who briefly reviewed the main points of these alterations.

DR. STEPHEN MIALI seconded, and the alterations were agreed to without discussion.

PRESENTATION OF MEDAL.

In presenting the Medal of the Society to M. Paul Kestner, Prof. LOUIS said:—"None of the duties devolving upon me to-day affords me greater pleasure than that which I am next called upon to perform, namely, the presentation of the Medal of our Society. M. Kestner, in the name of the Society of Chemical Industry I have to ask your acceptance at my hands of the Medal of the Society. You are aware that the Society presents this Medal to chemists who have achieved distinction in one or other of the numerous branches of our many-sided subject, and the list of our Medallists shows a long line of illustrious names, to which we now take pride in adding your own. Your work has lain essentially in that difficult and complex branch of the subject which we are learning to speak of as chemical engineering, and I may be allowed to express the hope that the award of this Medal to you may be taken as an indication of the importance that the Society attaches to this particular branch of the subject and as an encouragement to those who are devoting their energies to its development. The admirable results that you have achieved and the inventions with which you have enriched chemical technology are too well known to make it necessary for me to do more than refer to them here in very few words.

"In the first place I would like to mention the improvements that you have introduced in the appliances for acid manufacture, such as your fans for acid towers and for acid gases and your automatic acid elevator, an appliance that played an important part in chemical industry during the war.

"Here on the Tyne we are naturally interested especially in all that appertains to fuel and fuel economy, and in the latter respect the climbing film evaporator, with which your name is associated, takes particularly high rank. It has already shown itself capable of effecting remarkable economies in the consumption of fuel required for evaporation, and we are looking forward with great interest to those developments of the principle which you have in hand, as, for example, the application of the climbing film principle to water tube boilers. In the same way we are looking forward with interest to the applications of the same principles to your new methods of sugar manufacture, to which I understand you are devoting your attention at the moment. Having regard to the world shortage of this essential article of food, we can only hope that in the public interest you will persevere with this interesting work and that your labours may be crowned with complete success.

"In addition to all this mass of technical work which you are carrying on with such energy, I should like to mention some of your other activities in connexion with chemical industry, such as the prominent part that you have taken in founding in your own country the sister society to our own, the Société de Chimie Industrielle, and in firmly establishing it in a few years as one of the foremost French technical societies. You have played a great part in helping forward the formation of the Inter-Allied Association of Chemists, which will tend to draw yet closer together the scientific and technical resources of the nations which have devoted these to a common cause in the interests of the world's civilisation. Finally, whilst emphasising the fact that this Medal is a tribute to your own eminent personal merits, I may be allowed to express the hope that the chemists of both our nations will recognise in the homage that we thus pay you the earnest desire of both to continue in

times of peace the close and cordial co-operation that led us through the bitter stress of war to its ultimate triumphant issue."

M. PAUL KESTNER, after receiving the Medal, said that in granting the Medal of the Society to a foreigner the Society had not altogether departed from tradition. Not only was he an old member of the Society, but he was an old friend of Great Britain. He had always been a fervent admirer of Great Britain, and he was quite certain that he owed what success he had attained to the knowledge he had gained in an intimate connexion with this country—a connexion of over 30 years. Truly he had sat at the feet of the British men of science and British manufacturers. He felt he was one of their pupils. The honour which they had conferred on him was totally unexpected, and, in all sincerity, he felt it was the crowning point of his career. He fully realised that in honouring him the Society was honouring his country. In his modest person they had celebrated very nobly and delicately the everlasting alliance between Great Britain and France. He also realised that it was an honour to his colleagues of the *Société de Chimie Industrielle*. It was especially gratifying to him that the medal should be handed to him by his old friend, Prof. Louis. He was a distinguished Past-president of the Society, and under him the Society had achieved some very great work during the war. He would say to Prof. Louis: "*Vous avez bien mérité de la Société.*" He concluded by again thanking them for the honour they had done him and, through him, the *Société de Chimie Industrielle*.

NEXT PLACE OF MEETING.

THE CHAIRMAN read to the meeting the invitation received from the Canadian section to hold the next annual meeting in Montreal. He formally moved the acceptance of the invitation.

MR. H. M. RIDGE seconded, and referred to the very great field which Canada provided for the work of the industrial chemist.

The invitation was accepted unanimously.

PRESIDENT'S ADDRESS.

PROF. LOUIS, in the absence of the President, read the Presidential Address. (This address will be published in full at a later date, when the President is able to complete and revise it.)

SIR WM. J. POPE said that they had met in the expectation of hearing a Presidential address which would summarise the ripe experience of one of the foremost and most successful of British chemical technologists; he ventured to think that their anticipations had not been disappointed. The address would be read with keen appreciation and would furnish material for serious thought to all interested in the aims and objects of the Society of Chemical Industry. The subject of chemical technology now loomed much larger in the public eye than had been the case six or seven years ago, and for a two-fold reason. In the first place, the war had taught the whole thinking portion of the world that the possession of an effective chemical industry was absolutely vital to the continued existence of any modern nation; secondly, the magnificent results achieved by British technical chemists during the war had taught the whole world that this country was easily capable of developing chemical manufacture in such a manner as to render the Empire entirely independent of foreign production. The vast British output of chemical products, many of which had been never before manufactured in this country, which had been worked up between 1914 and 1918, had resulted from the labours of men like their President, of men who, from the exacting nature of their occupations, had come but little into the public view.

Whilst proposing a vote of thanks to the President for his address he was sure they would wish him to express the sorrow of all present at the reason for Mr. Gray's absence; their President was now paying the penalty of overwork undertaken in the national service. He had written to him (Sir William) expressing regret at his inability to attend the annual meeting, and his absence to-day was causing him the most bitter disappointment.

They were fortunate in having been able to entrust the delivery of the Presidential address and the conduct of the annual meeting to one who was a veteran in the service of the Society. Professor Henry Louis was always ready to step into a breach and to give them the benefit of his wide experience and of his well-known businesslike mode of conducting affairs.

He desired to propose that a very hearty vote of thanks be passed to their President for his address, that they should express their sympathy with him in his illness and their hopes for his speedy recovery, and that they should thank their acting President, Professor Louis, for the manner in which he had presented the Presidential Address.

MR. WALTER REID seconded the votes of thanks, which were carried with acclamation.

THE CHAIRMAN thanked the meeting, saying it had been a pleasure to discharge the duties and that he was always ready to do his best for the Society.

MR. L. G. RADCLIFFE moved, and MR. JULIAN L. BAKER seconded the re-election of the auditors, Messrs. Price, Waterhouse and Co. This was agreed to.

At the close of the meeting the Chairman announced that the Scrutineers reported that the following were elected to the Council as the result of the ballot:—*President*: Sir William Pope. *Vice-Presidents*: Mr. John Gray, Mr. D. Lloyd Howard, Dr. S. Miall, and Mr. E. Thompson. *Ordinary Members of Council*: Dr. T. H. Butler, Mr. W. H. Coleman, Mr. F. H. Carr, and Dr. F. C. Garrett.

CONFERENCE ON BY-PRODUCT COKING.

On Tuesday afternoon a conference on by-product coking was held in the Chemical Lecture Theatre, Armstrong College. Prof. P. Phillips Bedson, in the chair.

THE CHAIRMAN said that it was impossible to over-exaggerate the importance of the complete utilisation of coal—a subject which, during the past few years, had been brought home by the increased cost of coal, but it was a matter for regret that it had taken us so long to appreciate and make headway in the practice of coking coal, especially as regards the recovery of by-products. Of the factors which had contributed to the backward condition of the British by-product coking industry the first was that in this country, and particularly in Durham, such a good supply of excellent coking coal was available. The second factor had been that in making iron it had been found that a slightly larger amount of retort oven coke was needed than of beehive coke, and that undoubted experimental fact, supported by the dictum of Sir Lothian Bell, had deterred many from embarking on the recovery of by-products when coking coal.

BY-PRODUCTS FROM COKE-OVENS.

BY E. W. SMITH.

The supply of by-products from coke-ovens has now assumed a position of great national importance, and much more remains to be done before the commercial and financial possibilities of by-product coking can be said to have reached their limit.

In beehive practice—which still persists in some

districts—not only were all the valuable by-products lost, but also the gas and a part of the top layers of coke amounting to about 10 per cent. The beehive oven will completely disappear when the methods adopted in by-product practice are such as to provide a coke having the necessary physical characteristics of beehive coke for the particular industrial requirements and when some of the users of beehive coke realise that not all the characteristics of this coke are essential to their particular operation. Much uncalled-for prejudice exists amongst furnacemen in favour of the beehive coke; by-product coke can be made which, when properly used, will give equal results with beehive coke.

The following are authentic figures prepared by the National Benzol Association from official returns:—

Statistics of coke oven plants for 1918.

Tons of coal carbonised	14,635,403.
Galls. crude 65s benzol produced	32,162,598.
Galls. crude 65s benzol per ton coal (average)	2.18
Tons of coke produced	10,552,648.
Galls. of tar produced	102,781,090.
Galls. per ton of coal (average)	7.0
Tons sulphate of ammonia produced	134,357.
Tons concentrated ammonia (25%) produced	23,899.
Pounds of 25% ammonia, total per ton, calculated from above two figures	24 0

The actual number of by-product ovens in use may be seen from the following figures:—1914, 5893; 1915, 6491; 1916, 7719; 1917, 8219; 1918, 8412; and over 1000 further ovens were under construction during 1919.

From the above figures it would appear that even after taking into consideration the necessarily different conditions between coking and gas-making practices the yields of tar, benzol, and ammonia are low.

In by-product coking, gas is the chief by-product. In all cases about 60% of the stripped gas is used for heating the ovens, and the residual gas is used for steam raising and other heating operations on the works. Because this gas is looked on as "surplus" gas, very little attention is paid to the question of the efficiency of its use on the works.

The present methods of steam raising at coke ovens—with gas in Lancashire boilers—are not only in most cases wasteful because the gas might be put to more profitable use and the steam be raised by cheaper means, but in most cases the gas is inefficiently applied for steam raising. It is only where there is no other possible outlet that it is justifiable to use it for steam raising, and even though there be an ample supply of surplus gas for the whole needs of the works it will still pay to obtain a higher efficiency from the boilers.

Every conceivable opportunity should be seized for supplying coke-oven gas to gas undertakings that are requiring extensions. The gas is practically identical with that produced in horizontal retorts on gas works, and there are no "snags," as has been proved during the last eight years at the Saltley Gasworks, Birmingham, where there is an installation of 66 regenerative ovens taking a charge of between 9 and 10 tons of coal per oven. The yields of tar, ammonia, and benzol are quite up to the average of the horizontal retort results. Coke-ovens have this surplus gas, and it only requires freeing from hydrogen sulphide to make it fit for use in towns supply. If coke oven proprietors desire to sell their gas as a by-product for towns supply it will be necessary to study the subject from the consumer's point of view, as the operation is not one merely of piping the surplus to the inlet of the gas undertaking's purifiers.

The conditions of sale of gas for towns supply have radically changed during the last few years, and now that gas is to be sold on the basis of thermal units and not by cubic feet there is not quite the same latitude that there has been unless

a substantial B.Th.U. "overweight" be maintained. By means of recorders, official State Gas Examiners will test the towns supply and have records of the quality of the gas for every hour of the day. Consequently, as it is not possible to keep an absolutely constant quality, gas undertakings will have to work for a gas consistently above the declared standard, and as the charges are based on the multiple of cubic feet and declared calorific value all in excess is "overweight."

One of the difficulties coke ovens will have to contend with is the question of inerts—nitrogen and carbon dioxide. Although the Research Committee of the Leeds University and the Institution of Gas Engineers have shown, after exhaustive experimental work, that similar efficiencies are obtainable from gases varying widely in their inert constituents, no gas apparatus can adjust itself for changes in quality. The question of inerts is not, therefore, one of thermal efficiency, but one of density. A straight coke-oven gas stripped of benzol and containing no more nitrogen than that which came from the coal would have a density between 0.42 and 0.45. With about 10% nitrogen it would be between 0.48 and 0.51, and with 20%, 0.51—0.59. Not only does the calorific value usually vary correspondingly, but, with the same pressure, actually a smaller volume of gas can be consumed in the apparatus when the density of the gas increases.

The ideal to be aimed at is to produce and deliver to the consumer's premises a gas under as nearly as possible constant conditions, and the only factors of material moment are calorific value, density, and pressure.

Gas high in inerts, though no inconvenience to the consumer when paid for on a heat-unit basis, is a nuisance when the percentage of inerts varies considerably. It is also more costly to distribute and purify, and pressure changes in the distributing system are much more frequent.

In straight carbonisation practice such as that in horizontal retorts, B.Th.U.s. are dearer when associated with 30% of inerts than when associated with 15% of inerts—it being assumed that the ovens and retorts are in a reasonable state of repair and that the inerts come from the retort house.

Inerts in coke-oven gas are usually higher than in towns gas. This is due to the retorts being tighter than ovens and to better means being adopted on gasworks for controlling the pressure of gas as it leaves the retorts than is maintained at the offtake of the ovens. This is a matter that can be remedied.

Lastly, on this question of gas as a by-product, I am convinced that when large extensions of ovens are contemplated and where there is an outlet for the gas produced for boosting up the towns supply, the question of heating ovens by means of externally produced by-product recovery producer-gas should be fully considered, with a view to releasing for towns use not 40% of the make of gas, but the whole of it.

In the subsequent treatment of the crude gas entering the foul main the methods adopted on coke-ovens and in gasworks vary very considerably. On coke ovens the aim is to extract efficiently tar, ammonia, and benzol. On gasworks, tar, sometimes cyanogen, ammonia, occasionally benzol, and always hydrogen sulphide. Tar is eliminated by condensation and "frictional" devices, and usually finally by strong liquor washing.

Pitching troubles are usual on coke ovens and in gas works practice where the carbonising chambers or offtakes are leaky and the gas entering the collecting main is overheated. Short offtake pipes are also frequent causes of pitching troubles, and high ammonium chloride content of hot foul gas aggravates them. The circumstances of each case have to be studied separately, as there is no panacea

for the cure of pitching or emulsions. Not only are the troubles costly to handle, but the tar produced would, in normal times, be of much less value.

Tars from coke ovens are dealt with in a very similar way to those from gasworks. It would appear, however, that the temperatures attained during carbonisation are lower in coke ovens than on gasworks, although the long soaking heats usual in a coke oven make up for this difference somewhat. I believe it would prove of financial advantage to run many installations at higher temperatures in the combustion chambers—to 1300° C. Much lower temperatures than these are normal. There would be a greater throughput, a better gas, and better by-products.

I am still to be convinced that stamped charges containing 15–20% of water are so frequently necessary. They certainly do not improve the by-products, and are in many cases of doubtful necessity from the point of view of the coke.

Sulphate of ammonia.

Although it is the usual practice to produce sulphate of ammonia on coke ovens, in many cases it would be more profitable to make pure concentrated ammonia liquor. It is an open question whether it would pay, however, at the present time to scrap plants which were installed when capital costs were low and replace with plants at such high capital costs as are ruling at the present time. In the case of completely new installations this matter should be given very full consideration.

It is usual to maintain the gas entering the saturator at a temperature of at least 67° C., and the liquor in the saturator is still further heated by means of steam-heated lead coils. The heat of reaction inside the saturator also helps to maintain the temperature, and in this way a highly concentrated hot sludge is made, which, when released, drained, washed, and centrifuged, leaves a salt containing from 24% to 25% NH_3 , about 0.1% of acid and up to 2% of water. If this country is desirous of retaining the foreign markets wrested from Germany during the war greater care will have to be exercised by most makers of sulphate in the production of a dry, neutral salt. Higher prices are obtainable for such a product which cover the extra expense entailed.

In gasworks' practice it is usual to extract the hydrogen sulphide present in the crude gas. The manufacturing cost of this purification in the usual oxide purifiers is about 0.1d. per 100 cb. ft., after taking credit for the sulphur in the spent oxide. The sulphur recovered is more than sufficient for the whole production of ammonium sulphate.

On coke-ovens this sulphur is not recovered, but if the whole of the gas produced was used for towns supply the sulphur would have to be eliminated and would be available for acid production.

The present capital cost of oxide purifiers, including everything, is about £20,000 per million cb. ft. of gas made per day, so that there is ample room for new methods that can displace those ruling at present at a lower capital cost. Much work is being done on the subject both here and in America, and it would appear that a big advance will be forthcoming.

In some oven installations ammonium chloride is being successfully extracted from the hot gas before the free ammonia comes down. This is usually carried out at the tar extractors, and not only is there an enhanced financial gain but the tars are as a result comparatively free from chloride and the tar stills have a correspondingly increased life.

It would appear that the amount of ammonia recovered in coke-ovens is not so high as in gasworks from similar coals. This may be due to a number of causes—most of which are controllable. It is beyond the scope of this paper to go into this question more than to mention it.

Benzol.

During the war, gasworks starting *de novo* had opportunities of studying the question of benzol extraction which had not been available to coke-oven managers. Rhead's paper on the working of the benzol plant in Birmingham added much to our knowledge on this subject.

The quantity of benzol recovered depends (1) on the quantity produced in the ovens, (2) on the quantity destroyed either in the ovens or the offtake pipes, and (3) on the efficiency of extraction from the gas in the scrubbers. Higher temperatures of carbonisation will improve the first. Leakage in the ovens is largely the cause of the second, and high temperature of wash oil and unsuitable oil are largely the causes of low efficiency of extraction.

While coke-ovens are, in the main, not selling their gas for towns supply, the extraction of benzol makes very little difference to the financial value of the gas. In gasworks practice the stripped gas is equally as good as unstripped gas for towns supply, but the number of B.Th.U. sold per ton of coal is less when benzol is extracted than when the gas is sold unwashed. The calorific value is reduced about 13 B.Th.U. per cb. ft. per gallon on a gas of 530 B.Th.U. gross. The loss is worse than it seems where water-gas is used to any extent and the standard of quality to be maintained is normal. The value of the B.Th.U. is not necessarily that of the coal gas B.Th.U., but may conceivably be that of the alternative enrichment. Consequently gasworks at the present time must charge against the cost of extraction of benzol the value of the gross B.Th.U. to the gas undertaking as gaseous B.Th.U. This aspect of the question need never be taken into account on coke ovens as the gas is a by-product.

If it were not for the fact that gas undertakings must eliminate naphthalene from the gas distributed, it is doubtful whether it pays them to wash for benzol at the present time, but the circumstances of each particular undertaking must be taken into account before any conclusion can be arrived at.

Alcohol and cyanide as by-products are new to coke-oven practice. It must suffice to say here that as regards alcohol, the process being developed by Mr. Ernest Bury shows great promise, but there appear to be difficulties in connexion with the loss of acid and quality of fuel that have still to be surmounted. As regards cyanogen—where temperatures are maintained reasonably high in normal times its extraction would be profitable, but present capital costs of plant make them prohibitive.

Discussion.

Mr. H. E. WRIGHT said that Dr. Smith had spoken of 40% surplus gas in the coke-ovens. In the newer regenerative types it was more like 50–55%, and in the waste heat type 25–28%, depending on the quality of the coal used.

Mr. A. E. F. KNOTT said that he thought the losses in the by-product oven as compared with the beehive oven were nearer 5% than 10%. The coking temperatures given by Dr. Smith appeared more like the American practice. No doubt there were many good features in that method, but he did not think such temperatures could be adopted in England without rebuilding with a class of silica bricks which would withstand temperatures higher than any obtainable in England.

Mr. E. E. WOOD said that at the works at which he was engaged they had found that any coke which could be used in the ordinary cupola was suitable for the blast furnace; it was more difficult to get a good cupola coke from patent ovens than a good blast-furnace coke. Better coke was obtained by coking at lower temperatures and for longer periods.

Mr. S. A. WICKNER considered that more attention should be paid to keeping ammonium chloride

out of tar. When present it was difficult to remove and became a great nuisance.

Mr. W. DIAMOND said that there were burners now on the market which would burn coke-oven gas quite efficiently under boilers.

Dr. SMITH, in reply, said that the figure of 40% for surplus gas that he had given was in connexion with the average oven. Regarding the use of high temperatures in ovens, he said that at Saltley Gasworks, Birmingham, the ovens were kept at the high temperatures he had named; there was not much fear of the cracking of the hydrocarbons passing through the heated chamber at the top of the oven. At low temperatures benzol was not produced in such quantities as at high temperatures; the amount produced compared very favourably with the best horizontal retort practice. That indicated that high temperatures were not a deterrent. They used English bricks—he believed Leeds silica bricks—and the life of the ovens was quite satisfactory. In his opinion, if 1s. 6d. could be obtained for coke-oven gas for town supply it was wasteful to use it on boilers.

THE CORROSION OF COKE-OVEN WALLS.

By W. J. REES, F.I.C.

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In some of the coke-producing areas (for example, South Yorkshire and North Derbyshire) the corrosion of coke-oven walls due to the presence of salt in the coal is a serious factor in its effect on the life of the oven. In the Sheffield district, when deep coals are coked, this corrosion is very severe. In a particular case where coals from the following seams were being coked:—The High Hazels seam, at a depth of 120–130 yds.; the Barnsley seam, at 216–316 yds.; the Parkgate seam, at 460–560 yds., and the Silkstone seam, at 540–640 yds., extraction by water showed 0.37% of NaCl and 0.19% of Na_2SO_4 in the unwashed slack and 0.18% of NaCl and 0.08% Na_2SO_4 in the washed slack as delivered to the ovens. With continued extraction with hot 10% nitric acid, further quantities of both chloride and sulphate were removed from the slack, in several cases nearly 1% of NaCl being extracted. The pit waters frequently used in the washery contain large quantities of dissolved salts, as shown in the following table:—

	Pit water from pumping station	Water in circulation in washery	Drainage water from washed slack hoppers.
	Grams per litre.		
NaCl	0.451	1.529	1.944
Na_2SO_4	0.149	0.521	0.768
K_2SO_4	0.052	0.062	0.072
CaSO_4	0.243	0.168	0.165
MgSO_4	0.357	0.264	0.261
CaCO_3	0.095	0.057	0.062
MgCO_3	0.051	0.041	0.023

and the question arose as to whether the use of a pure water, such as that of a town supply, would be advantageous. Laboratory experiments showed that there was little or no gain in either the rate of extraction or salt extracted by the substitution of either distilled or town's water for the impure pit water. The use of a purer water would, of course, result in some diminution in the quantity of dissolved salts sent to the oven in the water adhering to the slack, but the expenditure involved would not be justified by the comparatively small reduction obtained. The rate of flow of water through the washery is a point which may be worth consideration where very salty coals are being washed, as with an increase in the rate of flow there will be a reduction in the concentration due to the solution of salts from the coal and a corresponding reduction

in the salt content of the water adhering to the slack. In some pit and hopper drainage waters considerable quantities of magnesium chloride have been found and, in view of its ready hydrolysis, this may have an important effect on the coke-oven walls.

Taking the figures given above, there will be carried into the oven with each charge of slack and adhering water approximately 50 lb. of NaCl and 20 lb. of Na_2SO_4 . The salt present will begin to volatilise at 800° C. (to a small extent at lower temperatures, as salt has an appreciable vapour pressure at temperatures below its melting point) and in addition to this removal of salt by volatilisation there will be some removed by hydrolysis as steam from the centre of the charge comes in contact with the hot coal. The sodium sulphate present will also probably have some activity as a corrodant, as by interaction with the coal at the temperature of coking, sodium carbonate will be formed. H. V. Thompson,* in discussing the dissociation of salt, has shown that when a current of dry air is passed over molten salt at 1100° C., the salt vapour carried forward by the air was 0.0554 g. per litre. With a current of moist air, the salt carried over was increased to 0.0782 g. per l. The coking of wet slacks may thus be a factor of importance in cases of oven-corrosion. The moist air with the salt vapour and the products of hydrolysis was then passed over (a) quartz chippings, (b) ferric oxide, (c) alumina, (d) clay. With the quartz there was a superficial attack only, a little sodium silicate being formed; with the ferric oxide, a little ferric chloride was formed, and there was, incidentally, a conversion of the bulk of the Fe_2O_3 into black crystals of Fe_3O_4 ; with the alumina, the attack was even less than on the quartz, a little sodium aluminate being formed. In the case of the clay, however, severe corrosion was observed, increasing with the iron content of the clay. In a paper recently communicated to the Refractories Section of the Ceramic Society† I have described a series of experiments in which the action of salt vapour (at 800°–950° C.) on silica, silicious fireclay, semi-silica and fireclay bricks was observed. The attack was, in general, much greater on the fireclay than on the other bricks.

The action of salt on clay has been extensively studied in connexion with the phenomena of salt glazing, and this directs attention to an important difference between the conditions of salt-glazing and the conditions which result in the corrosion of fire-clay coke-oven bricks. In salt-glazing, the kiln atmosphere will be much richer in salt vapour than a coke-oven at any period, but the salt is not applied until the articles to be glazed are at a temperature of about 1200° C. At this temperature the salt immediately combines with the clay, forming a glass which coats the exposed surface of clay and protects it from any further attack by the salt vapour. The important factor in salt glazing is that the articles to be glazed shall be at the temperature at which interaction between salt and clay is rapid. In the coke-oven the conditions are essentially different, the internal wall of the oven never reaching the temperature at which salt glazing is rapid. J. W. Cobb‡ has shown that interaction between sodium carbonate, alumina, and silica will take place at temperatures much below that of fusion of the mixtures, and it is evident that this type of reaction is important in coke-oven corrosion. In the coke-oven, the salt vapour instead of reacting with the surface of the bricks will penetrate them; there may be some deposition of salt inside the brick, but a good deal of the vapour apparently passes on until it reaches a zone in the brickwork at which the temperature is high enough for interaction to take

* Trans. Ceramic Society, 1918; see J., 1918, 333 A.

† Spring Meeting, 1920.

‡ J., 1910, 69, 250, 335, 399, 603, 799.

place with some rapidity. The joint result of the slower interaction in the cooler portion of the brick and the more rapid action in the hotter zones is the formation of a weak, spongy mass which becomes increasingly open to the attack of the salt and which may fall away or be dragged away by the moving coal or coke. Another important phenomenon in connexion with this corrosion is the very general presence of a layer of hæmatite in that portion of the brick where disintegration and shearing take place. This appears to be due to the formation and volatilisation of ferric chloride by the action of salt vapour on iron oxide present either in the coal or more probably in the bricks. The ferric chloride vapour penetrates the porous bricks until it reaches a zone to which oxygen-containing gases from the combustion flues penetrate, and it is there oxidised with the formation and deposition of hæmatite. In this way layer upon layer of hæmatite may deposit until it reaches the thickness which may frequently be observed in old corroded bricks. The formation of ammonium chloride (and possibly also the presence of free chlorine) may also be a factor in corrosion; the passage of ammonium chloride vapour over fireclay bricks at coking temperature shows only very slight indications of corrosion of the brick, but that continued action may result in the formation of ammonium or ammonia-alumino-silicates is evident from the action of ammonium chloride in the Lawrence Smith method for the determination of alkalis in silicates. Attempts have been made to prevent the penetration of vapours or increase the resistance to corrosion by facing or glazing the surface of the bricks, but up to the present these have been unsuccessful, the face falling away owing to the difference in the coefficient of expansion of brick and face.

The question now arises as to which is the best type of brick to use for coke-oven walls. When the coals to be coked contain little or no salt, it is probable that a good, well-burned fireclay or fireclay with silica grog (semi-silica) brick will be satisfactory, but in the case of salty coals the experimental evidence indicates that corrosion is likely to be much more severe on fireclay than on silica, alumina, or magnesite bricks. With an alumina brick the physical changes will, during use, be in the direction of continuous contraction, and, unless an electric furnace product such as alundum is used this type of brick is therefore ruled out. There is at present no quantitative evidence which indicates whether or not the high initial cost of alundum or magnesite bricks would be counterbalanced by the increased life of the oven. The physical changes in a silica brick during use are rather complex, the most important of them being the growth due to the gradual transformation to tridymite of the quartz in the brick. But if, in burning the brick, a maximum conversion of the quartz to the form of lower specific gravity is obtained, it is evident that the increase in volume of the brick during its subsequent use can only be small. Further, if by special grading of the raw materials and the formation of a suitable matrix, the recrystallisation of the silica takes place so that the newly-formed crystals tend to interlace instead of to push against each other, the mechanical strength of the brick will be increased and its porosity reduced. The characteristics of the ordinary type of silica brick which render it unreliable for coke-oven use are its continued growth during use, its rather high porosity and its tendency to spall from the repeated changes in temperature; but it is now possible, by combining the two factors of suitable grading and adequate burning, to produce a silica brick with physical properties such as should make its successful use in coke oven practice a distinct probability. Such bricks are already being made and put into use in situations where corrosion trouble has been severe.

A factor which will be common to all coke-oven bricks in its influence on disintegration is that due to the deposition, in the pores of the brick, of carbon from the decomposition of hydrocarbons in the coke-oven gas or from the gases in the combustion flues. The reduction to the ferrous state of any iron oxide present and the possible formation of easily fusible ferrous silicate will also be a factor in the behaviour and life of the brick.

The heat conductivity of the brick used for the coke-oven needs some consideration. This is so much affected by factors such as the temperature of burning, porosity, etc., that no figure short of that obtained by actual determination on a particular brick is of any great value. Experience in American coke-oven practice indicates that at coking temperatures the conductivity of a good silica brick is decidedly greater than that of a fireclay or semi-silica brick. This should be a further point in favour of the use of silica bricks in coke-oven construction.

MODERN BY-PRODUCT COKE-OVEN CONSTRUCTION.

BY W. A. WARD.

In Great Britain even yet an inordinate amount of our metallurgical coke is produced in beehive ovens, although it is well known that the yield of coke from by-product ovens is much higher and the quality of the coke quite as good. This is due to the prejudice of iron manufacturers in favour of the larger coke generally yielded by beehive ovens and its appearance, and also to the higher capital cost of a by-product coking plant and the greater importance of continuous working.

It is, however, possible to approach nearer to the theoretical calorific intensity of combustion with smaller coke and thus make the most economical use of it because it will lie closer in the furnace and render possible a reduction of the amount of excess air passing through.

It must, nevertheless, be admitted that some of the by-product coke made in years past was very poor. It was uneven in quality and structure, soft, and contained too much volatile matter and moisture. These defects were in part due to the ovens, low uneven heats, leakage, and careless quenching. Quite as certainly, however, they were due to the use of unsuitable or badly prepared coal.

The suggestion is frequently made that we in Great Britain should pool our resources in the matter of coal supply and manufacture coke, by-products, and gas on a large scale in joint works. It would have enormous advantages in the mixing of coals, continuous operation at full load, reduction of working costs, products of uniform quality, and higher all-round efficiency.

The choice of site for a coking plant is a matter requiring very careful study. The first essential is the traffic in connexion with the plant and its incorporation as an integral part of the general traffic scheme of the works. Where no really suitable place from the traffic standpoint can be found on the pillar where there will be freedom from sinking due to mining operations, as also when no alternative site of any kind is available, resort must be made to the use of aerial ropeways, overhead conveyors, large ovens, and the recent developments in coke handling plant described later.

Future extension should always be considered and provided for when laying out the original scheme. Use should be made also of any natural advantages of position such as ground rising from the coke side which will cheapen the discharging machine terrace whilst not seriously interfering with the by-product roads behind.

Type of oven.

The choice of a type of coke-oven to be used at a by-product plant will be decided by the manner in which the surplus heat available is to be made use of, and no other consideration really need be taken into account.

It is often stated that regenerative ovens are more efficient than non-regenerative, because through regeneration a larger yield of surplus gas is available. *Ipsa facto* this is somewhat erroneous. There have been cases of such a difference, but they are due to defects in design or construction or else to faulty operation. The surplus energy is the same, because the amount of heat necessary to coke the coal is the same, whatever the type of oven, unless its form be such as to increase losses by radiation etc., or the arrangements for combustion of the heating gas are faulty. The difference lies simply in the manner in which the surplus heat is made use of. By the provision of adequate heating surface and equal care in general design and insulation, the gases entering the stack may be at exactly the same temperature in either. In fact, generally there will be more excess air in regenerative oven flue gases which will carry a lot of heat to waste.

This being so, the difference in power available for use outside the plant and after all its demands have been met as between the two types, is simply the fundamental one as between the efficiency of the gas engine and the steam plant. It is impossible in practice to attain the same efficiency with the steam raising plant, but this is not the fault of the type of oven. Furthermore, steam is necessary on every complete plant whatever the main source of power, and this must be taken into account when drawing up a complete energy diagram.

Generally speaking no attempt is made on coking plants to make full use of the hot waste gases from the ovens, whether these be regenerative or non-regenerative. It is quite common to find gases entering the chimney at 310° C. from regenerative ovens and 460° C. from waste heat ovens and neither steam superheaters nor economisers in use; 250°—280° is sufficient if the chimney and flues are correctly designed. Again gas will often be burnt for the purpose of drying, subliming, evaporating, etc., while these "waste" gases are doing nothing. Of course, they cannot be used any further for direct steam raising in the boilers, but the obvious solution is to use them for superheating the steam and raising the temperature of feed water etc., extensions of the total heating surface which are common in almost all other industries and which are suitable for the low temperature differences. They might also be used for superheating exhaust steam, from back pressure turbines, or ordinary reciprocating engines which could then be used in the by-product plant or for reheating gases etc.

Superheated steam should be more generally used on coking plants not only for power purposes, but in ordinary process work where it is most important that the steam should arrive at apparatus as steam with some latent heat to give up, and not merely as hot water.

An extension of heating surface outside the non-regenerative or non-recuperative oven would make it approach nearer to the heating surface of the regenerative oven, which came into being through this necessity for heating surface suitable for abstracting heat from the tail-gases.

Another very important point on this question of heating surface is that presented in the oven wall and the application of heat to it. This will be referred to later.

The non-regenerative or waste heat oven cannot be economically used unless there is a continuous and regular demand for steam, and the boilers can be placed close to the battery of ovens.

In the case of intermittent loads or wide fluctuation, regenerative ovens should be chosen

whether power is generated direct in gas engines or by steam, because the surplus gas can be transmitted almost any distance and stored in a gas-holder until required. This applies also when the steam raising plant is remote from the ovens.

Many waste heat ovens are arranged on the recuperative principle. That is to say an attempt is made to recover heat which would otherwise be lost by radiation from the brickwork of the oven foundations into the atmosphere, or by conduction into the ground. At the same time the current of air for combustion of the gas by which it is done serves to keep the galleries in which the gasmen have to work in a moderately cool and comfortable state. Generally recuperation is carried no further than this at the present day, although in the past, as in the old Simon-Carves oven, special flues were constructed for the purpose.

A novel form of producer-gas-fired oven with continuous recuperation for both gas and air has recently been introduced by Messrs. Simon Carves, Limited, and a small trial battery erected at Cargo Fleet Ironworks, nr. Middlesbrough. I am not able to give details at present, but the principle has been carried much further than ever before.

The producer- or blast-furnace-gas-fired oven of regenerative type, both simple and compound, for the use of poor or rich gas or both, is an accomplished fact.

There is no fundamental reason for any great difference in results to be obtained from the various types of oven, and it is undoubtedly true that very good results are regularly obtained from all types. The best are frequently obtained from ovens which would appear of inferior design. This, however, is to the credit of the working staff. A good feature in one type will often be counter-balanced by one of a different kind in another type not containing the first. For instance, distribution of heat may be more even and easier to maintain in that state in a particular type of regenerative oven than, say, in a waste heat oven with horizontal flues, owing to the greater volume of hot gases and air at the point where distribution to the walls takes place. This may be balanced by the fact that there is a larger mass of hot brickwork in the horizontal flue oven walls to act with a fly-wheel effect, also that the gas tuyères are more accessible and of better type permitting of a more thorough primary mixture of gas and air and a nicer regulation of the amount of excess air.

Description of various types of oven.

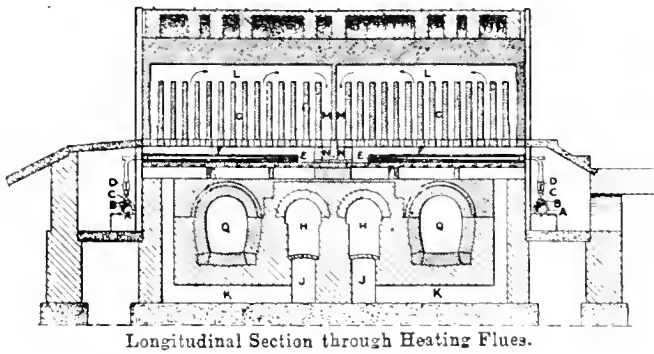
The Coppée waste heat oven (fig. 1) is of the vertical flue type having dual waste gas flues. Each half of each oven wall is entirely separate from the other with regard to gas and air supply and regulation and served by one of the aforementioned waste gas flues. This arrangement is excellent as tending towards even distribution of heat, even expansion of the battery and distribution of weight on the ground, and permitting very good recuperation by the air for combustion and insulation of the foundations at the same time. It is heated by rich gas brought along each side of the battery in mains, A, from which separate branch pipes, B, lead to gas conduits, E, under the centre of each oven wall. Before reaching there the gas passes through mixing boxes, D, designed on the Bunsen burner principle, and primary air is thoroughly mixed with it. These mixing boxes are each capable of regulation by the gas man as he watches ignition through a convenient sight-hole in the gas tuyère. Secondary air for combustion of the gas enters the flues or conduits, H, at each end of the battery and passes into distributing flues, J, from which small conduits, K, lead to each oven. These conduits each feed three air-distributing canals communicating with three groups of vertical flues, making six

groups per wall. Dampers are provided in each canal for regulation.

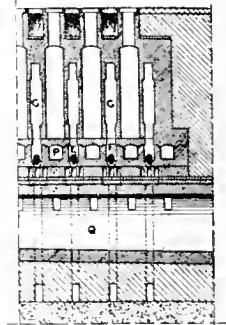
Each waste gas flue is completely surrounded with air ducts, so that full advantage is taken of the heat otherwise lost from the substructure.

Fig. 2 shows by vertical sections the design and arrangement of the Coppée regenerative oven. It is of the vertical flue type, and is built on similar

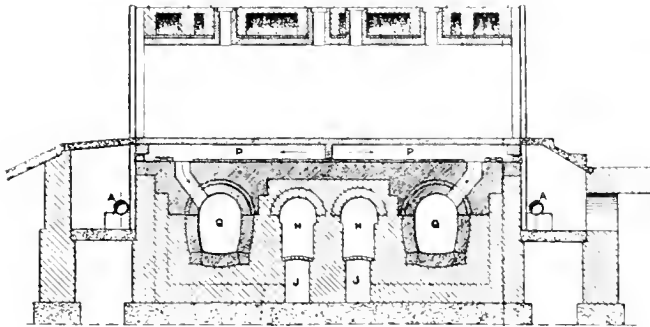
duits lead to each oven. This arrangement permits of a solid construction of practically the same class of material throughout, and as the whole of the substructure is kept at approximately the same temperature there is no unequal expansion with its consequent distortion and cracking etc. The air is drawn by means of a fan through galleries and ducts under the regenerators and collecting



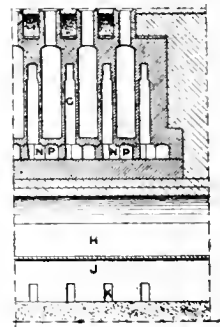
Longitudinal Section through Heating Flues.



Cross Section through Ovens and Heating Flues.

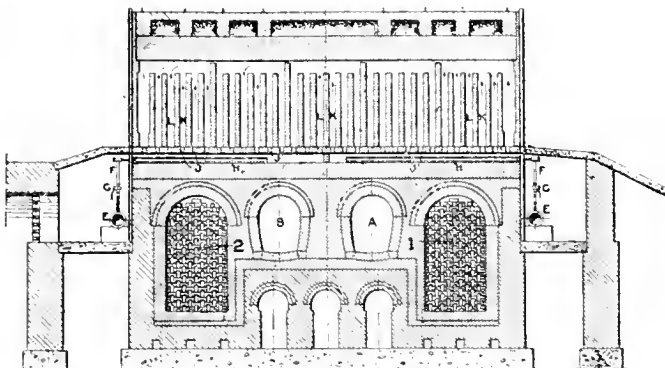


Longitudinal Section through Oven Chamber.

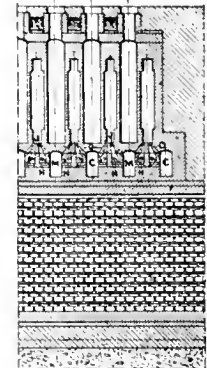


Reversal of the Heating Process.

Fig. 1.



Longitudinal Section through Heating Flues.



Cross Section through Ovens and Heating Flues.

Fig. 2.

lines to the waste heat oven except that the air for combustion of the gas is heated in the regenerators to an intense degree, viz., over 1000°C .

Unlike most other types, the air passes through the whole length of the regenerator and then into collecting flues, from which the distributing con-

duits lead to each oven. This arrangement permits of a solid construction of practically the same class of material throughout, and as the whole of the substructure is kept at approximately the same temperature there is no unequal expansion with its consequent distortion and cracking etc. The air is drawn by means of a fan through galleries and ducts under the regenerators and collecting

flues, so receiving a preliminary heating and serving to keep the foundations and ground reasonably cool.

The weight of the oven structure and mountings, also of the coal in the ovens, is distributed on arches and piers running at right angles to them;

this feature, which is shared by other designs of oven such as the Simon-Carves, is the most correct from an engineering standpoint.

The 30 vertical flues in the oven wall are divided into five groups. The six flues of each group are divided into two groups of three, each being used alternately for ascending and descending gases by simple reversal of the damper.

There are two gas tuyères on each side of the oven with cocks for reversing, in addition to valves for regulating and shutting off the supply. The reversing cocks are all operated simultaneously by levers and a wire rope when the main damper is reversed.

The distribution of the 15 flues in which gas is burning, throughout the entire length of each wall, and the alternation of these in adjacent walls, effect a very even distribution of the heat; this is an improvement on the systems in which the whole of the flues in one half the oven are used for ascending gas and all those in the other half are used for descending burnt gas. There is also less tendency to overheating at the top due to the passage of very large volumes of hot gases through a more or less restricted space. This is of advantage in preventing the decomposition or degradation of by-products. The same effect is obtained in this and other ovens by keeping the top flue some distance below the crown of the oven.

The ovens in a battery like this are not independent either in air supply, regenerators, or combustion of the gases.

Another type is built with separate regenerators for each oven arranged under the oven chamber and athwart the battery. This is a later development, and is common to most firms of coke-oven engineers. The advantages claimed are, a greater regenerator capacity in the same space or equal capacity in less, complete independence of each oven so that the others are not affected if some are off for repairs, or "sick" for any reason, better facilities for examination and repairs to regenerators, better regulation of draught and air supply. The last-named is probably the greatest, because air supply and draught are regulated separately instead of relying on one damper setting more or less permanent to regulate draught and air successively on reversal as in the older types.

All the advantages do not rest with the newer type, however. Stability has had to be sacrificed to a certain extent, and much greater care has to be exercised in connexion with longitudinal expansion of the battery. Giving to each oven a separate existence precludes help being given to a "sick" oven by the remainder. Further, the very facilities for independent adjustment and the more accurate regulation and distribution of heat, which make a larger output and better general quality of both coke and by-products possible, are in themselves a danger if neglected or left in the hands of men not properly instructed or in any way uncertain of the results likely to follow changes they make in regulation.

In connexion with the problem of stability, I would draw attention to the five longitudinal walls in the Coppée ovens of this type, the retention of the arches and piers in the oven designed by Messrs. Coke and Gas Ovens, Ltd., and also to the retention of a central flue or duct in the original Koppers oven. Provision is also being made for expansion and stability by shape, arrangement, and bond of blocks.

The Coppée independent regenerator oven is divided into four sections athwart the battery as regards arrangements for heating. Instead of one-half the flues in each wall being used for ascending hot gases in the initial stage of combustion and the other for descending gases as in other types, two alternate sections are used in each case. The

aim is a more even distribution of heat and a smaller area of the oven affected by changes due to reversing. Dampers control the inlet of air and exit of waste gas, and reversing is effected by cocks and dampers operated by wire ropes from a winch at the end of the battery. The gas supply is arranged in a similar way to that of their older oven previously described, except that there is of course no primary air admission.

These arrangements appear to make it possible to regulate the amount of gas burnt to compensate for the natural tendency towards a hot zone at the centre of the oven due to radiation losses from the ends, and also for the extra heat required to coke the greater thickness of coal at the discharge or coke bench side of the oven, or the greater density at the narrow end due to withdrawing of the peel in the case of an oven charged by coke.

The Coppée compound oven is modelled on similar lines save that each regenerator chamber is divided into two parts by additional walls running from side to side of the battery, thus providing regenerators for both producer- or blast-furnace-gas and air. It is necessary, owing to the low calorific value of these gases to raise their temperature to about 1000° C. before ignition.

The producer- or blast-furnace-gas is brought along each side of the battery in the regulating galleries, and specially designed connexions convey it to the inlets at the bottom of each regenerator. Reversing of gas and air is carried out by wire rope gear as previously described.

The distribution of gas and air to the vertical heating flues in the oven walls is effected by inclined ducts from a compound sole flue as before, and waste gases follow the same course in the reverse direction, thus heating up the chequer work before entering the waste gas flue, ready for the next reversal.

In addition to the arrangements for heating by means of poor gas, there are supply pipes and tuyères with reversing valves for rich gas, so that if for any reason a change becomes necessary, a few minutes is sufficient to convert the oven to the ordinary regenerative type using a portion of the gases evolved from its own charge. In this event a simple device enables the whole of the regenerators to be used for air in the usual way. Furthermore, a small amount of this rich gas can be used at any time along with poor gas if there should be a falling off in supply or value. It can also be used to stimulate a backward oven, and so enable the regular programme to be maintained.

There is no doubt that the compound oven is the oven of the future, whatever the design, because of the greater value of the rich gas set at liberty for other purposes such as lighting, power, steel manufacture, and chemical processes. With the poor gas oven the whole of the gas evolved from the charge is available after recovery of by-products for extraneous purposes, whereas from the ordinary regenerative oven only 45–55% is available for purposes outside its own demands. In the case of the non-regenerative or waste heat oven no more than 5–25% of "live" gas is available, according to the natural yield of the coal and the extent to which recuperation is carried. It is often assumed that the percentage of spare gas will be practically the same for all coals, but this is not so. The losses from ovens of the same design are approximately constant.

Coke breeze or "bad coal" can be used in the producers for the supply of gas for heating purposes, and I believe actual experience has shown that the objection at first raised to ovens of this type that thermally and economically there was no advantage is groundless. In fact the yield is found to be higher from ovens of this kind because the greater volume of the gas and air mixture per equal number of calories effects a better and more

even distribution of heat, and owing to the specific heat of the waste gases less heat is lost via the chimney. R. Biermann has shown that a reduction of 7% in coking time is effected.

If blast-furnace gas is used it must be cleaned thoroughly, so that the mains and valves will not become clogged with tar, or fine dust containing compounds which will act as fluxes, choke the regenerators or damage the oven brickwork. There is not the risk of naphthalene blockage or carbon deposits as with the use of coke-oven gas.

spyhole, giving an uninterrupted view of the bottoms of all vertical flues in each half of the wall.

The patent covered the control of each separate vertical flue with its gas jet from the outside, or any arrangement of them into groups. In practice groups were adopted as it was too costly to arrange for each flue separately, and a finer division with multiplication of controls was judged to be unnecessary. These groups were arranged so that the amount of gas burnt in each was proportional

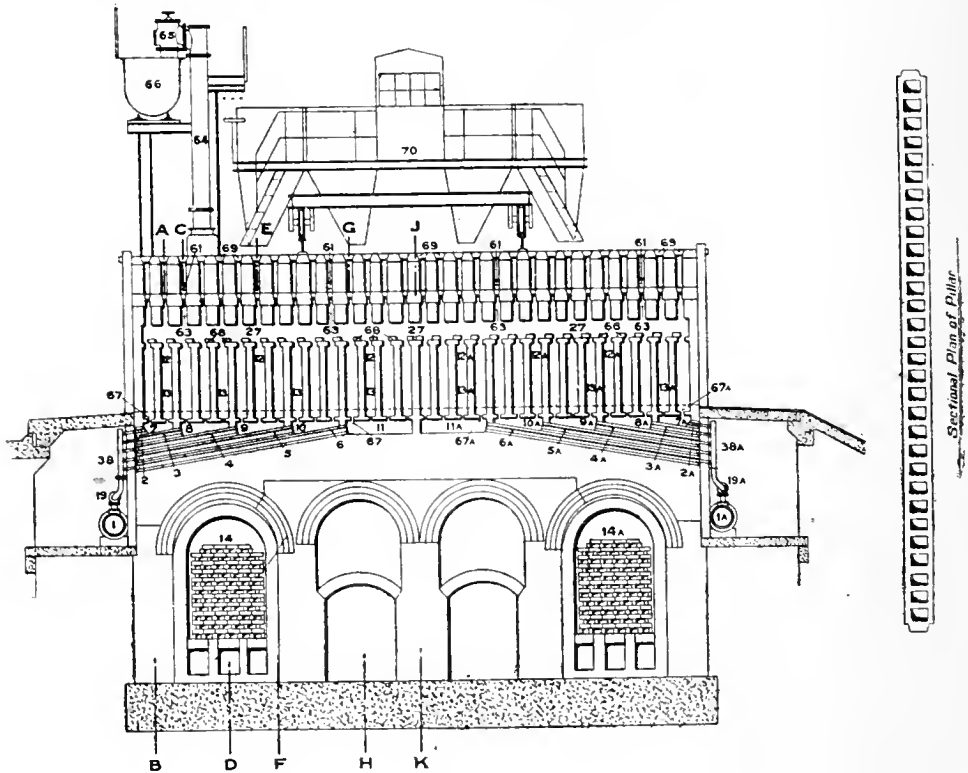


Fig. 3.

The Simon-Carves vertical flue regenerative oven, shown by figs. 3, 4, and 5, on its introduction about 1906 showed a marked advance in the matter of general design, especially the arrangements for distribution of gas and air, regulation of draught, accessibility, and facilities for continuous inspection during use. Some improvements have since been made, but substantially the design is the same, and it still holds a very high place in the coking world, giving results which compare favourably with those of other designs possibly more attractive on paper.

The fundamental difference in design was that the controls were all brought to the front and back of the oven and arranged in galleries under the discharging machine track and the coke-bench. Previous to this it had been usual to regulate the admission of gas and air by means of dampers or adjustable blocks and nozzles situated at the base of each vertical flue from galleries under the battery, or by rods inserted through openings in the brickwork. In practice this was so difficult and often uncomfortable owing to the heat and cramped space that it was neglected or improperly carried out.

The new arrangement meant that the nicest regulation could be effected in a few minutes, while the result was watched through a convenient

to the demands in order to make up for radiation losses and difference in thickness or density of the charge. The draught in each of these groups of flues was separately regulated at the outlets of the several ducts in the sole flue with which they communicate on the outlet side.

Further improvements in this oven are the dividing off of each group of vertical flues at their base and the use of gas canals of large section each provided with a removable plug in the gas distributing box, 38, through which each section can be separately examined.

In addition, means have been adopted for checking and regulating the draught at the top of each vertical flue. The last named is almost universal now in vertical flue oven practice.

The oven structure is supported on five massive brick piers and four arches forming tunnels running the length of the battery and at right-angles to the oven walls, thus forming the stable construction referred to earlier.

The regenerators, 14 and 14a, are accommodated in the two outer tunnels and built so as to be free to expand within same. The air for combustion of the gas traverses the two inner tunnels and enters the canals under each regenerator in turn, traversing freely from end to end and up through the deep mass of chequer work to each oven inlet,

15 and 15a, the correct amount being regulated by the setting of dampers, 59. There is no travel of either air or waste gas along the regenerator, itself, and therefore the heating surface is exactly the same for each oven and the friction uniform. No fan is used at these ovens, the chimney giving all draught required. Reversal of air and waste gas is by vertical dampers operated by a winch which simultaneously reverses all the gas tuyères.

From the air inlet, 15, compartments, 20 to 24 (fig. 4), are supplied through ducts, 17, 18, 29, and 30, which communicate with the bases of the vertical flues, 12, through inclined passages shown in transverse section in fig. 5. These it enters immediately above the gas and starts combustion about the sole level of the oven. The ducts, 61, 62, and 63, are for use when starting the ovens from fires in the oven chambers at each end, with which they communicate through openings in the sides of the charging holes. These openings are sealed with fireclay blocks when the oven is heated up and ready for normal work.

The Simon-Carves vertical flue non-regenerative

The oven is partly recuperative, the air being led through the two central galleries and over the tops of the waste gas flues to the distributing channels at the front and back of the oven where regulation is effected.

The well-known Simon-Carves horizontal flue waste heat oven is non-recuperative, and is arranged with one waste gas flue at the coke ram side of the battery. Its construction is very strong, the ovens being separated by a solid firebrick wall, usually 14 in. thick, which carries the superstructure, gas collecting main, charging car, etc., thus relieving the oven linings of any of this weight. The linings are tied into these central walls between each horizontal flue, the whole being made of tongued and grooved bricks so that an exceedingly strong and rigid structure not liable to distortion or leakage is the result. The ends of the walls at both ram and bench side are protected and maintained by massive cast-iron armour plates between the brickwork and steel buckstaves. The gas tuyères, air ports, and inspection plugs are attached to these armour plates by set screws, and

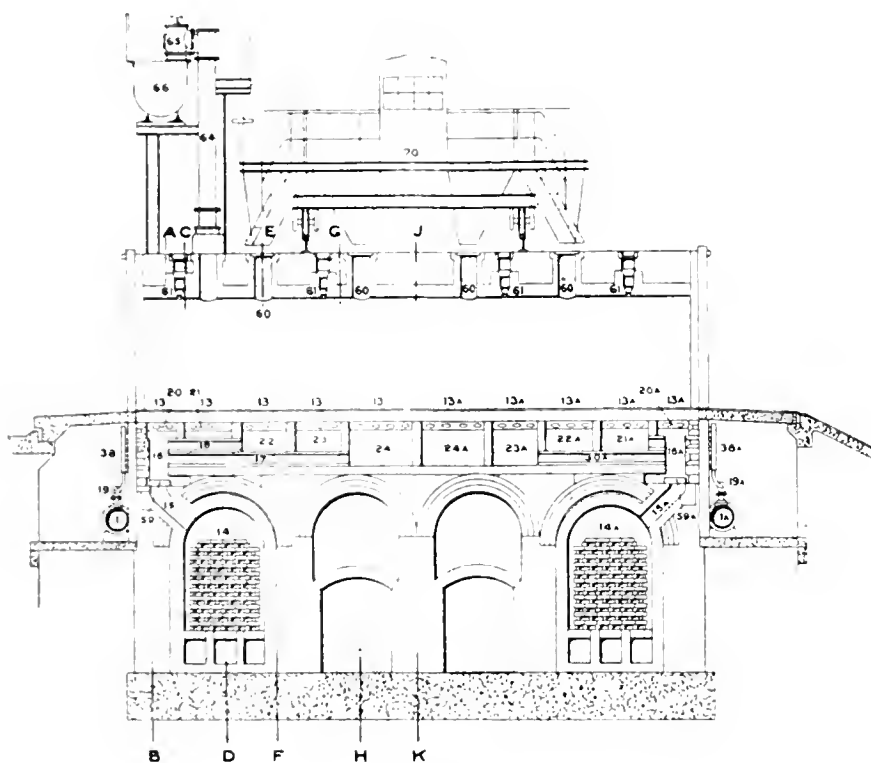


Fig. 4.

or waste heat oven is of similar design to the above—indeed it is covered by the same patents. In place of the regenerators there are two waste gas flues of smaller area. The arrangements for distribution of gas and air, regulation, and inspection are identical. The wall is divided into two halves, each served by one of the waste gas flues, and a number of the vertical flues at the centre of the wall (usually two) conduct the hot gases down to the sole flue and thence to the waste gas flues. The sole flue is not split up into sections, and the distributing canals for air and gas are side by side under the oven wall, but separated by an independent wall to render short-circuiting impossible.

are thus kept in place and proper alinement. By this arrangement the battery is less susceptible to the adverse influence of high winds in exposed situations, and retains its form at the doors with great advantage in the matter of luting and exclusion of air from the charge.

The gas and air for combustion are mixed in special tuyères referred to above, which are of Bunsen burner type and provided with regulators for both gas and air, before actually entering the oven flues. The intimate mixture so necessary is thus attained with certainty, and any extra oxygen which may be required in the lower flues due to partial saturation of the gases with CO , can be admitted. On the other hand, excess air can be

admitted to the top flue to avoid overheating of the oven crowns. By this means the heating effect of the gas can be increased considerably at the lower portion of the oven where the greatest amount is needed, without danger, as the lower air inlets give facilities for instant admission of excess air.

In practice it is scarcely possible to regulate and control combustion with such nicety as to obtain an absolutely even temperature throughout the length of the horizontal flues in this or any other oven, but the length of flame and facilities for regulation of air give a wide latitude and scope to the operator.

some form is common to nearly all designs of vertical flue ovens.

A second feature is the dividing off of each separate vertical flue from its neighbours at the bottom and provision of a separate gas nozzle in each which can be changed for regulation or renewal by means of a steel rod with key end through the top of the oven if necessary. A large sight-hole is provided outside the oven for inspection during operation.

The air entering the regenerators is evenly distributed over the bottom by means of ducts arranged and proportioned for this purpose, and rises evenly through the chequer-work to the dis-

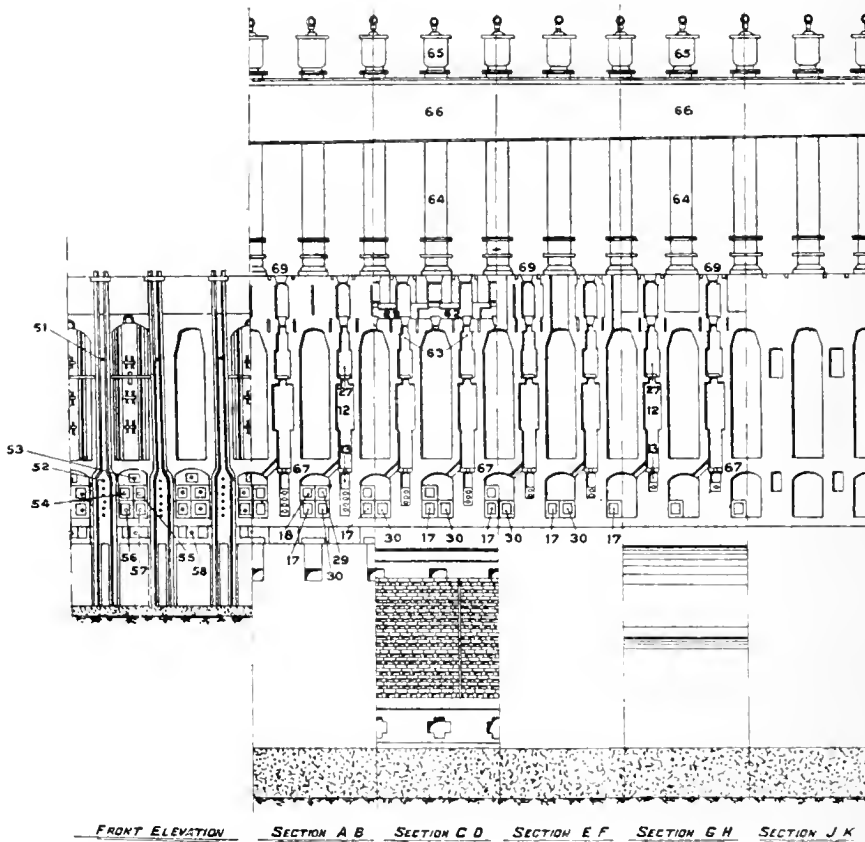


Fig. 5.

The type is still popular, the opinion being held that it is not so delicate as the vertical flue oven either constructionally or in heat regulation. The large mass of brickwork surrounding the oven chambers holds a tremendous reserve of heat and contributes in no small degree to this, enabling rapid recovery after recharging etc.

The Koppers regenerative oven is of the vertical flue type with separate regenerators, and displays originality and the fearless spirit of the pioneer at many points. First among these, in my opinion, is the provision made for regulating the draught in each separate vertical flue by means of an adjustable damper on the top of it. The patentee realised the difficulty presented by parallel flows, and overcame it by controlling the draught in each flue just beyond the point where even distribution was of importance, or, as he put it in his patent claim, "the point of attack" of the chimney or draught producer on the flue or group of flues. At the present day this device in

tributing chamber under the sole of the oven. From here it passes through inclined openings into the bottom of each vertical flue, which it enters above the gas. By this arrangement good admixture is effected, and deposits of carbon in the gas jets avoided. Any deposits at the air ports can of course be easily burned away, but these are unlikely. I believe in some batteries the central flue was used for carrying a portion of the waste gases off for steam-raising purposes.

In the later ovens the air for combustion is admitted to the regenerators through separate cast-iron change valve boxes instead of through the flues with a change valve at the end of the battery. These separate change valve boxes are operated by wire rope from an automatically operated throw-over gear in exactly the same way as the gas-reversing valves. They admit air in one position with the gas outlet closed, and when reversed allow the waste gas to get out into the flue whilst the air inlet is closed.

The burning gases ascend in one-half of the wall and descend the flues in the other half. The Koppers regenerative gas oven and compound oven are similar in general design and arrangement to the above. In these, however, each regenerator

in sets of two of the air and gas regenerators makes for simplicity in the change valve boxes, which are alternate for air and lean gas, and avoids cramping of the gear.

The Koppers vertical flue waste heat oven is similar in design to the regenerative oven with regard to the vertical flues and arrangements for distribution and regulation of gas, air, and draught, except that the air is only preheated to a moderate degree by its passage through the conduit under the coke ram side of the battery and the flues under the oven sole. Gas is brought along the coke ram side of the battery only in a main and fed to the gas canals under each wall by large tuyères, which can be arranged to admit a small amount of primary air and thoroughly mix it with the gas. The secondary or main air supply is distributed to each vertical flue by cross passages, entering just above the gas jet. The gas burns upwards in all except two of the vertical flues and, descending the two at the coke bench side, is led through an inclined duct to the main waste heat flue and so on through boilers to the stack.

The oven may be arranged with waste gas flue under the ram track and air admitted from an observation gallery under the coke bench. In this case only one of the vertical flues is used for descending waste gases. It is an arrangement suitable for compressed charges in which the density is greater against the door at the charging side of the oven.

The latest development in the Koppers coke oven, and one which can be applied to either type, concerns improvements in oven wall construction and aims at rapid and uniform coking by the more efficient application of heat without sacrifice of strength and stability. The vertical flues in the oven walls are triangular in plan and staggered, as shown in fig. 7. Two independent parallel gas canals supply the burners along each wall, and the openings above are arranged so that the whole of each face of the flues along the oven wall can be inspected. The number of gas jets is increased, as also is the heating surface. A new method of bonding which is an absolute lock, and said to tie each side of the wall together in each layer of blocks independently of the blocks in the layers above and below, is also a part of the design. With these improvements the designers claim to have solved a problem which has been exercising the minds of coke-oven engineers for years. I am not in possession of full details, but it would appear from the above that a definite step forward has been made.

The Semet-Solvay waste heat oven is of horizontal flue type, and is illustrated by fig. 8. It is of exceedingly strong design and construction,

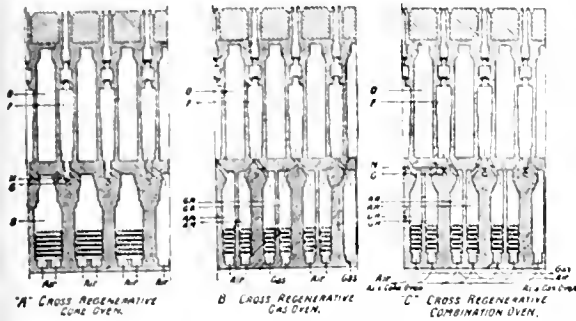


Fig. 6.

is divided by a vertical partition wall under the oven chamber, as shown by the sections on fig. 6. The regenerators under one oven serve for the lean gas, and those under the next oven for air. This destroys to a certain extent the independence of each oven, but this is of little importance in practice, as it is usually unnecessary to repair the regenerators or sole flues, and it is always necessary to lay three ovens off to repair the walls of one oven of this kind owing to the heat. In any case, the advantages of a clear separation of gas and air regenerators by the thick wall supporting the oven pillar, thus obviating all danger of leakage and short-circuiting, far outweigh considera-

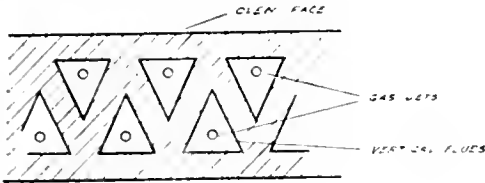
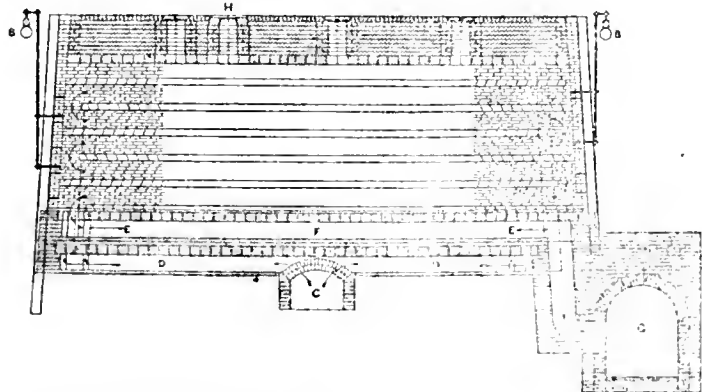
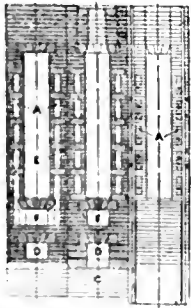


Fig. 7.

tions of this kind. It is an arrangement common to most ovens of this type.

The cross-sections on fig. 6 show the differences in the three ovens, and also indicate the position of the rich gas canal and burners in the compound oven. It will be realised also that the alternating

Semet-Solvay oven. Waste heat type.
Fig. 8.

having firebrick walls of considerable thickness between the ovens and independent of the oven linings. These thick walls carry the weight of the oven tops, mountings, and charging cars, etc. Thus the linings are not subjected to excessive weight, and as each oven has its own set of heating flues each side, absolutely separate from those of adjacent ovens, repairs can be carried out without in any way interfering with them. Further, owing to the thickness of the division walls it is only necessary to lay one oven off at a time. In this oven also the mass of brickwork carries a huge reserve of heat, which means quick recovery after recharging.

There are five horizontal heating flues on each side of every oven, to four of which gas is supplied by tuyères connected to mains running along each side of the battery. Each of these gas inlets is made so that air can be admitted if necessary, but the oven is designed and intended to work on the principle of supplying a large amount of the total air required in the top flue. The object is to keep the top portion of the oven as cool as may be consistent with maximum yield of by-products. The top flue is also kept well below the oven crown for the same reason. Sight-holes are provided at each end of every horizontal flue so that the heating may be accurately regulated and the condition of the oven walls kept under constant observation.

The oven is partially recuperative as the air for combustion passes under the centre of the battery in the flue, C, and branches out under each sole flue in canals, D, to vertical ducts in the dividing walls supplying air ports close to each gas jet. These ports are so proportioned and regulated that the amount of air supplied to the top flue is greatly in excess of its requirements. By the time this excess air reaches the second and subsequent flues it has attained a high temperature and increased considerably in volume, the result being excellent distribution of heat, thorough mixing with the gas, and economic combustion with attainment of the highest temperature where it is required, at the lower portions of the oven walls. The waste gases combine in the sole flue, F, and then pass out to the waste gas flue under the ram track. Dampers are provided for regulation of draught.

The horizontal-flue oven of all designs has an advantage over vertical-flue ovens in the very important matter of thorough mixing, because of the length of travel of the combustible mixture and the change of direction at the end of each flue. At the same time, the area of the flues for a given heating surface is larger than in vertical flue ovens, which discounts to some extent this advantage. There is obviously a limit to which the principle of admitting a large excess of air at the top flue can be carried consistent with a reasonable percentage in the final waste gases and avoidance of undue losses, owing to partial saturation of the mixture with inert gases in the later stages. This compels the admission of primary air at the gas jets and a small amount of secondary air from the vertical ducts, or either of these. Nevertheless, I think that with the facilities for mixing the arrangement is an excellent one and capable of giving good results.

The Semet-Solvay regenerative oven is designed on similar lines with regard to the structure and arrangement of the flues and gas supply. The air is all preheated in the regenerators as usual, and during one period the gases travel downwards through the wall flues in the same way as in the waste heat oven. After reversal they travel upwards to the top flue, and thence by vertical down-take to the opposite regenerator. The regenerators are arranged athwart the battery in two sections for each oven, with two longitudinal flues

between them, which are used for air and waste gases alternately. In this oven the gas supply does not need reversal.

The C.G.O. (Coke and Gas Ovens, Ltd.) regenerative oven is of the vertical flue type. The general design commends itself as an admirable combination of the best established practice and sound theory in the aim to produce an oven which will meet all the demands of the present day. The essential principles of solidity and uniformity of stress under working conditions, already referred to, have evidently been kept in mind by the designers, while the important questions of heating surface, rapid coking, accessibility, and uniformity of heat distribution have received due attention.

The oven structure is supported on five massive pier walls and four arches forming tunnels, all of which are utilised as regenerator space. Thus maximum economic heating surface is attained without undue depth of foundations or any sacrifice of strength and stability. Uniform expansion of the whole structure is made possible, so that distortion and cracking are a minimum. Notwithstanding this, the regenerators are divided by cross walls into separate chambers, so that the advantages of independent regenerators are obtained. These separate chambers are two oven pitches in length, and those in the outer and inner tunnels serve alternate ovens. The hot air from the regenerators enters the sole flue at the centre of its length, so that distribution to the inclined ports leading to the base of each vertical flue is even.

The burning gases ascend the flues in one half of the oven wall and descend those in the other half. They collect in the sole flue under this half of the oven, and are led into the regenerator and waste gas flue under the regulating gallery and on to the stack. The outer regenerator chamber at one side connects with the inner chamber at the other side of the battery. Regulation can be made by the dampers between regenerators and waste gas flues, and also by the small damper blocks at the top of the vertical flues.

The C.G.O. compound oven follows the same principles of design and arrangement. The regenerators are subdivided so as to provide for both air and lean gas, as in the case of ovens previously described. An important difference, however, is that the gas and air are kept in separate tunnels, with the main walls of the structure between, so that leakage is impossible. This arrangement also makes it perfectly safe to keep the regenerators for both gas and air under the oven which they serve, whilst the sole flues are arranged in pairs, and the gas and air never approach each other until they issue from the ports for combustion in the oven flues. Separate cast-iron valve boxes are provided for both gas and air at each oven, and reversal is effected by wire ropes in the usual way. Independent regulation of air and waste gas is done by butterfly dampers, the setting of which is not affected by reversing. There are also sliding fireclay dampers, and dampers at the top of each vertical flue for distribution. The oven can be worked as an ordinary regenerative coke-oven, in which case all the regenerators turn on to air, and rich gas is supplied through the usual tuyères and canals which are always there in readiness. A small amount of rich gas can also be used in conjunction with lean gas if required.

The outstanding feature of the Otto waste heat oven is the method of distributing the gas with absolute certainty to the base of each vertical flue without any reliance whatever on chimney draught, and this principle is also applied in the regenerative oven. The whole of the oven structure is supported on continuous brick arches forming a platform like a wide viaduct, so that although the piers run at right-angles to the longitudinal centre line

of the battery there is little danger of damage through expansion. The spacing of these arches with relation to the oven pitch is such as to cause a very evenly distributed load on the arch rings. Openings in the pier walls and the access gallery under the coke bench make the whole basement into one huge cellar. The gas for heating the ovens is brought into this cellar by a main in the access gallery, and branch pipes from it feed the vertical gas tuyères under each pair of vertical flues. Each tuyère is provided with a regulating valve for the gas and a rotating shutter for primary air supply on the Bunsen burner principle.

The secondary air is admitted through a star-shaped brick in a fireday channel, which also serves to centralise the tuyère pipe. By this means intimate admixture of gas and air is effected, and perfect combustion under absolute control is the result. All the secondary air is drawn in round the gas nozzle by the influence of the chimney, and the only regulation is of gas and primary air and main draught on each oven. The hot gases ascend the vertical flues, traverse the top horizontal flue, and descend the two vertical flues at the coke ram side to the waste gas flue under the track. The sole flue is not used except when the oven is starting up or working as a non-by-product oven.

The Otto regenerative oven is built on arches forming a cellar in a similar manner to the former. The arrangements for distribution of the gas to the vertical flues are the same, except that there is of course no primary air admission. The regenerators are built outside the battery under the coke bench and ram track, and are of narrow and deep design divided into two parts by a vertical partition. Each half serves half the combustion flues in the oven walls with the object of securing a more even distribution of air. The waste gas flues run along one side of each regenerator, with which they communicate at intervals through special ducts provided with dampers.

Air is admitted to the regenerators from the cellar through separate openings, also provided with regulating dampers in addition to the reversing flaps. The waste gas is reversed by dampers at the end of the battery, and the same which reverses the air flaps and the three-way gas cocks between the main and the distributing pipes. There is no separate regulation of air distribution on this oven, but the arrangement of the inlet and canals is conducive to a very even draught, and the absolute control of the gas in the vertical flues obviously makes it possible to increase or decrease the temperature at any point within very wide limits owing to the amount of excess air necessarily admitted in all regenerative ovens operating on rich gas.

There is a constriction at the top of each vertical flue in the style which has now almost become standard, but no dampers or openings in the oven tops. These were found by actual trial to be unnecessary in this oven because of the perfect control of the gas distribution.

The gas burns upwards in one half of the wall, and descending the flues of the other half is collected in the double sole flue and led into the corresponding halves of the regenerator, dampers for general regulation of the oven being provided in the inclined ducts leading thereto. There is ample space in the cellar under the battery for regulation, inspection, etc. The gas tuyères can be instantaneously removed for cleaning without undoing any bolts or set screws.

The Otto combination oven for use either as a coke oven fired by part of its own gas or a gas oven fired by lean gas is of the vertical flue type with independent regenerators athwart the battery. I am not able to give details, but I believe a battery is under construction at the present time.

The concrete foundations for a battery of ovens should be reinforced with steel bars, old rails, or the like, even if the nature and strength of the ground does not demand it, on account of expansion. Even with the best provision for expansion of the battery there is the possibility of cracking the concrete, so that it becomes a series of disconnected blocks which may sink unevenly either on account of differences in the nature of the ground or the distribution of the load on same. Such reinforcement is frequently placed diagonally in two layers and considerably higher in the concrete than design for purely construction considerations would dictate. It is desirable to avoid wet ground, and any material of a combustible nature must be removed, whatever the cost or efforts in the direction of heat insulation.

General principles of design.

The first is that there should really be no such thing as a standard oven. The nature and properties of the coal must be taken into consideration first of all and the oven chamber and heating flues proportioned accordingly. For instance, the width of the oven is a most important matter, and may vary from 16 in. to 24 in. according to the class of coal.

If an oven is made too wide in an attempt to obtain "long" coke it may be found, as in certain Staffordshire seams for instance, that there is a most marked difference in the density at the outside and centre which will result in fracture with the least handling.

A very high oven is unsuitable for coals of indifferent coking properties, which have to be compressed, on account of the difficulty of making and handling the cokes. A long oven without taper is obviously unsuitable for coals which yield coke tending to be soft or friable, or which expand on coking.

The heating surface, as well as the number and arrangement of gas burners in the oven walls, should be proportioned to the requirements of the coal and the quantity, composition, and calorific value of the gas evolved. The area of the flues and ducts for gas, air, and waste gas should also be decided by these considerations. The position and capacity of the gas ascension pipe should also be settled from considerations of quantity to be dealt with and the maintenance of regular and correct relative pressure in the ovens.

There are many other considerations of this kind, but these will be sufficient to illustrate the point.

The coke oven is what engineers call a block-work structure. True, it is of a special type and subject to all kinds of stresses, both internal and external, which have to be provided for without recourse to internal reinforcement and are dependent on interlocking, tying, and bonding of the blocks for stability, when the stresses cannot be balanced against each other or neutralised by armature. This being so, it is a vital necessity to minimise the forces and arrange for their even distribution as far as possible and to relieve those parts of the structure subject to the greatest heat, and, therefore, in danger of softening and losing their strength, of as much external load as possible.

These parts are also where the maximum movement will take place due to expansion and distortion, and damage will be in proportion to the importance allotted to them as members of the structure. The best possible material is the cheapest, and adequate strength must not be sacrificed to space or rapid transmission of heat.

The structure should be looked upon as a series of retaining walls and arches, and the load, from whatever cause arising, should be transmitted to them in the proper manner for such. The design of any parts of the structure as beams should be discountenanced, and the use of large bridge

blocks avoided. It is urged that a minimising of the number of joints by such construction is a great advantage as tending to reduce the risk of leakage. When the difficulty of making, drying, and burning the blocks and keeping them true to size and shape, to say nothing of the contraction question, is taken into consideration, however, and also the result of the cracks which are almost certain to occur, it is seen that there is little, if anything, in the contention. Another and important point in this connexion is that all blocks do not turn out the same, and the results following the failure of a large block are much worse than in the case of a small one.

I do not advocate the use of exceedingly small blocks by any means. The blocks should be of reasonable and something approaching uniform size, which will tend to cheapen and speed up manufacture as well as be of great advantage in the finished structure.

In considering the general question of the design of a battery of ovens by far the most important and at the same time most difficult problem is that presented by expansion. This is not only on account of the ovens proper, but the surrounding structures and plant. The battery must stand alone, and although it may be used to support other work outside itself, allowance must be made for movement in three directions and it can therefore only support such things as can lift with it between times of adjustment without damage.

Attempts have been, and still are, made to gain support for the battery from the coke ram terrace or the coke bench. They will always fail, because the expansion is irresistible. Even if the above structures could be made of adequate strength the result would only be to damage the oven itself. Many ingenious devices and arrangements are resorted to with the object of providing for expansion within the oven structure without any alteration to its outside dimensions and shape. Their scope is limited, however, on account of the difficulty of accurately determining what that expansion will be. Therefore some external movement must be provided for if close brickwork and stability are to be attained. The longitudinal expansion of the battery is the most difficult to deal with on account of its direction with relation to the walls of the oven and the flues and canals below the sole. It also gains in importance with the number of ovens. The oven tops and the mass of brickwork at the oven sole have to carry the pillars or oven walls with them as they expand, and must do this without rupturing them if possible.

A good deal depends on the care with which the battery is heated up, and extra time and fuel given to this will be well repaid. It is not sufficient however, and special constructional devices have to be provided at intervals to "take up" the movement. All builders have their own well-tried arrangements, and it would be of little general interest to attempt to catalogue them. They usually take the form of a checked or halved joint between adjacent blocks at some well supported point. In building this is filled with material which will either partly burn away such as mixtures with pitch as a binder, or with material such as asbestos, silicate cotton, or dry sand, which will compress under high pressure whilst of sufficient strength normally to support the stresses put upon it during building operations. Careful calculation based on actual trial of the refractory material to be used determines the position, number, and "setting" of these expansion spaces, and they are carefully watched during building and heating up.

All parts of the oven structure are not subject to the same temperature, and changes of state of the refractory components are different, so that the amount of movement differs.

Thus from a variety of causes there is relative

movement between the bricks and blocks at different zones, and layers have to be designed to permit this to occur without damage or leakage ensuing. The arrangement of small air and gas canals so that they are not affected by such movement calls for careful study and experience, as even a small miscalculation might upset the eventual distribution and render it impossible to get good results in the heating and output of the oven. Worse, it may cause short-circuiting and fusion of the bricks.

Perfectly dry joints are not usually permissible unless the bricks are rubbed to a perfect fit as in gas retort and glass furnace practice. Usually a distinct cleavage can be obtained at a joint without this. One method is the insertion of paper, another powdered carbon. In the lower portions of the oven, on foundations, and at the end buttresses dry sand is used. It is necessary on occasion to provide for eventual expansion in the initial setting out of the ovens.

The bonding of the blocks in the oven walls equals in importance almost any other problem in coke oven construction. The object of all designs is to get the maximum "cover" by the use of a minimum number of different shapes, and to tie the actual linings together through the wall, and so make it as nearly equal to a solid wall in strength as possible. This has to be done without increasing the thickness between the flues and the oven face, which would tend to prolong the coking time.

It used to be considered essential to arrange the bond so that repairs or relining could be effected with a minimum amount of general disturbance of the wall as a whole. Experience has taught, however, that such considerations are insignificant compared to the advantages of strong construction and tightness. Therefore the use of ridges and recesses in both horizontal and vertical joints has become almost universal. Several different arrangements are shown in fig. 9.

The amount of clay used should be the absolute minimum practically possible. In the first place the clay, however treated, is different in actual composition to the bricks because of the chemical changes during manufacture of the latter. Changes in the fireclay in the joints are also bound to occur when the oven is heated up, and as the most important do not occur below about 800° C. a preliminary heating up followed by re-pointing with fire cement is commercially impracticable.

Secondly, as the clay has to be used in a plastic state, there will be water in proportion to its quantity to be driven off and shrinkage accordingly. This is partly distinct from, and in addition to, chemical change.

Thirdly, there is aeration of the clay, the bad effect of which is also in proportion to the amount.

Unless the bricklayers are carefully trained and constantly watched they will leave air bubbles or voids in the joints instead of making them solid, thus defeating the object of the use of all mortars, which is exclusion of air in order to attain more or less perfect contact and increase friction and stability. Furthermore, this air must expand on heating, and will find its way out where resistance is least, viz., at the joints. Subsequent leakage of gases will, of course, occur through the channels or pores so formed.

Armature.

The buckstaves and tierods for binding the brickwork have an importance greatly increased by the trend of modern design to deep structures due to size of ovens and independent regenerators. In the past they were nearly always too weak and of little use in maintaining the structure. In many instances the most glaringly unsuitable steel sections were used or old railway metals of inadequate strength. Buckstaves should, if possible, be connected at each extremity by tiebolts, so that the

binding of the oven brickwork is independent of anything outside the structure. This is seldom feasible, however, so the bottoms are fixed into the lower portion of the structure where movement due to expansion is a minimum. It is desirable, especially with the newer designs of ovens, to carry them right down to the concrete foundation; the tops being connected by tierods across the battery over the oven walls.

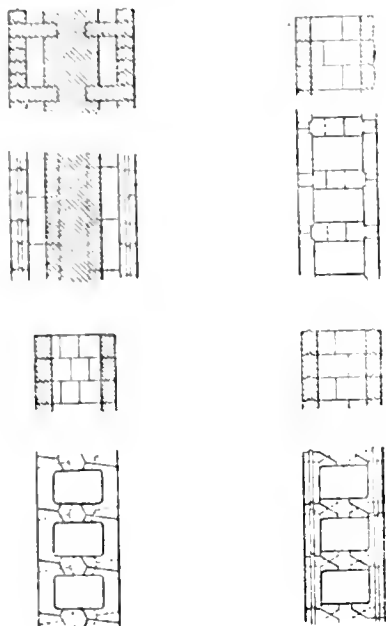


Fig. 9.

Their function is not the prevention of movement due to expansion—this is impossible—but the maintenance of the form of the battery, prevention of bulging, strengthening of the brickwork at wall ends by holding the blocks in firm contact with those further along the wall, closing of cracks due to periodic contraction, and compelling the taking up of expansion in the places designed for it, which the weight and friction of the outer ends of the wall might not be sufficient to do.

They are designed as beams, and much discussion has attended the question of determining the actual load which is likely to act on them. One method suggested was to assume a crack through the whole height of the structure at the angle of repose of loose broken firebrick on itself, starting from the buckstave toe; and calculate the conjugate thrust. Another, to find what diameter of tierrod would break and take the ultimate strength of it as half the distributed load; a third method probably more nearly accurate was to calculate the force necessary just to move a mass of bricks one quarter of the oven in length and of height above various horizontal planes, all assumed to be loose joints, and use these as loads on the beam. A German method was to work from the load which an equal depth of water would give and assume the centre of pressure reversed. None of these methods are accurate of course, but they are genuine attempts and as good as we have.

With silica oven construction the problem gains in importance on account of the amount of expansion. Adjustment of the tierods during heating up must be made, and it is best done by hardwood temporary washers. They should never be slackened right out. I know of two fairly recent cases of furnace collapse through a mistake of this kind. With silica construction we shall probably find it

necessary to provide for adjustment at the bottom as in glass tank practice owing to permanent expansion.

Exceedingly high refractory qualities are not the first essential for coke oven blocks as they are in the steel and glass industries. Contraction, whether due to imperfect manufacture or the nature of the fireclay, *i.e.*, high in alumina, is the bugbear of coke oven construction because the opening of joints cannot be satisfactorily dealt with. The leakages from this cause mean tremendous losses of rich gas when an oven is freshly charged and pressure cannot be avoided, and, later, destruction of coke and by-products, excess nitrogen and carbon dioxide in the gas, as well as great irregularity of heating and danger of fusion at all times.

In the old days managers became expert in blocking leakages with deposits of carbon by manipulation of draught and air supply, but frequently deposits occurred where they were not required, and this irritating juggling had to be constantly practised, as further openings were always developing. Complete repointing with carborundum, sodium silicate, and fireclay, or patent cement of a similar kind, was found to be of little avail. Some natural fireclays were found to be suitable and hold a high place still. Others were made suitable by admixture of silica and grog.

Many attempts were and are still being made to evolve mixtures which when suitably manufactured into blocks and burned will be without serious permanent expansion or contraction, and yet fulfil the other conditions required as to temperature, strength, and resistance to corrosion. Some of these have been very successful.

The silica oven was tried out in America and has come to stay. It is capable of being worked at high temperatures continuously, and, owing to its relatively high conductivity, rapid coking and uniform oven temperatures are easy of attainment. There is a very great expansion, but unlike contraction this can be dealt with. The expansion is of two kinds, *viz.*, that due to change of temperature only, which is temporary, and that due to change of state of the silica and its compounds at high temperatures, which is permanent.

There appears to be no doubt that the size of particles is of great importance, as fairly large pieces, or "peas," of free silica do not combine to the same extent with calcium in lime-bond silica bricks or the impurities in clay-bond bricks. The manufacturer must therefore discover the best grading from the standpoints of strength, homogeneity, and freedom from voids on the one hand and excessive fineness with danger of chemical change on the other. The question of porosity is also of importance because it is found that porous blocks do not spall so readily as the faced, and stand rapid changes of temperature better. It is also probable that a rough surface radiates heat better than a smooth one.

In spite of the difficulty with high-alumina blocks, a large battery was built with them and successfully operated in America, with, it is stated, excellent results. Such blocks would have great advantages in certain cases and may some day be adopted here. They are used of necessity in the glass industry, and special construction providing for the contraction difficulty is not beyond the bounds of possibility in coke-oven practice.

Working of the ovens.

For the efficient mixing of gas and air nothing in the direction of impinging or whirling jets arranged in the flues can equal outside admixture of gas and primary air, and the surrounding of the gas current with secondary air, particularly when using rich gas. If this is not done independent currents are certain, with the result that complete

combustion only occurs at changes of direction, which is often where great heat is not desired, such as the tops of the ovens and the sole flues. Sight plugs in accessible positions must be provided so that combustion can be watched while regulation is made, and the greatest possible amount of the flues must be open to inspection of the state of the brickwork without difficulty or inconvenience.

The three great anxieties of a coking plant manager are repairs, shortage of steam, and coal supply. The first two have already received treatment, and possibly the last forms a more fitting subject for a separate paper. Large storage bunkers, the contents of which are all accessible, are a vital necessity, however. They should at least be capable of holding three days' supply to tide over the week-ends easily without traffic troubles through blocked sidings.

The question of deterioration of the coal has, of course, to be considered, but in the majority of cases this is of rather less importance than continuous working. Whether by absorption of oxygen from the air or simple loss of methane, finely divided coal certainly does lose its coking properties as well as some of its calorific value. This is more marked in some coals than others and always worse the finer the coal. It may be minimised in several ways, such as mixing a proportion of larger coal with the slack, crushing immediately before use or at least at the top of the storage bunker, storage in comparatively air-tight ferro-concrete or steel bunkers after draining by mechanical means, such as the Baum conveyor, instead of in drainage bunkers or wagons, and the use of hoppers bunkers which are self-emptying.

The position of the storage bunkers depends largely on the system of charging the ovens and whether conveyors are to be used on account of mining etc.

It is necessary to compress some coals of different coking properties in order to make good dense coke. Even coals which yield a good coke without compressing are often improved by it, and, compared with hand charging from corves or dandies, the compressed cake is an excellent labour and time saving device, especially for large ovens. The upkeep of the machines, power consumption, and time required to make cakes, however, put it out of the running with the modern electrically driven top charging machines. These are capable, in conjunction with an efficient coke ram and leveller of dealing with six ovens of large size per hour, whereas only 3 or 4 at most can be managed when cakes have to be made.

The coal when charged into the oven should not contain more than about 10% of moisture. Some trials made by the author eleven years ago, on North Staffordshire coal, showed a difference of over 1% in the amount of breeze produced, due to difference of moisture content.

The position, size, and length of the gas ascension pipe needs attention; it is a mistake to put it at one end of the oven, especially when the latter is filled as high as possible. Convenience in the arrangement of the method of charging should not be allowed to dictate this. Far better have two separate cars, or, better still, two ascension pipes and collecting mains, one each side of the battery. There would be no difficulty in this on the score of cost when two qualities of gas were required, and it would be worth the expense in other cases owing to increased yield of by-products and output of coke through regularity of working. The quantity of gas given off in the first few hours is much greater than towards the end of the coking period, and, in addition, there is a very large volume of steam. Thus high pressures are the rule immediately after charging, which give a great deal of trouble in luting doors and charging holes and even so result in losses by leakage into the atmosphere and the

combustion flues. Similarly, in the later stages air leakages occur through excessive suction and the unavoidable neglect of the luting.

I do not think the fact is generally realised that the early part of the coking period is the most efficient looked at from the standpoint of the application of heat, because of the greater temperature difference. Everything possible should therefore be done to make the most of this. Even a small difference of pressure makes quite an appreciable difference in the distillation of substances like coal. This leads to the question of oven isolating valves which can be regulated, in conjunction with larger ascension pipes. These have been adopted by at least one of our leading firms.

The mushroom type of valve is scarcely suitable owing to the large increase in area for a small movement and consequent danger of blockage. The butterfly type is better in spite of some disadvantages. The flat bench on to which the whole mass of coke was pushed, pulled over, and quenched by hand, is now obsolete.

As the size of coke-ovens, number per battery, and speed of coking all increase mechanical means of quenching and loading coke become more necessary. The manual labour problem also compels their use. I shall therefore briefly describe one or two:—

1. The inclined bench covered with smooth cast-iron plates and set at an angle down which the coke will slide on to a tray conveyor at the bottom, is a good arrangement provided the conveyor is of sturdy construction and has working parts capable of resisting the rough wear of fine coke dust and water. In case of breakdown there is room to store the coke whilst repairs are carried out. The conveyor is usually inclined at one end in order to gain the elevation necessary to deliver over bar screens for separating breeze, into ordinary trucks or blast-furnace skips. The bottom portion of the bench is arranged as a tilting flap, which retains the coke on the bench in one position and acts as a shoot on to the conveyor in the other. These flaps are in separate lengths and operated from the gangway outside the conveyor.

2. The Coppée coke screening and loading machine is arranged to travel by electric power the whole length of the inclined coke bench. There are gates at the bottom of the bench operated from a gangway, which deliver the coke and breeze into a continuous bucket elevator. This raises it to a sufficient height to deliver over a bar screen into trucks or blast-furnace skips on a track outside the machine. The breeze is collected in a box below the screen and discharged at intervals into separate wagons. The screens and shoots can be arranged to deliver both large coke and breeze into wagons on the same siding. Where space demands, or it is desired for any reason, a vertical self-tipping hoist replaces the bucket elevator. In hilly country the machine can be arranged without either, the whole movement of the coke being by gravity. The makers claim a great saving in cost of coke loading, the machine being operated by one man, facilities for selection of any batch of coke on the bench on account of quality or the position of wagons, rapidity of loading (the rate being 25 tons per hour), simplicity of construction and operation. The machine is also away from the steam from the quenching operation.

3. Another type of coke car consists of a shallow steel car with inclined bottom, and is in effect a short portable coke bench. There are gates at the bottom of the slope to retain the coke, which is thinly spread out over the bottom of the car by slow translation of it along the battery as the coke is discharged from the ovens through a Darby type quencher. The object of this is to allow for cooling and steaming off, and to this end the area of the car bottom is made as great as pos-

sible. There is usually an incline at the end of the track up which the car is hauled by wire rope to deliver over bar screens to coke wagons below.

4. The Goodall quenching and loading machine consists of a rotating table on to which the coke is pushed through a quencher of ordinary type. The movement of the table whilst the coke is passing on to it parts the pieces gently and spreads the coke out in a thin layer to steam off and cool. There is a steel cylindrical casing round the table about 6 ft. high, so that no direct air current can play on the coke at this stage. There is a wide shoot on the side of the machine remote from the ovens, with jiggling screen and breeze boxes below. The portion of cylindrical wall of the machine opposite this shoot is loose and pivoted at one side. When coke is to be loaded into wagons the table of the machine is rotated in the reverse direction, and the free end of this loose piece at the same time slowly travels towards the centre of the table, thus forming a plough and guiding the coke down the shoot. The whole machine travels by electric power on rails along the battery and can receive coke from any oven and deliver to any wagon, thus permitting selection of specially good quality and avoiding necessity for shunting of trucks. It effects great saving in the cost of coke handling.

5. The size of ovens and number per battery, also the rapidity of coking under American conditions, has compelled the adoption of high-speed coke handling, and it has been found that no system in which coke was allowed to remain in close proximity to the ovens was possible. With ovens coming off every few minutes, it will be realised that freedom from any incumbrance, such as coke cooling off and steaming, is vital. The best arrangement, and one which now finds most adherents, is central quenching; i.e., the coke is not quenched as it leaves the ovens, but taken to a central quenching station, at high speed, in a special coke car, to be quenched and afterwards either spread on a coke bench apart from the battery or delivered direct to screening and loading plant.

In a typical arrangement of this kind, constructed by Messrs. Simon-Carves, Ltd., the coke is discharged from the ovens by a very powerful high-speed ram in about half a minute, through a coke guide, which is like the ordinary quencher without water, into a short and deep coke car. This car is rushed at high speed along a horizontal track by electric locomotive to the quenching tower. This is a high, chimney-like structure, usually of ferro-concrete and provided with large water tanks round the central chimney, at the top. A measured quantity of water is instantaneously doused on to the coke in the car, which is then conveyed to the wharf and on to the screening plant by tray conveyor. The gates at the front of the sloping bottom of the car are pneumatically operated by the driver of the locomotive, so that no time is lost, and the car returns to the ovens for the next batch. Such a car is capable of serving a battery of 150 ovens.

A novel form of screen, called the "grizzly," is being put forward in connexion with this plant, consisting of a series of shafts set slightly below each other and parallel to the track on which the coke car runs, or the feeding conveyor. On these shafts discs are threaded at intervals of an inch or so and meshing alternately, so that the appearance in plan is a huge grid. These shafts rotate at different speeds, that at the top being the slowest. The result is that the coke is drawn from one on to the other in the same manner as fabrics in many devices used in the textile industries. The breeze falls through into hoppers, and the large coke is gently lowered without risk of damage into wagons or furnace skips at the bottom end of the screen.

In conclusion, I wish to express my thanks to the various firms of coke-oven engineers for the loan of

slides and drawings and courtesy in giving me information asked for.

THE MORE ECONOMICAL UTILISATION OF COKE-OVEN AND BLAST FURNACE GASES FOR HEATING AND POWER.

BY G. W. HEWSON, A.I.MECH.E., AND S. H. FOWLES, A.M.I.MECH.E., A.M.I.E.E.

We shall endeavour to show in this paper that in the manufacture of metallurgical coke, and in the use of such coke after leaving the oven, large quantities of gas are produced which can be more efficiently utilised than is the practice at present.

Professor Bone states that in 1913 the output of pig-iron in the United Kingdom amounted to 10·48 million tons, from which 7·66 million tons of steel was made. The average coke consumption amounted to about 25 cwt. of coke per ton of pig-iron produced. The corresponding amount of coal would be about 37·5 cwt.; thus the total coal used in producing 10·48 million tons of pig-iron would amount to 19·65, or, say, approximately 20 million tons. In 1916 Professor H. E. Armstrong stated that "fully two thirds of this coke is now produced in recovery ovens of various types." This leaves one third still being produced in the wasteful beehive ovens—a figure confirmed by the Nitrogen Products Committee—and it is safe to assume that all the recovery ovens in use are not working upon the most economical lines.

Taking these figures as a basis, the potential possibilities, after supplying the needs of the coke-ovens and the blast-furnace, may be stated thus: *From the coke-ovens:* $20 \times 10^6 \times 5000 = 100,000$ million cb. ft. gas at, say, 500 B.Th.U. per cb. ft. *From the blast furnaces:* $10 \cdot 48 \times 10^6 \times 75,000 = 786,000$ million cb. ft. gas at, say, 100 B.Th.U. per cb. ft. These figures assume 5000 cb. ft. of surplus gas from the ovens per ton of coal carbonised, and 50% of 150,000 cb. ft. of surplus gas from the blast-furnace per ton of pig-iron produced.

How best to utilise the enormous potentialities of these gases requires careful consideration. Engineers in this country have had more extended experience in steam plants than in gas engine plants, whilst the reverse is the case on the Continent. There the large gas engine using coke-oven gas is more popular, and the special difficulties in dealing with a rich gas with a high hydrogen content have been overcome.

Use of coke-oven gas.

The following have been taken from Brame's "Fuel" as average figures obtained on several classes of work, and we consider they represent good average working:—*Steam:* Steam raised per lb. of coal carbonised, 1·25 lb. Steam required per h.p.: reciprocating engines 15 lb.; turbines 11 lb. H.P. per ton of coal = 187 and 255 respectively. *Coke-oven gas:* Surplus gas per ton of coal carbonised 5000 cb. ft. at 500 B.Th.U. Heat units required per h.p. 9500 (27% efficiency). H.p. per ton of coal = 266.

In tests made at Messrs. Cockerills, 1 kw. at the terminals consumed 35·3 cb. ft. of coke-oven gas at 450 B.Th.U., approximating 9000 B.Th.U. per h.p. on the gas engine with 95% efficiency of the generator.

Dr. H. G. Colman gives the consumption with 458 B.Th.U. gas on a Nürnberg engine of 1200 h.p. at 100 r.p.m. as 21·3 cb. ft. per h.p., equalling 9750 B.Th.U. per h.p.

These results were actually obtained on long runs quite apart from test figures, and give quite a good lead to the internal combustion motor over other prime movers.

Professor Bone in "Coal and its Scientific Uses" points out the possibility of saving 0.9 ton of coal per ton of iron produced by having the coke-ovens in close conjunction with the iron and steel works, instead of at the colliery, and by utilising the surplus gases from the ovens mixed with blast-furnace gas for heating the steel furnaces and soaking pits, and for generating power.

We agree that blast-furnace gas alone should be used for gas engines in the power house for reasons which we shall state later, and the surplus coke-oven gas will be better employed in heating steel furnaces and soaking pits, either alone or diluted with blast-furnace gas.

Open-hearth steel furnaces have been successfully operated with coke-oven gas, thus effecting a considerable saving against producer gas. Other advantages are that coke-oven gas is anhydrous and low in sulphur, and it is possible to provide a reducing atmosphere over the bath, which is a decided advantage in the production of high-grade steel, excessive oxidation and decarbonisation being prevented. Coke-oven gas does not require preheating; it suffers decomposition, and its calorific value is reduced by 25–33% if preheated in the gas chambers of an open-hearth furnace. The manganese consumption is lower throughout than when working with producer gas. In addition, a saving is effected in refractory bricks, in wages, and in steam.

On the basis of 8000 cb. ft. of coke-oven gas required for making 1 ton of steel and 4300 cb. ft. for reheating furnaces per ton of steel, the quantity of coke-oven gas potentially available is sufficient for about 8 million tons of steel per annum.

The purpose for which surplus coke-oven gas is actually used is largely dependent upon the location of the ovens. The demand for such gas may be sufficiently great in some instances to make the gas a more important product than the coke. In such cases the plant would actually be a by-product gas plant.

The production and utilisation of blast-furnace gas.

The relative proportion of blast-furnace gas made and utilised profitably in Germany and this country in 1911 was—

	Germany.		England.
	B.H.P.		B.H.P.
Produced ..	1,340,000	..	1,060,000
Utilised ..	448,000	..	23,000

In 1911 the total power production from blast-furnace gas in large gas engines was 1,033,509 b.h.p., of which Germany contributed 46.5%; America 32.5; France 5.4; Belgium 4.6; Austria Hungary 2.4; Great Britain 2.4; and other countries 6.2%.

The quantity of gas produced in a blast furnace using coke as fuel, is usually considered as approximately 150,000 cb. ft. per ton of iron produced; this may be materially increased if the blast furnace is considered as a gas producer, since the use of blast-furnace gas for the production of power, and its more economical use as fuel gas has materially altered the necessity for economy in the use of coke in order to keep down the cost of manufacture of pig iron. It has been demonstrated that by a more intensive working of blast furnaces, by increasing the coke in the charge, and, incidentally, the yield of pig iron, blast-furnace gas could be improved in calorific value. Trials have shown that by increasing the coke 18% an increased metal yield of 7% is obtained without affecting the quality of the iron, together with 15% larger volume of gas, with its calorific value raised 12%, the heating power of the total gas produced being increased 25%. Hitherto all the gas produced by a blast-furnace plant has been utilised on that plant alone, and in some cases has been augmented by the use of coal on boilers to raise steam for the blowing engines and auxiliary plant.

With modern and more efficient modes of utilisation at least 50% of the gas is released for purposes outside the blast-furnace requirements. This is equivalent to 25% of the heat value of the coke charged into the furnace. This economy results from the higher temperature attainable in the stoves by means of a smaller consumption of gas. Compared with stoves working on dirty gas, and requiring 50% of the gas produced by the furnace, modernised stoves working on clean gas have given higher temperatures to the blast over longer periods, with a consumption of 28–30% of the gas produced. In spite of the loss of the sensible heat of the gas, and allowing 12% for the blowing engines and 8% for leakage, losses, etc., there is available for power generation or other purposes 50% of the gas made. If, however, the blowing engines are not gas driven, then the quantity available will be reduced considerably, if not entirely wiped out.

Increase of our knowledge of blast-furnace gas should give an impetus to the construction of internal combustion engines and gas turbines. The most recent guarantee figures for large water-tube boilers give about 218 cb. ft. of blast-furnace gas at 100 B.Th.U. per cb. ft. as the lowest requirements per kilowatt generated with turbo-generators, which gives a thermal efficiency of 15.5% over boiler, turbine, and generator; while to-day, with gas engines at 10,000 B.Th.U. per b.h.p. hour it is possible to get an efficiency of 25.5%.

The number of engine-room attendants required is the same in each case, but more attendants and cleaners are wanted for boilers and turbines. The maintenance and repairs on gas engines are sometimes greatly in excess of that for turbines, but this does not apply to engines working on clean blast-furnace gas. Two engines have recently been examined after running without any trouble for 15,000 hours each, and they required no cleaning. Other engines have run on this clean gas for equally long periods without requiring the gas throttles cleaning.

Again, the greater efficiency of the gas engine more than compensates for the loss of sensible heat due to the necessity of cooling the gas for the engine, against retaining it for boiler use. It has been computed that on an average about 7% of the heating value of the gas is lost by cooling it. Taking 1 cb. ft. at 100 B.Th.U. cooled by engines, we get $(100 - 7) \times 25 / 100 = 23.25$ useful B.Th.U. in the engine; hot gas for boilers gives $100 \times 15 / 100 = 15$ useful B.Th.U. in the turbine. Comparing the thermal efficiency of gas engines with steam turbines, we see what a saving can be effected by the former, and this points to the importance of installing the most efficient machinery to utilise the gases evolved from the blast furnaces of the country.

Comparison of thermal efficiencies.

	Efficiency.		Ch. ft. blast-furnace gas reqd. to give 1 theor. h.p.
	%		
Large gas engines ..	25	..	114
Large turbines with boilers ..	15	..	190
Small " " " " ..	12.5	..	228

Taking two plants of similar capacity, producing and cleaning approximately the same amount of gas, the following comparisons have been compiled from actual experience of the use made of the gas:

	Use of gas.	Gas engine plant.	Boiler & turbine plant.
On stoves	45%	45%
On blowers	28%	55%
On electrical generators	27%	nil.

The main factors which render blast-furnace gas when properly cleaned such a good fuel for gas engines are:—(1) Freedom from tar and from dust. (2) Regularity in quality or calorific power. (3) Comparative constancy of volume. (4) Freedom from premature explosions, even under compression. (5) Lower temperature of the exhaust gases.

It has been demonstrated recently that if our existing plants were modernised, and brought up to a reasonable state of efficiency, the saving would approach the saving of power anticipated from the super-power station scheme.

We venture to suggest that, from a national standpoint, it would be better to harness all the potential power of the 300 blast furnaces at work in this country and, linking up with existing power companies, supply the country with a large proportion of its requirements at a more reasonable rate than is at present possible with stations using coal-fired boilers and turbines. It has been stated recently on high authority that if the steam turbine be increased from what is considered a reasonable size to-day, viz., 15,000 h.p., up to 60,000 or 70,000 h.p. the steam consumption can only be reduced $\frac{1}{3}$ lb. per kw. generated, which is a very small amount compared with the increased size of unit.

This large size of unit has another great disadvantage, that if with a station of two such units one set fails, serious inconvenience will be caused, even allowing for the possibility that such a plant may be linked with the greater scheme. Again, some of these larger stations may be built at a distance from the coalfield, and this will not mitigate our transport difficulties.

In order to compare the generation of electricity from blast-furnace gas with the capacity of existing or prospective power stations, the following table gives the units generated and consumed over a period of four weeks, using the surplus gas from three blast furnaces:—

Units generated	1,450,000
Consumed by steel works	540,000
.. .. shipyard	400,000
.. .. blast furnaces	230,000
.. .. gas cleaning plant	90,000
.. .. power station auxiliaries	30,000

Apart from cost of running, the reciprocating gas engine is more in favour as a blast engine than the turbo-blower from the blast furnace view-point.

The cleaning system.

Of equal importance with the type of engine to instal is the question of the most efficient type of cleaning plant. There are three principal systems, viz.:—The electrostatic, the wet washer, and the Halberg-Beth.

In the opinion of the authors the Halberg-Beth is pre-eminently the most efficient plant for cleaning blast-furnace gas. The plant they have been associated with has run successfully and smoothly for three years, and they are satisfied that with proper care and attention it will continue to do as well in the future.

The electrostatic system, while retaining the sensible heat, has not proved the possibility of cleaning sufficiently low for engine consumption. The best that can be hoped for is 0.3–0.6 gram of dust per cb. m. of gas. Taking into consideration the large quantities of gas dealt with over a period of months, even this comparatively small amount reaches a large quantity and will cause trouble in stoves or boilers, much more so in engines. The wet washers require a large amount of water to clean the gas for engines.

The Halberg-Beth plant gives gas cleaner than the surrounding atmosphere, often for long periods not exceeding 0.001 g. of dust per cb. m.

If all the power is cut off the cleaning system would pass dirty gas, the wet-washer would soon choke up, while the Halberg-Beth plant continues to supply the engines with clean gas. With the Halberg-Beth, 5% of any unit can be shut off for repairs, and the remainder continue working. This cannot be done by other systems.

In order to emphasise the superiority of gas engines over boilers and turbines, the following

table of comparative costs is given, starting with the same cost of preparation of the gas.

A. Cleaning 3 million cb. ft. of gas per hour by the Halberg-Beth plant.

First cost of plant, £62,000.	£
Interest on outlay at 7%	4340
Depreciation at 10%	6200
Power and light at 35 k.w. per mill. cb. ft. and 0.5d. per unit	1860
Plant labour costs	1064
Dust house costs	1560
Supervision	300
Repairs and stores at 6d. per mill. cb. ft.	638
Renewal of filter bags, one set per annum	900
Steam for reheating 0.2 lb. per 1000 cb. ft. at 2s. per 100 lb.	510
	£17,972
Less cost of boiler and flue cleaning	1594
Less cost of stove cleaning and lining	3360
Less revenue from sale of dust	4411
	9365
Net cost of gas cleaning per annum	8607
Cost per 1000 cb. ft., 0.081d.	

B. Cost of generating 1 B.Tr.U. of electricity from blast-furnace gas, using gas engines.

First cost of engines, £55,000.	£
Interest on outlay at 7%	3850
Depreciation at 10%	5500
	£9350
Load factor 40%	Pence.
Engine cost per unit	0.102
Value of gas used	0.075
Cleaning of gas	0.012
Labour	0.055
Maintenance	0.008
Oil and stores	0.054
	0.306

C. Cost of generating 1 B.Tr.U. from blast-furnace gas, using boilers and turbines.

First cost of boilers and turbines, say 75% cost of gas engines, £41,250.	£
Interest on outlay at 7%	2887
Depreciation at 10%	4125
	£7012
Engine cost per unit	Pence.
Value of gas used	0.078
Cleaning gas, 218 cb. ft. at 0.081d. per 1000	0.109
Labour	0.017
Maintenance	0.110
Oil and stores	0.016
	0.054
	0.384

Conclusions.

By building coke ovens near the blast furnaces and steel works upon the regenerative principle, about half the gas generated can be utilised for the latter. Further surplus gas might be released if the ovens were fired with gas produced from inferior fuel, unsuited for other purposes than conversion into producer gas.

With cleaned blast-furnace gas, better results are obtained on the furnaces themselves, as the result of higher and more regular heats being obtained upon the stoves. More regular blowing is possible with gas-driven engines than with the old steam engines.

By means of clean gas the requirements of the blast-furnace plant can be easily met, and surplus gas is available for other purposes, preferably for the generation of electricity by gas engines, and can thus supply all the power required by a modern iron and steel plant.

The production of electric steel, or the finishing of steel manufacture in an electric furnace, is gaining favour in America, and it appears to the authors that the policy they have advocated may lead to an impetus being given in this country to the finishing of steel manufacture in an electric furnace, which, as a piece of apparatus for refining steel, has no competitor.

Discussion.

Mr. J. E. WEYMAN said there was no doubt that the Halberg-Beth system of cleaning blast-furnace

gas had been successful, but a later American plant was said to be more effective, there being no cooling of the gases.

Mr. E. E. Wood asked the authors on what authority they stated that to make one ton of steel required only 8000 cb. ft. of coke-oven gas; his own figures were at least 20,000 or 30,000 cb. ft. The authors' figures showed a gas engine to be a more economical proposition than a steam turbine, but the figures which he had obtained showed just the contrary. The question of the relative efficiencies of the two was a very debatable one.

Dr. G. S. WEYMAN said that coal for coke ovens must be suited to the product, and the sulphur, ash, and phosphorus should be strictly limited. It was not necessary to be so strictly limited in coal gas manufacture in the ordinary way. The coal for gas works need not have any coking properties. If all the gas works were replaced by coke ovens a certain amount of coal would not be available for coke ovens and, he presumed, would become a drag on the market.

Mr. Hewson, replying to Mr. Wood, said that the figure of 8000 cb. ft. of coke-oven gas per ton of steel had been given in the *Journal of the Iron and Steel Institute* in 1914, and confirmed by Sir Robt. Hadfield.

Mr. FOWLES added that the American gas-cleaning plant referred to had not yet proved suitable in this country. By the adoption of the Halberg-Beth system they were saving about a penny per unit on the system; they had been buying at a 1½d., and were now producing at less than a farthing. Blast-furnace managers who had formerly preferred the steam turbine now preferred the gas engine, as it kept the burden of the furnace free. As regards gas engines versus steam turbines, he stated that the Newcastle Electric Supply Company, who for years had used the waste gas under boilers, now used gas engines with excellent results. The gas engine had suffered in the past because it was not properly handled and looked after, nor was the gas properly cleaned. In using blast-furnace gas they were free from tar troubles, the engines required little cleaning, and there was the additional advantage of the potash recovered.

COKE-OVEN GAS FOR TOWN SUPPLY.

BY HAROLD E. WRIGHT.

The object of this paper is to deal with the economic application of coke-oven gas to town supply.

If the true economy of our fuel resources is the main consideration, then the regenerative type metallurgical coke-oven stands first as a means for producing town gas with the least waste of the original fuel. In round numbers the relative efficiency of the various methods expressed in percentage heat units of original fuel remaining available in the final product is:—(1) Regenerative coke-oven 90%; (2) average gasworks plant 85%; (3) average gas plant afterwards converting all surplus coke to water-gas 55%; (4) water-gas plant 49%. The steaming of retorts and the making of water-gas are directly opposed to the conservation of fuel. This procedure is also only made financially practicable if town gas is sold at prices very much exceeding its true fuel value. The cost of distribution and general charges which can be reduced *pro rata* by increased sales are important items in a gasworks' cost account. The true reason that gas is not more largely used for heating and cooking by the public to replace coal and coke is because the latter means is less costly, and if the economic policy of gas manufacturers were directed to providing gas at a cost nearer its heating value, they would by thus encouraging consumption reduce one of their principal costs. This is a strong argument in favour of the use of the regenerative coke

oven for manufacturing gas for town supply, where it is possible to adopt it, because in a proper economic situation it can supply gas for town use at a price very little in excess of the cost of distribution and general charges. On the other hand, it is unfortunate that it may only be economically sound to adopt this method at a limited number of places for reasons stated later. In the regenerative coke-oven the surplus heat from the combustion of part of the gas to carbonise the coal is retained by reversible regenerators, and this being carried back to the oven walls by the air supplied for combustion releases all surplus heat as surplus gas. The actual gas realised varies with the quality of coal used. In the average case the surplus would be 55% or about 6000 cb. ft. per ton of coal. By proper construction of ovens and regenerators to enable producer gas to replace the gas used in heating the ovens, all the coke-oven gas could be obtained as surplus, but there is no saving of thermal units in this procedure—rather the reverse. Therefore, unless the gas so liberated can be sold at a higher price than the cost of the producer gas required to liberate it, this proposition is economically unsound, and from the point of fuel conservation it is totally unsound.

Where surplus coke-oven gas is available at adjacent coke-oven plants under separate control, the quality of which can be readily ascertained, its use for supplementing or replacing existing gas supply is a mere matter of ascertainment and bargaining. Public authorities so placed can most probably secure a supply of gas at a lower price than their existing plant can manufacture it. This, however, is only likely to be the case in isolated instances. The general case will require coke-oven plant to be installed and existing gas manufacturing plant replaced.

Of course this cannot be applied to every case, because many points need very careful consideration before the change is made. A few such points may be outlined. Successful coke-oven undertakings derive their revenue from the sale of metallurgical coke, and only in areas where such fuel can be sold readily can they succeed. There must be a ready demand, as continuous operation is necessary to the success of coke-ovens. In my opinion the coke-oven cannot be successfully operated to produce inferior coke.

The coal for coke-ovens must also be suited to the product required. The sale of metallurgical coke becomes much more difficult as the ash in it exceeds 10% and the sulphur 1%. On this account many good gas coals are not as suitable for coke-oven use. With this limitation on the coal supply the regenerative oven calls for twice the amount of coal to produce the same gas, and this in many cases will present great difficulty to-day.

Each separate case thus opens up difficult phases demanding the highest commercial and technical judgment to be applied to the study of the varying factors likely to operate for and against the proposal. The substitution is probably only worth considering where much extension or renewal of manufacturing plant is contemplated, and then only when the regular supply required is fairly large. Thus it is to be feared that all the chief influences will in general operate collectively against the introduction of the thermally more efficient coke-oven method of gas supply. There will be cases favourably placed, and the following matters should enter into their consideration.

The main economic difference between gas plant and coke-oven is that in the former handling of material and charging and drawing retorts etc. is done piecemeal and by a much more complicated method and at double the cost of the simple and large operations in coke-oven practice. The coke-oven derives its principal revenue from the sale of coke, while at a gasworks the sale of coke seems to be rather neglected, the principal revenue being made from the sale of gas.

Changes in costs and prices to-day are so kaleidoscopic that it appears preferable to illustrate this by figures drawn before the war than to attempt any present-day estimate. The following figures therefore represent the operation of a coke-oven and gas plant in the same area where cost of carriage on coal from the collieries is equal to both cases. In both cases the cost of the coal used is represented as 100, and the other figures are given comparatively, assuming 2 tons of coal in coke-ovens as required to yield the same gas as 1 ton in gas practice or, say, 12,000 cb. ft. per ton and per two tons, respectively:—

<i>Carbonising cost.</i>		
	Gas plant.	Coke-oven plant.
Cost of coal	100	200
Manufacturing cost	26	24
Interest, redemption, etc. ..	10	24
	136	248
Profit on sale of coke and breeze ..	45	200
Profit on sale of tar and ammonia ..	24	48
	69	218
Nett cost	67	Nil
<i>Purification, distribution, and general cost:—</i>		
Purification and general charges ..	65	
Interest, redemption, etc., on general plant ..	36	101
Plus loss on carbonisation as above ..	67	
	168	
Less:—		
Sale of gas	162	
Income from other sources ..	6	
	168	

It is obvious in this case that the coke-oven plant could pay all gasworks costs on distribution etc. plus the 10% for interest, redemption, etc. on gas plant laid out of use, and sell its gas at 101 instead of 162 without actual loss.

The chief point to note in these figures is the great difference in value received for coke which makes the above reduction in price of gas possible. The consumption and distribution cost on gas in the gasworks case appears to be handicapped in competition with their own coke, because roughly 8 B.Th.U. can be bought as coke at the same price as 1 B.Th.U. in the gas.

We have now to deal with the quality of coke-oven gas and the question whether it can be wholly used for town supply. Average coke-oven gas from which benzol and its homologues have not been removed only differs from coal-gas resulting from the destructive distillation of coal in a plain retort in that it generally contains about 5% by volume more nitrogen. If the benzol is extracted the illuminating value is low, but when this is not done average coke-oven gas can be taken as generally equal to the gas supplied in any town to-day, because the latter is a mixture of coal-gas with water-gas more or less carburetted. Further, it is possible to control and improve the quality of the portion of coke-oven gas taken for town supply if proper arrangements was made. Without going into close detail, in coking Durham coal it is found that illuminants rapidly diminish after the first half of the coking period. Methane diminishes, being replaced by hydrogen after two-thirds of the coking period. It is only in the final stages that nitrogen increases rapidly.

Probably the first offer to supply coke-oven gas on a large scale for town supply in England was made by Sir B. Samuelson and Co., Ltd., in 1903. They offered to supply gas to the Middlesbrough authorities to the extent of 800,000 cb. ft. per day at 7d. per thousand feet. The offer was not accepted, and as the official gas costs went on showing well over 8d. and up to over 9d. for some years the conclusion was that it may have been rejected on the score of quality. Therefore in 1912, when the company was considering the replacement of some old ovens, the question was reopened, and the suggestion made to overcome any objections on account of quality by setting apart the gas from

the early coking period for town use. To prove the effect of this on the quality of the gas the following experiments were carried out on an existing set of ovens. As it was decided to leave the benzol etc. in the gas supplied to the town, the extraction of benzol was discontinued by stopping the flow of oil on the benzol recovery scrubbers. The gases from ovens of lengthy coking period were shut off the main and blown away into the air during the time of the test:—

	Candle-power.	Gross, B.Th.U. per cb. ft.	nett.
Average gas before the test ..	8.8	541	498
Gas from ovens under 21 hrs. coking ..	10.9	579	535
" " " 18 " " ..	12.7	612	564
" " " 15 " " ..	14.1	647	598
" " " 15 " " ..	15.0	669	619
Average gas after the test ..	11.1	572	524

These experiments proved conclusively that by the provision of separate mains so as to isolate the gases from the early and later coking periods the quality of the gas supplied for town use could be controlled. The author would advise that this provision should be made if regularity of quality and especially illuminating value are in future to be considered of importance in town supply, because if the provision is not made there may be fairly large variations at times. Coke-ovens are exposed to atmospheric influence, and when a gale blows direct on the oven doors it is impossible to prevent some penetration of air, leading to combustion of gas inside the oven which destroys illuminants and increases the nitrogen content. The uplift caused by the column of hot material in the oven induces a suction upon the lower part of the oven even if considerable pressure exists in the gas space on the oven top.

Under the conditions of this separation of the gases the suitability of coke-oven gas for town supply has been proved by the arrangement which was carried through between Sir B. Samuelson and Co. and the Middlesbrough authorities. Since October, 1914, practically the whole gas supply of the town of Middlesbrough has been coke-oven gas. The average daily supply runs about 2,000,000 cb. ft., and no manufacture of ordinary gas has taken place since 1915. Shortages in the supply from the coke-ovens are made good by the manufacture of carburetted water-gas during the periods when town requirements exceed the oven supply. There has been no serious hitch in this undertaking, and its success has been assisted by harmonious co-operation of the representatives of the firm with those of the municipal gas authority. The public of Middlesbrough have obtained considerable financial benefit from this supply of gas, because, in the first instance, they could buy the gas cheaper than they could manufacture gas of equal quality, and they have not been called upon to pay an increased price due to the rise in price of coal. To-day this gas, with a selling price approx. 2s. 4d. per 1000 cb. ft. net, compares very favourably with any fuel for heating and cooking. The Gas Engineer informs me that its use has extended until the demand for cooking at meal hours exceeds that in lighting hours. This must be of importance in its influence in reducing distribution costs etc.

The author advocates that in all places where it can be adopted the regenerative coke-oven producing metallurgical coke is from a thermal point of view the most efficient means for gas manufacture, and that, compared with ordinary gasworks returns as published, if it can be adopted, it can supply cheaper and better gas to the town consumer at prices nearer the true thermal value.

I am indebted to Messrs. Dorman, Long and Co., Ltd., and Sir B. Samuelson and Co., Ltd., for permission to place this paper before you.

DISCUSSION.

Mr. T. HARDIE said that the results obtained by his firm in pre-war days did not correspond with

the figures given by Mr. Wright. Their returns from coke and breeze sales were nearer 55 than 45, and from other residuals nearer 28 than 24. In modern gas works it was possible to recover the waste heat not only from the settings, but from the coke itself, and the efficiency of such a plant was probably as great as that of any coke-oven plant, where much heat was lost in the discharge of coke. Coke ovens might have some advantage as regards handling large quantities of materials, but by modern mechanical handling methods that difference was considerably reduced as between gas and coke works. The use of coke-oven gas at Middlesbrough had been very successful, but he believed that a plant for the manufacture of gas had to be maintained there to supply gas in the event of the coke-oven supply failing. That plant had been put into operation fairly recently.

Mr. WRIGHT said that it was easy to understand that profits on coke and residuals at Newcastle were higher than they would be in the South or Midlands. The current price of gas in Middlesbrough was 2s. 4d. per thousand ch. ft., and he knew of no gas plant selling at less than that. The auxiliary gas plant had been erected because the coke-oven output was too small to deal with the increased demand.

On Tuesday evening the Lord Mayor gave a Civic Reception to the members at the Laing Art Gallery.

Wednesday, July 14.

A conference was held at Armstrong College on Wednesday morning, at which Prof. Henry Louis presided.

Dr. E. F. Armstrong, F.R.S., and Mr. T. P. Hilditch gave an interesting paper on "Catalytic Chemical Reactions and the Law of Mass Action," in which they described some of their recent work on the subject (see Proc. Roy. Soc., 1919, 96A, 322; 1920, 97A, 259, 265; also J., 1920, 120r).

SOME PROPERTIES OF 60:40 BRASS.*

BY CECIL H. DESCH.

The brasses used in engineering may be divided into two main groups, one of which is typically represented by the alloy containing 70% of copper and 30% of zinc, often known as cartridge brass, and the other by the 60:40 alloy. Alloys of the former class are composed of a single homogeneous solid solution, the α -constituent, and their most valuable characteristic is their great ductility, enabling them to undergo severe cold-working without cracking. On the other hand, the typical alloys of this class cannot be rolled at a red heat without great difficulty. The second class includes alloys with a duplex structure, the α -constituent of cartridge brass being accompanied by a second or β solid solution. The proportion of β increases from zero to unity as the percentage of zinc is increased from 36.5 to 46.5. This constituent has the property of being very plastic at high temperatures. Moreover, the ratio of β to α increases with the temperature, the normal ratio being restored on sufficiently slow cooling to the ordinary temperature. This property of plasticity makes it possible to shape the alloys which have a duplex structure by rolling, hot stamping, or extruding at or above a red heat, and rolled or extruded bars find many applications in engineering workshops. When cold such bars are readily machined.

In the course of using very large quantities of brass of approximately 60:40 composition in processes which involved many turning and drilling operations on automatic lathes, it was found that rods supplied by different manufacturers gave

widely differing results in the machine shops. In one instance a change from one brand of brass to another, nominally of the same character and composition, led to a drop of 40% in the output from the automatic lathes, whilst at the same time the wear on the tools was so greatly increased as to reduce their average life to one-fourth of its former value. The present investigation was undertaken with the object of throwing some light on the cause of such remarkable differences. A selection from the results has been made, and the present paper contains data relating to rods from sixteen different manufacturers. The experiments were mostly carried out in the Metallurgical Laboratory of the University of Glasgow, a few additional experiments having been made after removal to the University of Sheffield. I have much pleasure in expressing my indebtedness to Messrs. Wm. Cubitt and Co., London, to my father, Mr. H. T. Desch, and to Mr. Alex. Wylie, late of the Ministry of Munitions, for most of the material used in the investigation, and to Mr. A. N. McQuistan, of Messrs. R. Maclaren and Co., Glasgow, for permission to make experiments in the extrusion of trial mixtures in the works of that firm.

The composition of brass of this class always departs more or less from the nominal standard of 60% copper and 40% zinc, owing to accidental variations in the manufacture and to the presence of small quantities of other metals. A certain amount of lead is almost invariably added, with the object of improving the machining qualities; iron is usually present as an impurity; manganese or aluminium is frequently added as a deoxidiser when casting; and tin or nickel is sometimes added with the object of increasing the strength of the alloy. The rather high percentage of some of these metals in a few cases is no doubt to be attributed to the use of scrap in making the billets from which the bars have been rolled or extruded. The composition of the alloys used is given in Table I, the numbers in the first column being laboratory numbers, which are repeated throughout for reference. It should be stated that the zinc in all the analyses has been determined by difference. A few bars of exceptional composition have been included, as they were supplied to meet the same specification as the remainder, and they indicate the variations which are liable to occur in the supplies of metals which are nominally of the same composition.

TABLE I.

No.	Cu.	Zn.	Sn.	Fe.	Pb.	Mn.	Al.	Ni.	Apparent Zn.
351	60.02	39.57	0.13	0.03	0.23	—	—	—	40.04
352	59.98	39.50	0.13	0.04	0.31	—	—	—	40.07
609	59.92	37.84	tr.	0.08	1.99	—	—	0.17	40.0
610	59.79	38.83	0.62	0.12	0.64	—	—	—	40.5
611	60.04	39.21	tr.	0.13	0.62	—	—	—	40.0
612	60.04	44.04	tr.	0.10	0.61	0.08	—	—	44.8
613	59.27	39.00	0.06	0.15	1.47	—	0.05	—	40.9
614	57.97	39.66	0.15	0.14	1.95	0.04	0.09	tr.	42.3
615	56.97	40.50	0.07	0.12	1.95	—	0.20	0.19	43.6
616	56.62	40.19	0.49	0.35	1.89	0.11	0.31	0.04	44.6
617	59.82	38.86	0.23	0.25	0.78	—	0.06	—	40.5
618	60.01	38.39	tr.	0.06	1.54	—	—	—	40.0
619	57.95	40.72	0.07	0.27	0.89	—	0.06	—	42.1
622	58.29	40.23	0.32	0.50	0.66	—	—	—	41.7
623	56.56	40.98	tr.	0.32	0.99	1.02	0.13	—	43.5
694	64.13	34.67	0.49	0.49	0.09	0.07	0.06	—	36.3
712	49.16	49.78	—	0.07	0.99	—	tr.	—	50.8

When the proportion of one of the metals mentioned above does not exceed a certain limit it does not alter the essential characteristics of the $\alpha\beta$ brass, but is taken into solid solution, chiefly in the β constituent. When a larger proportion is present a third constituent makes its appearance, but no such separation has been observed in any of the alloys examined in the course of the present investigation, showing that the limits of saturation of the solid solutions were not exceeded. An exception

* Read in abstract, in the absence of the author.

must be made in the case of lead, which is visible in minute isolated globules in those specimens which contain a high proportion of that metal. A certain influence is, however, exerted on the structure by the presence of these foreign metals, since each metal, in passing into solid solution, has the same effect as a definite quantity of zinc. The structure of a complex alloy of this class is therefore the same as that of a pure copper-zinc alloy containing a somewhat different percentage of zinc. For many purposes, it is desirable to know the composition of the pure alloy which is identical in structure with the alloy under examination. Coefficients of equivalence have been determined, 1% of tin, for example, having the same effect on the structure as 2% of zinc. The other equivalents are: Aluminium, 6; tin, 2; lead, 1; iron, 0.9; and manganese, 0.5.¹

Representing the coefficient of equivalence of an element by t , and its percentage in a given alloy by q , we may calculate the 'apparent' percentage of copper in the brass, that is, the percentage of copper which would be contained in a pure copper-zinc alloy of identical structure, by means of a formula

$$B' = \frac{(B + tq) 100}{A + B + tq}$$

where B' is the 'apparent' percentage of zinc, B the actual percentage of zinc, and A the actual percentage of copper in the alloy. Should the alloy contain more than one added metal, the value of tq is calculated separately for each, and the sum Σtq is inserted in the formula. The values so calculated have been inserted in the table. As an example of this it will be seen that No. 612, containing 44.04% of zinc, has almost exactly the same structure as No. 616, which only contains 40.09% of zinc, the percentages of apparent zinc being 44.6 and 44.8, the latter value being raised by the added metals, aluminium especially, on account of its high coefficient, having a very great influence. As might be expected from a consideration of the microscopic structure, the mechanical properties are found to correspond more closely with the apparent than with the actual zinc percentage.

The photo-micrographs which accompany this paper represent longitudinal sections from bars, mostly of 2.4 in. diameter. The sections have been etched with an acid solution of ferric chloride, so that the β constituent appears dark and the α light. The size of the β grains is determined by the conditions of cooling in the higher range of temperature, all these alloys being composed solely of β immediately after solidification, whilst the crystals of α separate at a lower temperature, and their arrangement is mainly determined by the conditions of temperature distribution during the process of extrusion or rolling. Whilst the differences of structure between different bars are often strongly marked, it has not been found possible to distinguish the products of one works or even of one manufacturing process from those of another, as equally great differences may be found between bars made from successive ingots in the same casting shop, or even, in certain cases, between bars successively extruded from the same ingot. In the process of extrusion, the temperature falls considerably during the passage of the bar through the die, and the difference between the temperature at which the front end of the bar issues from the press and that at which the last portion emerges may, when the speed of extrusion happens to be rather slow, amount to as much as 100° C. Microscopical examination then shows two widely differing structures in different parts of the same bar. The outer and inner portions of a bar at the same cross section may also differ considerably in size of grain, but it has been observed that

the most fibrous bars, such as No. 618, are practically uniform throughout their section. It may be said that bars from those makers who were most successful in meeting the requirements of the machine shop were the most uniform in structure, so that bars from different consignments showed very little variation.

There is at present no satisfactory test by means of which the machining quality of a brass may be determined other than that of the workshop, and this test is only conclusive when continued over a long time, and with a large quantity of metal, in order to eliminate accidental variations in the sharpness of the tools, etc. It is shown below that tests of hardness in the ordinary sense, as revealed by the Brinell or similar methods, are of no value in determining the hardness of a brass towards a cutting tool. It was therefore thought advisable to use a workshop tool under conditions which could be easily regulated, and for this purpose a drill was selected. The machine used was a vertical, belt-driven drilling machine, from which the feeding arrangement had been removed and replaced by a lever, to the end of which was attached a quadrant, carrying a weight by means of a cord. The depth of the hole to be drilled was fixed by means of a collar attached to the vertical spindle. The bar to be tested was marked with a centre punch and placed in the vice immediately under the drill. The lever was raised to a definite angle and the height adjusted. The drill was then started, and the time taken to drill a hole to a depth of 1 cm. was noted with a stop watch. Each test was repeated, the values so obtained being very concordant. Three series of tests were made, the first and third using a $\frac{1}{2}$ in. twist drill at a speed of 300 revs. per minute, and the second with a $\frac{3}{8}$ in. drill at a speed of 700 revs. Fifteen bars were tested in this way. The first series showed a general increase in the time of drilling with increasing zinc, but the points did not fall at all regularly about any curve. The second series, at the higher speed, gave still more irregular results, and this was found to be due to the rapid blunting of the tool. The bars containing a high proportion of the β constituent were conspicuously hard when tested at the high speed, Nos. 612, 616, and 623 taking more than ten times as long to drill as the softest bars. The order of hardness of a series of brasses in a machining test depends largely on the speed of drilling, the angle to which the drill is ground, and the condition of its edge. (These factors have been carefully investigated for the case of steel by A. Kessner.²) The fine-grained brasses gave uniformly the best results at a high speed, but the figures have not been included in the table.

In the third series of tests, reverting to the slower speed and larger drill, and using certain added precautions, the conditions were more favourable. Drilling was more rapid, and possible errors due to the blunting of the drill were eliminated by testing bar No. 618, which had proved to be the best, at the beginning, middle, and end of the series, identical figures being obtained in each case. The results are shown in Table 2 and are plotted in Fig. 1.

TABLE 2.

No.	Machining time in seconds.				
	Third series.				
609	Extruded	27
610	Rolled	40
611	Rolled	38
612	Extruded	45
613	Extruded	29
614	Extruded	27
615	Cast	30
616	Extruded	28
617	Rolled	40
618	Extruded	25
619	Extruded	43
622	Cast	31
623	Extruded	40

¹ L. Guillet, *Rev. Mét.*, 1906, 31, 213.² Iron and Steel Inst., Carnegie Mem., 1913, 5, 10.

It will be seen from the figure that whilst no single curve can be drawn through the points, they group themselves, on the whole, into two sets, each lying near to a line which rises with increasing zinc content. In one of these sets the machining times are all above 35 secs. and in the other all below. The softer bars include the two cast specimens and five extruded bars. The harder set includes three rolled bars and three extruded bars, two of which were abnormally high in zinc and therefore contained an undue proportion of the relatively hard β constituent. The bars obtained by extrusion which have a normal structure fall into the softer group.

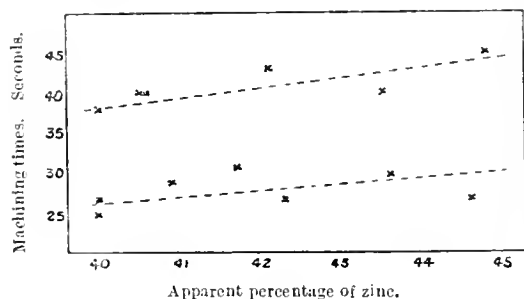


Fig. 1.

Remarkable differences in the appearance of the drillings were observed. Owing to the rapid blunting of the tool, the appearance of the drillings obtained at a high speed was less characteristic than in the other cases, but it was found that Nos. 611, 617, and 618, which have a fibrous structure, yielded long spiral drillings even at the higher speed. In the third series the longest spirals were given by Nos. 610 and 617. The cast alloy, No. 615, gave exceptionally light and feathery short drillings in all the tests that were made. The character of the drillings in the third series is shown in Table 3, the percentage of lead being also indicated, since the presence of lead is known to favour the formation of short drillings. The table shows that spirals are formed from the bars containing less than 1% of lead and short drillings from those which contain a larger proportion, the only exceptions being No. 612, which is high in zinc, and No. 622, the reason for which is not obvious.

TABLE 3.

Short borings.	Short spirals.	Long spirals.
609 .. 1.99% Pb.	611 .. 0.62% Pb.	610 .. 0.64% Pb.
612 .. 0.61	619 .. 0.89	617 .. 0.78
613 .. 1.47	623 .. 0.99	
614 .. 1.95		
615 .. 1.95		
616 .. 1.89		
618 .. 1.54		
622 .. 0.66		

It has been stated by several writers that the Brinell hardness of a brass is a satisfactory indication of its machining quality, and it was decided to test this point. No Brinell machine was available in the Glasgow laboratory at the time, so it was decided to employ the auto-punch method, which is known to give good results. A heavy Brown and Sharpe automatic centre punch was altered by removing the point and substituting an adapter holding a one-eighth inch bicycle ball. On holding the punch vertically against the specimen and pressing, a blow was delivered, and an impression was produced, the diameter of which could be measured under the microscope by means of an eye-piece micrometer. Four impressions were usually made on each part to be tested, each impression being then measured on two diameters at right angles, and the mean of the eight readings taken. The results are collected in Table 4. The hardness of

each bar was determined on a line about half-way between the centre and the circumference, and also on a transverse section, the portion near the centre

TABLE 4.

No.	Diameter of auto-punch impression.		Brinell hardness number.	
	Centre.	Edge.	Centre.	Edge.
609	1.35	1.30	80	80
610	1.20	1.15	85	129
611	1.32	1.27	82	95
612	1.21	1.24	107	107
613	1.35	1.25	87	97
614	1.32	1.29	97	100
615	1.27	1.33	85	89
616	1.24	1.30	97	97
617	1.18	1.22	103	107
618	1.19	1.17	113	110
619	1.41	1.23	82	95
622	1.42	1.33	107	92
623	1.32	1.24	97	121

and that as near to the edge as could be tested without distortion being tested separately. The results obtained at the centres of the transverse sections were, on the whole, the most concordant, but on plotting against either the real or the apparent percentage of zinc, a definite relation could not be found. The majority of the points lie in such a way as to indicate increasing hardness with increasing zinc, but three points lie so far from the curve that the differences must be more than accidental, and one of these three is No. 618, which is undoubtedly the best of all to machine, but which gives the highest value for the hardness by the auto-punch method of all the bars examined in that series. On repeating the experiments at a later date with a lever Brinell machine, the soft brass 618 once more gave as high a reading as the hard brass 612, containing an excess of the β constituent.

Table 4 also contains the results of tests made in Sheffield on the same specimens, using a Swedish Brinell machine of the usual type. The results are quite similar to those previously obtained.

It has been stated that the Brinell hardness of a brass may be accepted as a measure of the hardness in machining.³ Plotting the machining times against the Brinell numbers, however, no such relation is found, and an examination of the data contained in Mr. Ellis's paper shows that the conclusion is unjustified. When the data in Table E, on p. 36 of that paper, are plotted they are found to be distributed in an apparently random fashion, so that no curve drawn amongst them has any justification. The writer is convinced that the Brinell hardness is unconnected with the machining quality of a 60:40 brass.

The process of scratching a metallic surface with a diamond or other hard point closely resembles that of cutting with a tool, and it was therefore thought that a useful test of hardness might be made by means of the sclerometer, an instrument long used for the determination of the hardness of minerals, and modified for application to metals by Prof. Turner.⁴ Finding that the usual construction of this instrument was insufficiently rigid, so that the arm carrying the diamond was liable to shake during the making of the scratch, producing a wavy line on such a tough metal as brass, an improved instrument was constructed, in which the light pivoted arm was replaced by a rigid rhombic frame, supported by two hardened steel conical pivots six inches apart, and balanced so as to swing freely. The specimen was supported on a carriage moving between parallel guides, having an elevating pillar to accommodate specimens of different sizes, and screw traversing motion allowing several parallel scratches to be made. Four scratches were made on each specimen under the same load, and the breadth of each scratch was then measured at two different points under the microscope, using a Ramsden eyepiece

³ O. W. Ellis, J. Inst. Metals, 1917, 17, 25.

⁴ Proc. Birmingham Phil. Soc., 1886, 5, 291.

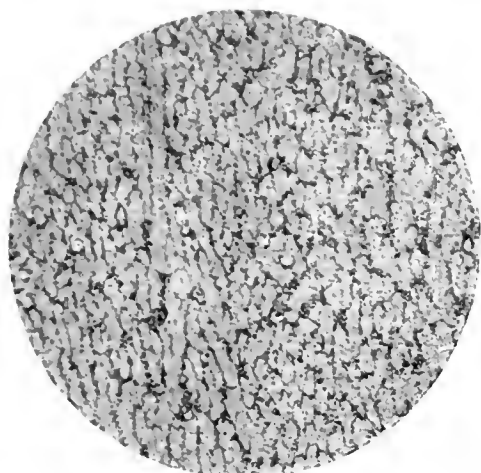


FIG. 3.
No. 351. $\times 50$.

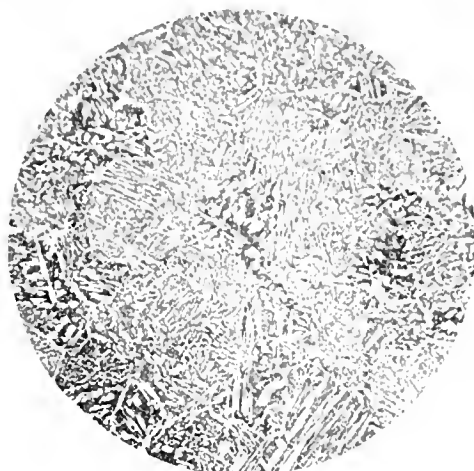


FIG. 6.
No. 615. $\times 20$.

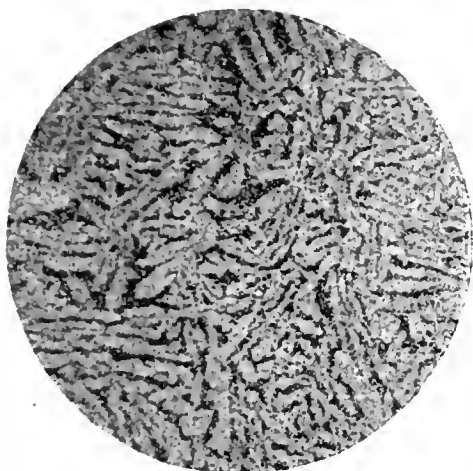


FIG. 4.
No. 352. $\times 50$.

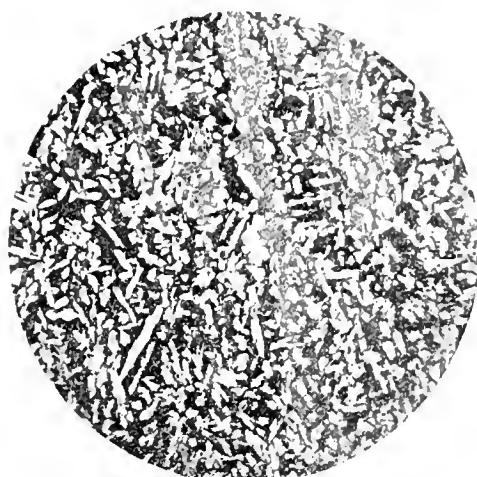


FIG. 7.
No. 616. $\times 50$.



FIG. 5.
No. 617. $\times 50$.



FIG. 8.
No. 618. $\times 50$.

micrometer. The surface of the specimen was always polished before testing.

The difficulty found in applying this method arises from its extreme delicacy. Duplex alloys, such as $\alpha\beta$ brass, show a scratch which is not uniform in breadth, but consists of a series of alternately broad and narrow portions, corresponding with the relatively soft α and hard β respectively. It is therefore necessary either to use a heavy load or to measure under so low a magnification as to obtain the average breadth. The results are shown in Table 5.

TABLE 5.

No.	Breadth of sclerometer scratch.		Divisions of eyepiece micrometer.	
			Centre.	Edge
609	53	52
610	50	50
611	52.5	51
612	44	40
613	47	37
614	50.5	36.5
615	53	50
616	49.5	43
617	42	33
618	52	50
619	37	35
622	53	52
623	39	37.5

Plotting the measurements against the machining times, no definite relation is observed beyond the sharp separation into two groups, previously noticed. It is intended to continue this part of the investigation, as it would seem desirable to have a laboratory method which would enable the working hardness of a brass to be determined with speed and accuracy, and a sclerometric method appears to promise more than any other as yet available.

It is frequently reported by machine shop workers that a bar has a hard skin on the surface, which causes blunting of the tool at the first cut. Brinell and scleroscope measurements across a transverse section of a bar frequently, but by no means always, show an increase of hardness as the edge is approached. This is due in part to greater closeness of grain in the outer portions and in part to cold working. The writer has found no evidence of hard particles of oxide in the outer layers. The bars as received are usually remarkably free from oxide, and in the few cases in which oxide has been found it is of a light, powdery nature. Neither can the skin hardness be attributed to rapid cooling at the surface, retaining the brass in the hard β condition. On the contrary, the outer zone is always slightly richer in α than the interior, owing to a small loss of zinc during the processes of manufacture. Attempts have been made to determine the hardness of the original skin, both by Brinell and by sclerometric methods, but the results have been unsatisfactory, the surfaces of some of the bars being so rough that accurate measurements could not be made, whilst grinding or polishing the surface would have defeated the object of the experiment.

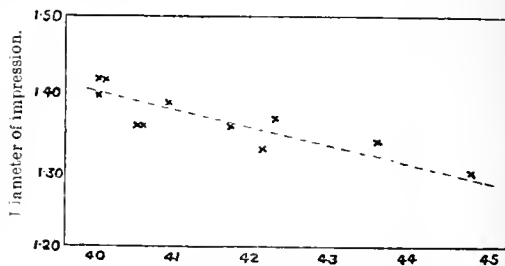
Two bars were received which had been pickled in acid in order to remove oxide. The machining tests did not indicate that the metal was appreciably softened thereby, and, in view of the evidence in the last paragraph, it is not to be expected that pickling would be of any great advantage.

Portions of some of the bars were annealed by heating for two hours in a muffle at 700° C., the specimens being packed in kaolin to avoid oxidation. They were then ground and polished, and tested by the auto-punch method. The effect on the structure was to coarsen it slightly and to cause rounding of the α crystals when these were previously angular. The diameter of the impressions is given in Table 6 and plotted in Fig. 2. The relation between the Brinell hardness and the per-

centage of apparent zinc is brought out clearly, the bars now being in a comparable condition.

TABLE 6.

No.	Hardness of annealed specimens.					Diameter of auto-punch impression.
						mm.
609a	1.40
610a	1.36
611a	1.42
612a	1.30
613a	1.39
614a	1.37
615a	1.34
616a	—
617a	1.36
618a	1.42
619a	1.33
622a	1.36



Apparent percentage of zinc.

Fig. 2.

The influence of the structure on the machining quality of the brass is well illustrated by Nos. 351 and 352. It will be seen from Table 1 that the composition of these two specimens is practically identical, but workshop reports gave the former as soft, and the latter as hard, in the automatic lathes. The photo-micrographs, Figs. 3 and 4, show that the α crystals are arranged in the one case in an almost parallel fashion, giving a fine fibrous grain to the metal, whilst in the other they are coarsely angular. The difference of hardness in this case is to be attributed to differences in the treatment of the metal after casting. Figs. 3 and 8 illustrate the conclusion, which the writer has reached as the result of this investigation, that a fibrous structure is favourable to ease of machining. It is only necessary to compare the photographs with the records of the machining times in Table 2.

A few words may be said on the subject of variations from the normal 60:40 composition. It is the practice of some manufacturers to use a considerably larger proportion of zinc in billets intended for extrusion. The higher the zinc the more readily is the alloy extruded under the usual conditions of temperature. This procedure results in the production of a bar which is hard to machine. By far the best results are obtained from an alloy containing 40% of apparent zinc, whilst the true percentage should be only slightly less, the difference being made up by lead. The quantity of the latter metal may approach 2%, but 1.5% is probably sufficient in most cases. Tin, iron, and aluminium should be reduced to the lowest possible proportions. The best alloy in the whole series, as proved by workshop experience extending over many months, was the American brass, No. 618, and this was used as the standard of comparison throughout the experiments. It has the highest chemical purity of any of the specimens and contains exactly 40.0% of apparent zinc. One of the worst is No. 616, which contains the largest quantity of impurities.

Extreme variations are not doubt sometimes due to errors in weighing out the ingredients before melting. Two examples are seen in Nos. 694 and 712. The former consists of the α constituent, with only occasional traces of β , whilst the latter con-

tains so high a proportion of zinc that the β crystals are bordered by layers of the γ constituent, an exceedingly hard and brittle substance of a light-blue colour. Brasses which contain even a minute quantity of γ are excessively brittle and may be broken by a light blow. Such a composition is only to be attributed to accident.

A comparison of the results leads to the conclusion that an arrangement of fine, fibrous α crystals, separated by a comparatively small quantity of β , is the most desirable structure for machining purposes. This is obtained by employing an alloy containing as nearly as possible 40% of apparent zinc, and extruding at a moderate temperature. Such an alloy, however, flows less readily than one with a higher proportion of zinc, and powerful presses have to be used. Some experiments were made with a series of billets containing 60.5% of copper, and these were extruded in the ordinary way from a 600-ton press. The billets proved to be very hard and were liable to stick in the press, so that three of the bars produced were only of one-half the normal length, half of each billet being left in the press. The structure was quite satisfactory, being fine and fibrous. It was ascertained that the presses used by the American firm which produced the brass No. 618 were of much greater power. It would be possible to use the presses of lower power for the 60:40 alloy, but the temperature would have to be high, and the fine structure would not be obtained.

DISCUSSION.

Mr. O. SMALLEY said that in a similar investigation, an alloy containing Cu 59%, Pb 1.25%, Zn 39.75% had proved to be the most easily machined so far as composition was concerned. With regard to physical examination, this had been limited by the fact of all the bars used having been extruded in a comparatively low pressure machine; the temperature range necessary for successful extrusion had been in consequence somewhat high, 760°–780° C. The result of this had been that the front portion of each bar had possessed a coarse angular structure and the back end the fine fibrous $\alpha\beta$ structure favoured by Dr. Desch. In view of this difference of microstructure, it had been of little use proceeding with the investigation on the lines taken by Dr. Desch, although experiments had been made to ascertain if any difference could be detected in the machining qualities of the front and rear ends of various bars, but with no result. During this investigation experiments had been conducted on the effect of cold work on 60/40 brass, and it had been found that the above-mentioned brass cold drawn 0.02–0.05 in. met all requirements, and machined as easily as the American bars which had been set as standard. The effect of this cold work on the physical properties of the brass was to increase the elastic limit 7–10 tons and the maximum stress 3–4 tons per sq. inch; structurally no change had occurred, the cold work penetrating uniformly to the centre of the bar. Subsequent to the completion of this investigation the workmen in one of the shell shops "downed tools" because of the difficulty in machining copper driving bands. Examination of these bands had shown that they had been made from pure copper containing 0.1–0.18% oxygen as cuprous oxide, and that they had been seriously over-annealed. By reducing the number of annealings in the cold drawing of the bands and finally annealing at 600° C., the elastic limit had been reached without materially affecting the tenacity or hardness. The bands had proved satisfactory and had met all requirements so far as machining was concerned. Later arsenical copper driving bands had been used. These had been found to machine satisfactorily, irrespective of the final annealing temperature. The effect of the arsenic (0.25–0.35%) was to raise the elastic limit without

materially influencing the other physical properties.

In view of these results he could only conclude that the elastic condition of the 60/40 brass was of more importance than the structural disposition of the microconstituents, as suggested by Dr. Desch.

CHEMICAL SHEET LEAD.

BY D. W. JONES.

Chemical lead is referred to by Percy ("Metallurgy of Lead") as follows:—"The purest lead, i.e., the softest, is stated to have been found best for sulphuric acid chambers, but . . . soft lead obtained direct from good ores is more durable . . . for such chambers than soft desilverised lead." Analyses are not given, and the probable reason for this observation will be referred to later. Lunge ("Sulphuric Acid and Alkali") quotes opinions, some of which incline to the belief that lead is the more acted upon (by sulphuric acid) the purer it is, whilst other opinions favour a lead containing small quantities of antimony and copper in order to make it more resistant. His own conclusion, arrived at after much work upon the subject is, that "at higher temperatures the purest lead resists both pure and nitrous sulphuric acid much better than regulus metal or hard lead." More recently Barrs (J., 1919, 407 T) has found an increase in purity in lead to correspond with an increase in the power of resisting attack by acid, but recommends the addition of 0.02–0.05% of copper to ensure obtaining the maximum resistance.

It is in the manufacture of and processes using sulphuric acid that the largest quantity of chemical lead is required, and, generally speaking, the property of resistance to attack by this acid which a lead may possess is a measure of its resistance to attack by other acids.

The quantity of lead required during the war for the above purposes exceeded by far any previous demands, and it was during this period that the following work was undertaken.

To determine the quality of chemical lead it is customary to make examinations by all or any of the following methods:—

(1) Heat in 96% sulphuric acid up to 290° C. in five minutes, cool to 100° C., and observe loss in weight.

(2) Heat in 91–92% nitric acid. No appreciable action should occur in fifteen minutes.

(3) Heat in a mixture of 40% of nitric acid, 52% of sulphuric acid, and 8% of water. No appreciable action should take place between 93° and 109° C.

(4) Heat a filed surface in a mixture of one vol. of hydrochloric acid, sp. gr. 1.14, 2 vols. of nitric acid, sp. gr. 1.50, and 3 vols. of water. The time required for spots of lead chloride to appear, and the time at which vigorous action takes place after boiling, are noted.

(5) Heat in concentrated sulphuric acid and observe the temperatures at which bubbles of gas are first evolved and at which the lead decomposes.

Of these methods No. 5 is the one most generally favoured. When under observation by this method in some cases the decomposition of lead is sudden, total, and violent, and is attended by a rise of temperature after removing the source of heat up to 27° C. With a good quality lead decomposition proceeds slowly, and is arrested when the source of heat is removed. The temperature recorded is that at which the solution becomes slightly turbid owing to the formation of lead sulphate. Since it has been found that 96% sulphuric acid (by wt.) gives figures comparable with those obtained when chamber acid is used, all observations refer to trials conducted with pure acid of this concentration.

The tests to be described were carried out under the following conditions:—A test piece of rolled lead 1 sq. in. in area and $\frac{1}{8}$ in. thick (corresponding

approximately to sheet 4 lb. per sq. ft.) is heated with 30 c.c. of 96% sulphuric acid at such a rate that the temperature of the acid reaches 300° C. in five minutes.

A suitable chemical lead when tested in this way evolves bubbles of gas at 190°—200° C. and decomposes at 300°—312° C.

pared from Parkes lead on a commercial scale by a process which is the subject of a patent granted to my firm in 1920. It contained Cu 0.0009% Bi 0.0007, Fe 0.0003, Ag 0.0005, Sb 0.0002, Zn 0.0019, Sn, As, Cd, Ni, Co nil, Mn traces, Pb (by diff.) 99.9955%. The results of these additions are shown in the following table:—

Behaviour of chemical lead when heated in sulphuric acid.

a. Gas evolved. b. Lead decomposes (°C.).

Percentage of addition.	Cu		Sb		Cu & Sb		Zn		P		Hg		Sn		Bi		Bi & Sb†	
	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b	a	b
Nil	202°	314°	202°	314°	202°	314°	202°	314°	202°	314°	202°	314°	202°	314°	202°	314°	202°	314°
0.005	211°	314°	208°	295°	197°	309°	—	—	—	—	—	—	—	—	205°	314°	204°	314°
0.01	209°	314°	198°	244°	204°	299°	189°	314°	—	—	—	—	197°	314°	198°	314°	210°	300°
0.02	205°	314°	201°	212°	211°	300°	180°	316°	219°	277°	189°	308°	—	—	198°	314°	188°	200°
0.03	196°	314°	203°	219°	207°	305°	200°	312°	—	—	—	—	200°	305°	199°	308°	—	—
0.04	193°	318°	203°	216°	192°	307°	197°	309°	—	—	—	—	—	—	198°	302°	—	—
0.05	209°	314°	201°	222°	202°	304°	176°	314°	—	—	—	—	—	—	198°	273°	—	—
0.06	207°	316°	205°	215°	193°	314°	—	—	—	—	—	—	197°	314°	198°	276°	—	—
0.08	—	—	—	—	—	—	—	—	219°	29	189°	283°	—	—	—	—	—	—

† With 0.04% Bi and 0.005% Sb a=199°, b=270°; with 0.04% Bi and 0.01% Sb a=198°, b=238°C.

Remarks.—With the original metal, and also when copper was added, partial decomposition occurred, the action soon ceasing when the source of heat was removed. With 0.005% Sb total decomposition was observed, lead sulphate being formed at 270°C.; with higher percentages total and violent decomposition occurred. With Cu and Sb there was partial decomposition, with indication of lead sulphate about 10° below decomposition point. With zinc partial decomposition occurred; no indication of lead sulphate except with 0.02% and 0.05%. With phosphorus and mercury, total decomposition was observed. With tin, partial decomposition occurred, with slight indication of sulphate at 260°C. With 0.005% and 0.01% Bi decomposition was partial, with 0.02—0.04% total, and 0.05—0.06% sudden, total and violent. With 0.005% Bi and Sb decomposition was partial, with 0.01% total, and with 0.02% more sudden, violent, and total.

The following figures have been obtained from the examination of many hundreds of samples of lead refined by the Parkes process, including English, Spanish, Australian, and American supplies:—Bubbles of gas evolved: average 187° C.; maximum, 212°; minimum, 146°. Lead decomposes: average 235°; max., 280°; min., 183° C.

Thus Parkes lead without further treatment is unsuitable for use in many operations. To test the effect of impurities on the chemical properties of lead additions of pure metal were made to a Parkes lead containing Cu 0.003%, Bi 0.007, Fe 0.0018, Ag 0.0005, Sb 0.006, Zn 0.0018, Sn, Ni, Co, nil, As, Mn, traces, Pb (by diff.) 99.9799%. The results of these additions are shown in the following table:—

Behaviour of Parkes lead when heated in sulphuric acid.

a. Gas evolved. b. Lead decomposes (°C.).

Percentage of metal added.	Cu		Sb		Cu & Sb		Na		Hg	
	a	b	a	b	a	b	a	b	a	b
Nil	191°	254°	191°	254°	191°	254°	191°	254°	191°	254°
0.005	190°	277°	179°	205°	186°	259°	—	—	—	—
0.01	198°	281°	180°	210°	190°	275°	—	—	—	—
0.02	195°	288°	179°	212°	189°	284°	194°	275°	173°	257°
0.03	190°	291°	180°	206°	203°	289°	—	—	—	—
0.04	176°	290°	179°	202°	198°	286°	—	—	—	—
0.05	175°	295°	182°	203°	197°	277°	—	—	—	—
0.06	185°	296°	192°	220°	190°	284°	—	—	—	—
0.08	—	—	—	—	—	—	193°	282°	—	—

With copper and with copper and antimony together, and also with sodium, partial decomposition occurred. At 0.06% Cu and Sb local action was observed, streams of lead sulphate being formed which dissolved 20° below the recorded decomposition temperature. With mercury total decomposition occurred.

It is of interest to note how the addition of a number of elements affects the resistance to attack by sulphuric acid upon a lead known to possess this property. For this purpose the lead used was pre-

This work has been confined to the addition of comparatively small amounts of impurities, many of which are known to exist in commercial lead, with the object of devising methods of eliminating those impurities found to possess a deleterious effect.

From the above tables it is evident that the presence of antimony in lead to the extent of 0.01% renders it unfit for use in the manufacture of chemical plant.

With 0.06% of added antimony the temperature of initial attack is restored, but as this amount is never found in commercial lead (which is usually of a purity of 99.98%) and it is not conceivable that an addition of 0.06% antimony would materially lead to additional strength, there can be little or no advantage derived from the fact.

Hard lead sheet and pipe containing 0.75—5.25% Sb in no case gave a higher decomposition temperature than 244° C., but at temperatures above 220° C. signs of gas bubbles are first observed when this type of lead is heated in concentrated sulphuric acid.

Antimony, therefore, if present in lead in practically any proportion above the most minute, will, in the absence of a known corrective agent, be the cause of immediate breakdown if temperatures much over 200° C. are employed.

Copper also has a marked effect in reducing the temperature of initial attack by sulphuric acid. The figures in the tables point to the fact that there is a danger zone lying beyond 0.03% in lead of the Parkes type, and in the "chemical" lead clearly defined between 0.03 and 0.045%. If copper be introduced, due consideration must be given to the above-mentioned factors.

The effect upon the decomposition temperature of the addition of copper upon a Parkes lead is most beneficial; 0.005% introduced shows a rise of as much as 23° C., and at the same time is sufficient to prevent the vigorous action which causes the whole of the lead to be converted into sulphate and consequent total destruction. The gain achieved by introducing 0.03% is 37° C., and brings an ordinary lead within measurable distance of chemical lead. No remarkable effect ensues from the addition of copper to a pure chemical lead; a gain of 4° C. at 0.04% addition is all that calls for comment.

Most probably the reason for the statement previously quoted from Percy is that the soft lead derived from pure ores contained about 0.01 % Cu; similar lead to-day approximates to that amount and compares very nearly in all respects with Parkes lead when 0.01 % Cu has been added. Such lead may have been more resistant to acid than desilverised lead manufactured at that period, especially when it is remembered that pan concentration of sulphuric acid was more universally in use. Lange ("Sulphuric Acid and Alkali") at a later date has stated:—"Hence the purest lead is subject to instantaneous solution by sulphuric acid at 260° C." Pure lead is, however, manufactured to-day which withstands the action of hot concentrated sulphuric acid without being instantaneously dissolved when heated beyond 300° C.

The effect of copper in counteracting the harmful influence of antimony in lead is evident from the tables. In no case is total decomposition brought about, which, with antimony only present to the same extent, would result in every instance. With between 0.02 and 0.03 % of antimony and copper there seems to be a rise in the temperature of initial attack; this fact may be of some value if lead containing these amounts of antimony and copper is used for purposes dealing with concentrated acid at comparatively low temperatures.

Zinc, when present in lead even in very small amounts, is generally supposed to destroy the power to withstand the corrosive action of acid in a marked degree; this is not borne out by any work undertaken by the author, which embraces the examination of chemical lead prepared by different methods to which additions of zinc have been made. The temperature at which the first action is discernible is lowered in all cases. At 0.02 and 0.05 % a degradation of resistivity is evidenced by the formation of lead sulphate much below the temperature of complete solution. At 0.03—0.04 % Zn a critical stage exists where this injurious effect is not operative.

Mercury when alloyed with lead has a deleterious effect upon its "chemical" properties. By amalgamation this effect is more serious. If 1 % of mercury is rubbed over the surface of a piece of chemical lead until amalgamation appears to be completed, allowing one hour as contact time, the product is very sensitive to the action of hot concentrated sulphuric acid. At 170° C. lead sulphate is formed copiously. If the heating be discontinued at this stage the action of the acid will cease on cooling to 70° C. After washing the lead sulphate from the trial piece it will be observed that pitting has proceeded over the entire surface, and minute globules of mercury can be seen under the microscope. If this piece of lead be again heated with sulphuric acid action will commence at 180° C., and sudden, total, and violent decomposition will take place at 220° C. Repeating the above, but allowing contact overnight between the lead and mercury for more complete amalgamation, a distinct action is noticeable on heating to 50° C. At 100° C. total decomposition accompanied by the usual phenomena takes place. Using 0.1 % of mercury on the weight of lead of standard size (i.e., just sufficient to cover one-third of the area), and allowing two hours for amalgamation, a distinct vigorous action commences at 100° C., and total solution takes place at 215° C.

These observations indicate that great care must be exercised when using hydrometers etc., for if these instruments are broken and the mercury permitted to amalgamate with the lead, local action in the affected part, followed by a breakdown, can be expected.

Tin has a degrading influence upon lead intended for chemical purposes, though not to the extent generally believed. Since it is easily removed by

refining methods of to-day, it is not usually found in lead in more than minute traces.

Bismuth, so frequently associated with lead of American origin, is decidedly injurious if present above 0.01 %, and even at 0.02 % it causes total decomposition in an otherwise resistant lead. The addition of 0.02 % of copper to lead containing 0.05 % bismuth will be sufficient to raise the temperature of decomposition from 273° to over 300° C.

The combined effect of antimony and bismuth is noteworthy. The addition of 0.005 % of each metal leaves chemical lead practically unaltered, but when 0.02 % antimony and 0.02 % bismuth are present there is a difference of only 12° C. between the temperatures of first action and of violent decomposition.

Doubtless the poor chemical quality of Parkes lead in some instances is due to its containing antimony and bismuth in some peculiar proportion, as singly their effect when added to a pure lead is not so pronounced.

Investigating the cause of the breakdown of lead concentrating pans, Hart (J., 1907, 504) stated that 200° C. is never exceeded in this class of work, and that the trouble occurs when the acid has reached a sp. gr. of 1.65—1.675, and may occur at sp. gr. 1.63 after constant use for a long period. A possible explanation of these defects might lie in the peculiar behaviour of pure lead to which antimony and bismuth have been added. Though these metals may not originally be present in lead in the exact proportion which leads to decomposition at or even below 200° C., a selective action of sulphuric acid upon lead known to contain bismuth and antimony may ultimately bring about conditions resulting in breakdown in pan concentration.

A practical method favoured by acid manufacturers of testing lead intended for chambers is to expose sheet, previously weighed and measured, to the exit gases of a No. 1 chamber (the gases at this point are considered most corrosive) and note the loss per unit of area exposed over a given period, usually not less than three months.

The following are the details of a trial, as described, upon four different samples of sheet lead exposed to the action of chamber gases for 130 days:—

Description.	Behaviour when heated in sulphuric acid.		Area sq. in.	Loss per sq. in. gms.
	Gas evolved °C.	Lead decomposes °C.		
Pattinson lead	200	316	19.500	1.274
Do. + 0.38 % Cu	215	325	19.132	1.323
Pattinson lead (old sheet from the side of a No. 4 chamber)	210	300	26.800	0.749
Chemical lead prepared from Parkes lead	202	314	19.750	1.254

The behaviour of sheet lead which had seen previous service is remarkable; this had a fine coating of lead sulphate on its surface, which was not removed before beginning the trial. It is possible that, not being an immersion test, a protective action is exercised by this coating of lead sulphate. Apart from this result pure lead shows the smallest loss, and a Pattinson lead shows a smaller loss than the same lead to which copper has been added.

Assuming the loss of pure lead to be unity, and that lead of 7 lb. per sq. ft. is under consideration, the increased loss suffered by Pattinson lead in chamber work as compared with pure lead would be 5.7 lb. per ton of lead per annum, and for Pattinson lead containing 0.38 % added copper 19.6 lb. per ton per annum.

Of the whole of the elements used for additive purposes, there are only two which consistently

exert a beneficial influence upon the chemical properties of lead, *viz.*, copper and sodium; this is most marked with non-chemical lead, and in the case of copper does not apply to any extent to additions made to a pure chemical lead. Avoiding those quantities which produce a fall in the temperature at which the first action is discernible, something may be said in favour of lead containing copper on account of a gain in strength and rigidity thereby secured. It is by fatigue, produced by constant use above normal temperature, that lead often becomes unreliable; here again the introduction of copper should prove advantageous.

Of other additions it may be said that they are injurious in their effect, though in most cases not to the extent generally supposed. There is one outstanding instance, *viz.*, antimony, which, if present in lead in quantities which a commercial metal may contain, will effectively debar its use as "chemical" lead.

Chemical lead is sometimes subject to local action, attributed to oxides which in places become rolled out into the sheet. By a careful control of casting temperature alone this source of danger can be largely eliminated. Lunge gives the oxygen content of pure lead as 0.0024%; with antimony and copper present the oxygen can be as high as 0.005%. This again is advanced as an argument in favour of the use of pure lead for chemical purposes.

Reference to the table (page 222 r) will show that local action is most pronounced when copper and antimony are together present in lead.

In spite of the fact that copper can function as a corrective agent and will counteract the injurious effect of antimony and bismuth in lead, the author is yet in agreement with Rhead (see J., 1920, 128 r), who has emphasised the importance of using pure lead in connection with acid plant.

In conclusion, my thanks are due to Messrs. Locke, Blackett and Co., Ltd., for permission to publish this work.

DISCUSSION.

Dr. WEYMAN asked to what extent the impurities were introduced into lead after it had been put into use. It was conceivable that lead used for lining iron vessels might become contaminated with iron.

Mr. R. T. HARRISON thought that nearly all impurities in lead came out of solution when the lead was cooled. It had been shown that by immersing lead in acetic acid the impurities were dissolved out and brittle lead was produced.

Mr. H. DUNFORD SMITH said that he could confirm nearly everything the author had stated. He rather doubted, however, whether the test with sulphuric acid was representative of the conditions in the chamber itself. The author had cited four instances of lead being hung in the exit of No. 1 chamber in which he had shown that old lead which had previously been used in the chamber underwent less corrosion and loss of weight than even his own pure lead which he placed in the highest position. It seemed to him, therefore, that while the test was very useful for sorting out the good leads from poor leads, it did not differentiate between the different degrees of the better class leads. He (the speaker) had made tests of lead containing copper in chambers which had shown up much better than many pure leads. If Mr. Jones could in some way correlate his tests with actual practice it would be of great value.

Dr. FLECK suggested that the beneficial effect of sodium might be due to the fact that it reduced the quantities of oxide present.

Mr. JONES, in reply, said that he had made no chemical tests of the lead lining of iron tanks, but he had had experience with the use of a tank lined with lead to dissolve sodium sulphide in river water; that tank had been in use for five years and

had required no attention. He thought that with a good lead there need be no fear of contamination from an iron tank. His experience indicated that the addition, say of 5%, of copper to crude lead would inhibit atmospheric corrosion. When sodium was added to lead there seemed to be evidence of reduction, and Dr. Fleck's theory was probably correct.

The CHAIRMAN said that the paper had been interesting to him as showing how the work of his old teacher, Dr. Percy, had stood the test of time. He was wonderfully accurate in spite of the difficulties of the early days in which he worked.

Mr. H. C. LANCASTER writes:—

"I should be interested to hear if the author has satisfied himself that the pure metals added to test the effect of impurities were actually present in the final lead in the same proportions as when added. I have proved conclusively that it is difficult even to get 50% of the added copper to be retained by lead, although it is a very simple matter to get retained almost a true equivalent of antimony and the other metals of low melting point. The author refers to a Pattinson lead containing 0.38% of added copper. My experience has proved conclusively that a chemical lead, and certainly a Pattinson lead, will not retain 0.38% of added copper. For instance, I have found that at the necessary temperature pure chemical lead dissolves quite easily 0.5% of added copper, but when the lead is afterwards melted and poured into a mould the resulting lead contained only 0.15% Cu, showing that this copper has a great tendency to liquefy out at comparatively low temperatures, and this is, of course, a condition always present when manufacturing sheet lead. In my opinion, it is impossible under normal conditions to produce a sheet lead containing 0.38% of copper, especially if the lead is otherwise very pure, although impurities do tend to keep copper in a lead, especially in the presence of arsenic and antimony."

RECENT DEVELOPMENTS OF THE ELECTRIC FURNACE IN GREAT BRITAIN.

BY D. F. CAMPBELL, M.A., A.R.S.M.

In no branch of metallurgy have greater advances been made during the war than in the development of iron and its alloys, which has given us materials of extraordinary strength and physical properties. Steel for aero engine parts owe their power and lightness to the addition of small percentages of metals, aeroplane wire its strength and bullet-proof steel its toughness to the addition of suitable alloys, such as those of nickel, chromium, molybdenum, or manganese, and the electric furnace has been responsible in most cases for both the reduction of these refractory metals from their ores and the subsequent manufacture of the alloy steels for these special purposes.

Newcastle has been the principle centre for the reduction of chromium, tungsten, and molybdenum ores, and at the time of the Armistice works were in regular operation of sufficient capacity to produce Britain's war requirements of these essential metals.

In 1914 the quantity of energy used on electric furnaces in Britain, excluding those used for aluminium, was probably less than 6000 h.p., but on the day of the Armistice the total capacity was in excess of 150,000 h.p., of which 135,000 h.p. was producing steel, and 18,000 h.p. was working on the reduction of chromium and tungsten ores. On the day of the Armistice electric energy was being consumed at the rate of nearly 300,000,000 kw.-hrs. per annum for furnace work alone. The electric steel production had then reached a total of over 200,000 tons per annum.

Similar progress has also been made in the electrolysis of brine for the manufacture of caustic soda

and chlorine to meet war requirements, and the older processes are now superseded by the electric method of manufacture.

The manufacture of large quantities of phosphorus by electric-furnace methods was necessary during the war, and the excellent fused silica wares for laboratories and chemical works, made with such remarkable success on the Tyne, is perhaps the most interesting example of the use of the electric furnace for fine work.

The making of large electrodes, an essential subsidiary industry for electric furnace work which was unknown in Great Britain in 1914, is now established in Newcastle and elsewhere, and also the manufacture of artificial graphite in the form of electrodes for furnace work and electrolysis and powder for accumulators and similar purposes.

But the main interest lies in the reasons for the extraordinary development of electric furnaces and the technical advances which have been made.

Electric melting enabled us to use advantageously the vast quantities of steel turnings made in the shell factories, and the continuous traffic of lorries removing turnings direct from the lathes to the furnaces of Sheffield soon disposed of the vast accumulations of rusting material which were so commonly seen in the early years of the war.

The re-melting of nickel-chrome steel is a wasteful and most difficult process in all other furnaces, but the reducing conditions and absolute control of the electric furnace make it possible either to retain or remove at will most of the chromium, and thus large quantities of a valuable metal were saved.

The economic manufacture of stainless steel has been rendered possible only by the electric furnace, which is required both for the preparation of the refined ferro-chrome and the steel melting. This remarkable steel, containing 13% of chromium and a low percentage of carbon, will surely have a wide range of usefulness in engineering as well as in domestic fields. Its properties of resisting vegetable acids and corrosion from many other causes has made it a most valuable substance for valves, hydraulic equipment, and other engine parts. It can only be made by the use of ferro-chrome containing a very low percentage of carbon, and the preparation of this metal presents very serious difficulties. These have, however, been completely overcome in this district, the ore from India, Rhodesia, or New Caledonia being reduced and subsequently decarbonised to the required extent in electric furnaces.

The application of electricity to metallurgy has given us the low-hysteresis iron that is now universally used in transformers, for this is made by the addition of high-grade ferro-silicon to the steel. A gain of $\frac{1}{3}$ % in efficiency on a single transformation of the thousands of millions of units generated annually in Great Britain is equivalent to saving scores of thousands of tons of coal per annum. This material is still capable of improvement, and it is hoped that extensive research will be undertaken forthwith.

The use of the arc furnace in one of its many forms is now almost universal, and the fascinating principle of induction heating is no longer used. Electric furnaces owe their value to their intense reducing conditions, as they utilise the only practical source of heat which does not require oxygen for its generation. The furnace may be a closed box, e.g. when used for the reduction and volatilisation of phosphorus or for the final deoxidation of steel. Another fundamental advantage of arc furnaces is due to the fact that the slag is necessarily hotter than the steel, and the refining action takes place at the surface of contact between the slag and the steel, whereas with induction the slag was always cold and refining practically impossible.

The tendency in electric furnace design is rather to increase the power of transformers on furnaces of moderate capacity than to build large furnaces.

One limiting factor is the size of electrodes that can be conveniently employed; but if there were any demand for larger furnaces, there is no objection to the use of six or more electrodes.

Where large tonnages of electrically-refined steel are wanted, it is customary to refine steel previously melted and treated by the Bessemer or Thomas process, and in that case the electric furnace need never be of more than 25 tons capacity, which is the largest vessel used for these processes.

There are technical disadvantages in electric furnaces of more than 25 tons or more than 3000 kw. capacity, and there is no great advantage in using the furnaces of 6000–7000 kw. which have been recommended abroad for making alloys and carbide.

In most furnaces of the arc type, regulation of load is effected by variation of the arc gap, either by hand or automatic regulation. The latter system has probably been more highly developed in this than in any other country, owing to the high cost of power, and the system by which individual furnaces are kept at any desired constant load by Thury automatic regulators is widely applied. The regulation also materially assists in keeping both the composition of the bath and temperature of the furnace within desired limits. In addition, the regulation of a battery of furnaces by a master instrument is of especial interest to power producers and those who pay for energy on a maximum-demand basis. An instrument is finding application which will keep the total load of a battery of furnaces within desired limits by slightly reducing the load on the furnaces if the demand at any time exceeds the total load which the station engineer or the power contract may set as a maximum total load to be utilised. Individual furnaces can be removed from the influence of the master regulator by drawing a switch, but the energy absorbed by that furnace is always included in the total power which the regulator will allow the furnaces to absorb.

The lack of understanding between the power consumer and energy producer often leaves much to be desired, and greater efforts should be made to realise the difficulties and requirements of the two undertakings, which are so entirely interdependent, if electro-metallurgical industries are to succeed.

The preparation of contracts for supply requires the most careful consideration by engineers who have experience both of power generating and also of electro-metallurgy, if harmonious working is to result.

The development of electrical energy on the North-East coast is undoubtedly the most progressive and comprehensive system in Great Britain, and it is hoped that the development of other districts will result in schemes for linking up existing supplies, exchange of surplus capacity with a general reduction of stand-by plant, and the erection of a few large stations at suitable points, so that the small and inefficient stations may be abandoned or relegated to the position of emergency stand-by plant.

There are great advantages in establishing certain electro-metallurgical processes in England in spite of the lack of water power, for this country is so highly favoured with the other essentials for successful industry that the supposed advantages of cheap hydro-electric energy are counterbalanced in the case of many processes in which skill and cheap raw materials are an important factor; consequently power economically generated from steam and efficiently distributed may form the basis of many electro-chemical industries.

Coal-generated power is constantly available, whereas water-power is only too often subject to great seasonal variations. Seasonal labour and variation of output all tend to raise the cost of production. Consequently, only few industries, comparatively easy of accomplishment and requir-

ing large energy consumption, flourish in the centres where water power is available.

The production of aluminium, ferro-silicon, and carbide are industries for which water power is usually essential, but in steel-making the quantity of energy used is comparatively small, and the skill and special plant required for its treatment are so great, that the local advantages of England outweigh the advantages of low power cost abroad.

Processes such as steel-making that only use about 40% load factor, i.e., four-tenths of the possible output of the plant reserved for their use, are more suitable for steam stations, which have low capital expenditure and high running costs, rather than hydro-electric stations, which involve the outlay of large sums for capital account, but small operating expenses.

The utility of the electric furnace did not cease on the day of the Armistice. Shops equipped with electric furnaces for war alloys have been reconstructed and are producing alloy steels. The manufacture of artificial graphite, a new industry to this country, has been established, and electrodes of a size and quality equal to the best in the world are being produced, and the manufacture of alloys and amorphous electrodes is being developed along the lines of present requirements.

The next immediate development will probably be the general application of electric smelting to the treatment of complex zinc ores and the gradual improvement of existing processes in the development of power and utilisation of the fuel we now waste.

Electro-metallurgy is still in its infancy, but its growth is rapid. Abroad generating stations of 100,000 h.p. are being used in some cases for single industries, and electro-metallurgical centres absorbing several hundred thousand horse-power are being developed in various countries, while many similar projects are under consideration.

Agriculture, the greatest of all industries, requires that nitrogen be rendered available for the growth of plants, and the whole problem of artificial fertilisers and the influence of electric discharge in agriculture affords an intensely interesting subject for research.

The rapid growth in the world's consumption of aluminium and the great possibilities of its alloys offer a fruitful field for investigation and industrial enterprise.

Improvements in the metallurgy of steel, and the study of alloy steels in particular, have enabled engineers to produce the aeroplane, and will doubtless lead to lighter and stronger structures, such as bridges, locomotives, and ships, with a corresponding reduction in weight and increase in efficiency.

Peace is demanding new products and extraordinary opportunities are apparent on every side for maintaining the supremacy of science and engineering which we established during the war.

DISCUSSION.

Mr. A. G. LOBLEY said that electrodes for aluminium furnaces had been made in this country ever since aluminium had been made here. Moreover, both the companies making aluminium in this country could produce a better electrode than was produced abroad for the purpose. No reference had been made to the resistance type of furnace. At least one furnace of that type was working now on a large scale. The development of the resistance type had presented many difficulties, but he had now overcome these, and a furnace was running efficiently in the hands of workmen without any electrical knowledge. The type had the advantages of clean working and general efficiency.

CHEMICAL ENGINEERING GROUP.

CONFERENCE ON FILTRATION.

On Tuesday afternoon and Wednesday morning the Chemical Engineering Group held a conference on Filtration, at which a number of papers were read and discussed. The full papers, with discussion, will be published later in the Proceedings of the Group.

Some of the papers are given below in abridged form.

THE PRINCIPLES OF TECHNICAL FILTRATION.

BY EMIL HATSCHEK.

Filtration in the accepted sense of the term is generally defined as the separation of liquids from solid particles of microscopic size suspended in them, by means of a porous septum which allows the liquid to pass but retains the particles. The definition is somewhat narrow, as, on the one hand, it has been shown by the author¹ that particles of one liquid suspended in another can be retained by suitable media, while, on the other, colloidal particles of sub- and even amicroscopic size can be separated by a variety of membranes. The procedure last mentioned is known as "ultra-filtration," and, while it is an important instrument of research, has not so far found application on an industrial scale; such application, however, should be quite feasible, a point which will be referred to again.

The first and fundamental point to be noted is that, for a given system of solid and liquid, the whole problem is completely determined physically. The size of the pores in the septum is fixed or has, at any rate, an upper limit; the largest number of pores of such size is, of course, desirable, but this is a technical matter and simply means a choice among a comparatively small range of materials. As soon as filtration begins the pores in the surface of the filtering medium are throttled more or less by the particles which settle on them, and as it proceeds the liquid has to escape through these throttled orifices and through a layer of particles of increasing thickness. The structure of this layer is also fixed by the size and shape of the particles, and is, for a given precipitate, remarkably constant. The resistance offered to the flow of liquid varies considerably from one material to another, but in all cases it increases as the thickness increases, until a limit is reached, i.e., until, with a given pressure the rate of flow becomes so small that it is advisable to discontinue the process. This limit is an *economical* one only; it depends, roughly speaking, on the way in which higher pressure increases the first cost of the plant, and, the pressure being given, on the balance between the cost of obtaining a thicker "cake" and the cost of removing it at more frequent intervals. The proper adjustment of these factors presents no difficulty to anyone experienced in designing filtering apparatus, and should be left to him.

The second point is that since the structure of the cake is fixed by the size and shape of the particles, not only the pressure required to force through a given thickness of it, but also the percentage of mother liquid remaining in it at the end of the operation is fixed within surprisingly narrow limits. It is not affected by the design of the apparatus nor by the pressure used—a point which seems self-evident, but is realised apparently by an exiguous minority of technical men. No experience is more common than to be asked to design a filter which will turn out a cake containing "not more than 20% of mother liquor," the cake being, e.g., calcium sulphate, which contains about 40%. This

figure is the same whether filtration has taken place in filter presses or on vacuum filters, through filter cloths or through porous tiles. Only actual experiment can, in the case of an unknown residue, give any information regarding the amount of liquid retained by it. It is hardly necessary to say that the appearance and consistency of the cake are no guides; an excellent "cake," say, of calcium carbonate may turn to a fairly fluid sludge during carting, owing to a slight rearrangement of the particles. In cases where the particles are not solid crystals, but themselves aggregates, the moisture content may be remarkable while the cake can still be handled perfectly. Thus a cake of the "magnesium carbonate" used as non-conducting covering retains about 75% of mother liquor, owing to the very peculiar structure of the material.² The author once showed a specimen of this cake to a client used to China clay, who guessed the percentage of moisture as 25%, or thereabouts.

The case appears even more complicated when the residue is highly hydrated, *e.g.*, anorphous silicic acid. In this case the residue contains not only the mother liquor between the particles, but the water of hydration in the latter, all, or nearly all, of which would be driven off on drying at 100° C. The only way to determine in such a case the percentage of mother liquor, as distinct from water generally, is to titrate whatever constituent of the mother liquor is most characteristic.

In a great number of processes it is necessary to remove the mother liquor as completely as possible, either because it contains the valuable constituent or because its presence in the cake is detrimental. The most common method is to displace it by water (or other solvent), *i.e.*, by "washing" the cake, as it is usually called. The ideal—displacing the mother liquor by an equal volume of water or solvent—is practically unattainable, as a certain amount of diffusion is unavoidable, but where this is slow the volume of wash-water may be very little in excess of the volume of mother liquor to be displaced. The process of washing resembles the process of filtration, with two important differences: the liquid passes through a constant, instead of a gradually increasing, thickness of residue, and the process, unlike filtration itself, is not self-regulating. To illustrate this point, we may take the case of a suspension being filtered through a material, the porosity of which varies slightly at different portions of the surface. The liquid passes more rapidly through the more porous portions, and a correspondingly larger amount of solid is deposited in a given time, which checks the flow and tends to equalise it with that through the less porous parts. If wash water is now passed through, the difference in porosity, although greatly reduced by the layer of deposit, will still cause the flow of wash water to be more rapid at some spots than at others, and this inequality no longer can correct itself, so that washing is uneven. Differences in the structure of the cake, due to other causes, will have the same effect.

The point to which washing is carried is again settled by economic considerations—such as the balance between the cost of evaporation and the value of the dissolved substance—unless, for instance, traces of the mother liquor are absolutely detrimental to the purpose for which the residue on the filter is required. If this is the case, the end point is by no means easy to determine; generally speaking, it is not sufficient to test the washings only, but the cake itself should be examined. Apart from very exceptional cases, adsorption always occurs on the particles of the cake, and although the washings show no reaction, a thin layer of adsorbed solute is nevertheless present, which may be discovered by testing the cake itself. As the latter is dried, the concentration of the adsorbed solute in-

creases and may attain quite objectionable proportions even when washing has been carried so far that the last washings show no reaction. The case is aggravated when the particles of cake are themselves porous, *e.g.*, materials produced by fusion with alkali or the like. In these cases the wash water may quite rapidly displace the mother liquor between the particles, but the liquor in the pores of the particles can escape only by diffusion, which is necessarily slow. When these conditions prevail, even the cake may appear neutral after prolonged washing, but shows marked alkaline reaction again after standing for some time, during which diffusion out of the particles into the water between them occurs. It is therefore quite useless to hasten washing, and the sound procedure is to make it so slow that it proceeds *pari passu* with diffusion. This, again, is a state of things which cannot be altered by the design of the apparatus or by increasing the working pressure, but must be accepted as following inevitably from the nature of the material treated.

Both during filtration and during washing it is necessary to maintain a difference of pressure between the two sides of the septum. How great this is to be and how it is produced are technical and economical problems. The function connecting the rate of flow through unit area of a given material with the difference of pressure is not known, and it must obviously be complicated in the technical case, *i.e.*, a layer of increasing thickness building up on a porous surface which remains constant. Even for a porous material alone, or for such a material covered by a definite layer of particles, the function is certainly not linear, or, in other words, Poiseuille's law does not hold. It has been quite frequently assumed that it does, but even *a priori* there appears to be no reason why it should, since the pores in the filtering materials used technically are quite certainly not cylindrical. In the few cases investigated by the author³ the curve plotted with rates of flow as ordinates against pressures as abscissæ shows an inflexion and then approaches a parabolic type.

As the pressure is applied to or through the liquid in which the particles are suspended, the latter are in no sense pressed together, but are simply left behind as the corresponding quantity of liquid escapes through the septum. It therefore follows that the structure of the cake and its content of mother liquor are not affected by the pressure employed—a point on which the most common and serious misconceptions exist. It need hardly be said that this statement does not apply to centrifugal filters, in which the structure of the cake also depends almost entirely on the shape of the particles, but in which acceleration is imparted to the liquid, so that—in ideal cases—the whole of it may be removed.

If a difference in pressure of about 14 lb. per sq. in. is sufficient, it may, of course, be produced by maintaining a vacuum on one side of the septum, while the liquid to be filtered is exposed to atmospheric pressure on the other. The case does not differ theoretically from any other, but the technical differences between it and filtration under pressure (in the technical meaning) are so great as to justify separate treatment. The most striking feature of the vacuum filter, as this type is called, is the accessibility of the cake during and after its formation. Apart from the facility of removal, this is of advantage in permitting defects in the uniformity of the layer, which render washing imperfect, to be corrected. On the other hand, vacuum filters cannot be used for liquors having a high vapour pressure at the time of filtration (*i.e.* volatile or hot) unless a condenser is provided.

The accessibility of the cake makes possible the design of the various types of "continuous" filters, into which, as into other purely technical details,

it is not proposed to enter here. Another very interesting possibility remains so far unexplored, *viz.*, the combined use of filtration and decantation. Many of the more common precipitates settle fairly rapidly, either because the particles, although of low specific gravity, are large—*e.g.*, calcium sulphate or calcium carbonate as arragonite (*i.e.*, precipitated from hot solutions), or because the specific gravity is high, as with barium or lead compounds—over 6 in the case of the chromates or sulphate of the latter. To take a fair numerical example, we may assume a rate of settlement of 1 cm. per minute. If this suspension is now fed at one end of a long vacuum filter at such a rate that it will take, say, 1 min. to reach the opposite end, there will be a depth of 1 cm. of clear liquid from the surface, which can be continuously drawn off over a suitable weir, provided, of course, that the rate of feed is sufficient. Calculation can only be quite approximate, as liquid escapes already through the filtering medium on the way to the weir, and as, furthermore, the velocity of settlement is affected by the horizontal flow across the filter and the vertical flow towards the filtering surface. Experiment would be quite easy, and the method seems to deserve trial in many cases, especially where the solid matter is coarse or heavy and the liquid itself runs to waste, so that a very slight turbidity would be of no consequence.

It is obvious from the foregoing that the process of filtration is quite independent of the nature of the filtering material employed, and the point would hardly deserve mention were it not that misconceptions seem to exist even on this self-evident matter. The conditions the filtering material has to fulfil are three: It must have pores of the required size and as numerous as possible; it must be of sufficient strength mechanically to stand the pressure applied, suitable methods of support being assumed; finally, it must be chemically indifferent to the liquids to be filtered. As regards the first two requirements, textile materials have very considerable advantages, and especially the slight thickness sufficient to bear considerable tensile strains makes possible the design of apparatus, such as filter presses, possessing a large filtering surface in a small space. As regards the third one, they are less satisfactory, and the use particularly of woollen filter cloths (for acid liquors) is becoming economically impossible. These cases can be dealt with by the use of porous tiles or plates either consisting of silica or materials of high silica content, or of carbon. The latter material is ideal, as it resists both acids and alkalis; it is, of course, understood that the material consists entirely of carbon and not merely of carbon particles agglomerated by a silicious binding agent. Such porous plates, made by carbonising at high temperature a mixture of ground coke and tar, have been made in Germany for a considerable time, but their manufacture has not so far been taken up in England or the United States. This material enabled the author to solve a problem which, if not important or of frequent occurrence, is certainly interesting, *viz.*, the filtration of hydrofluoric acid. A vacuum filter of antimonial lead, with carbon plates as filtering medium, was used, and it seems doubtful whether any other combination could have answered for the purpose.

A drawback of such porous plates is their thickness, which is called for more by reasons of manufacture and transport than by the conditions of use, as (apart from the difficulties of using suitable materials of construction) it is next to impossible to devise apparatus of the filter press type. The filtering surface to be obtained with a given volume and weight of apparatus is therefore necessarily smaller than in filter presses. In cases where a small amount only of solid material, which can be run to waste, has to be removed, ceramic materials offer an interesting possibility, *viz.*, the use of hollow cylinders,

i.e., "filtering candles" of large size. A large number of these can be assembled in a small space, the residue being removed by reverse flow and subsequent rinsing and flushing. The author has designed such filters for corrosive liquors having a filtering surface of about 105 sq. ft. in a volume of about 22 cub. ft., a ratio comparing favourably with that of filter presses (which would have been inapplicable). At present such porous cylinders are not manufactured in this country, but there appears to be no reason why they should not be.

A third type of filtering material remains to be mentioned, which so far has found application only in research and on the laboratory scale, but appears to be capable of much more extended use. This comprises the membranes generally described as "ultra-filters," which are capable of holding back particles of submicroscopic size. Those used in the laboratory are prepared by impregnating filter paper with collodion (*i.e.*, solutions of nitro-cellulose in ether-alcohol or in glacial acetic acid) and gelatinising the latter by immersion in water. This removes the solvent and leaves behind a gel of nitrocellulose, the permeability of which can be regulated within wide limits by choosing the concentration and controlling the removal of the solvent. There seems to be no reason why textiles should not be treated in the same way as paper, in which case the membranes could be employed in apparatus of the filter press type. They have to be protected from drying, but this is not difficult, and such filters could probably take the place of pulp and similar filters where very brilliant filtrates are required.

(1) E. Hatschek, "The direct separation of emulsions by filtration and ultra-filtration," *J.*, 1910, 125; E. Hatschek, "The mechanism of filtration," *J.*, 1908, 538; (2) *loc. cit.* Fig. 27; (3) *loc. cit.* Figs. 13 and 14.

THE FILTRATION OF COLLOIDS.

BY W. R. ORMANDY, D.SC.

The ordinary methods employed for the separation of solids from the liquids in which they are suspended are:—1. Subsidence and decantation. 2. Ordinary filtration. 3. The use of centrifugals. 4. The use of the filter press. Whatever method is employed, efforts are always being made in the direction of producing a precipitate which is as dense and as crystalline as possible. Unfortunately some precipitates cannot be converted by any ordinary treatment into a form which render them amenable to easy treatment by any of the methods referred to above. Precipitates such as hydrated oxide of iron and similar substances of a slimy character are often very difficultly amenable to any treatment. The precipitates may be formed in hot solutions, they may be allowed to stand, or boiled; but in many cases such treatment does not lead to a suspension which can be readily handled.

The special method of treatment with which we have to deal now depends on the utilisation of certain properties of finely divided particles in suspension. This new process for the application of physico-chemical laws is in many respects so new that it is necessary to deal shortly with the underlying principles before the methods of their application, and the limitations of the process can be properly understood.

Colloids can be divided into two groups, the reversible and the irreversible. To the reversible group belong such substances as glue, gelatin, which can be dissolved in water, dried, and again dissolved in water without any great alteration in properties. To the irreversible group belong such substances as albumin, which after treatment with boiling water become absolutely insoluble. For the most part, however, the nature of the colloid is largely dependent upon the size of the particles.

Sufficiently fine particles when suspended in water exhibit what is known as the Brownian movement, which is more energetic the smaller the particle. This Brownian movement is now regarded as the visible symbol of molecular bombardment, as has been largely proved by the beautiful researches of Professor Perrin. Generally speaking particles of matter exceeding 0.0001 mm. in diameter will eventually settle from solution. It happens that this is about the limit of microscopic visibility. The fact that particles below this limiting size do not settle means that the molecular and electrical forces outweigh the action of gravitation. So far back as 1869 Jevons noticed that the intensity of the Brownian movement of clay particles was increased by the addition of traces of certain types of substances and inhibited by the addition of other types of substances. In 1895 Bliss noticed that a clay suspension exhibited increased Brownian movement with the addition of traces of alkali, but that the addition of further quantities again reduced the movement.

It has long been known that the addition of certain types of chemicals tended to cause coagulation of colloids suspended in water; thus the addition of calcium hydrate and aluminium sulphate to bring about the settlement of sewage sludge is ancient history. Weber many years ago showed that the addition of considerable quantities of sodium carbonate and sodium silicate to mixtures employed in the making of pottery ware caused such mixtures to be thinner. This principle was utilised by him to enable "slips" to be prepared containing much more clay substance, flint, Cornish stone, ball clay, and other ingredients employed in pottery making to the gallon, and such "slips" are employed in the casting of pottery-ware in plaster moulds.

These are all illustrations to show to some extent the position of knowledge relating to the effect of electrolytes on colloidal suspensions. It remained for Count Schwerin to see the bearing of these little-studied laws in their commercial application. A very brief history of his work will be useful in showing you how a few accidental observations scientifically followed up have led to developments of the greatest technical importance. Count Schwerin at the University was working on the problem of removing the musty products of decomposition from albuminous materials, and in the course of some electrical experiments noticed that his colloidal products collected on the poles which he was employing in his experiments.

Being greatly interested in the problem of drying peat he tried similar experiments on a peat suspension, and found that the peat actually did travel to the anode and was deposited thereon. His efforts at that time were directed rather to the utilisation of electro-endosmosis to the de-watering of peat, and to justify this end the peat magma was pressed between perforated plates which were respectively anode and cathode. On cutting through the dried peat mass so obtained he noticed that, although the peat suspension originally employed had been thoroughly mixed, nevertheless the section of the dried peat showed layers parallel to the anode, and he gathered from this experiment that under the influence of the current the various bodies in the suspension travelled with different velocities. Finding that one of the layers consisted largely of clay substance, the experiments were tried with a clay suspension, and it was found possible to deposit clay out of a water suspension on the anode. Starting from this basis the "Osmose machine" was gradually developed (this machine will be described later), but it was found that while one clay would deposit, another apparently similar clay did not react. Much experimental work led to the discovery that

certain clays shaken with water have particles which apparently carry no charge. The next development was to make use of the fact that most colloids can and do adsorb considerable quantities of electrolytes to which they may be exposed, and it was found that by the addition of very small traces of suitable electrolytes such as caustic soda, and the like, the previously inert clays were rendered amenable to electrical action. Even now the difficulties were not overcome, for it was found that a few clays were still impossible of treatment. In many cases this proved to be due to the presence of traces of divalent salts which were soluble in water, and, as has already been pointed out, these divalent and trivalent compounds have an extreme power of neutralising the so-called peptising action of the univalent alkalis, bringing about the coagulation of the clay subsidence. Such cases can only be met by previous chemical treatment directed to the conversion of the coagulating electrolytes into insoluble salts.

It is by the utilisation of selective adsorption and by choosing diaphragms of suitable polarity that it has been possible to separate from blood serum the fibrinogen, the euglobulin, and other substances, leaving a pure paraglobulin behind. The practical application of this discovery becomes obvious when we learn that in the preparation of anti-diphtheritic serum from horse serum it is possible to separate an anti-diphtheritic product having ten times the strength of that usually prepared directly from the serum. This achievement is based not only on the application of the principles above referred to, but on the discovery that the anti-toxin in blood serum is practically entirely combined with the paraglobulin, and that by isolating the paraglobulin from the anti-diphtheritic horse serum the concentration for medical purposes is increased manyfold. Obviously the removal of large groups of little-known and complex nitrogenous substances from the serum leaves a product which is not only medically more active, but carries with it a greatly diminished quantity of not only inert, but possibly harmful products. So much then for the general laws upon which the Osmose process is based.

In what follows reference will be made to the behaviour of a clay suspension, because clay is in many respects a typical colloid suspensoid, and the methods of dealing with clay have been further developed than any others. It is found that if one part of plastic clay be mixed with one part of water, a creamy mass is produced. In the case of a very plastic clay, i.e., generally one having very finely divided particles, the mixture will be almost as thick as butter, whereas with a meagre clay not very plastic, such as ordinary china clay, the mixture may be only a thin cream. If to these mixtures we add a small amount of alkaline electrolyte, say 0.03% on the weight of the clay, a wonderful alteration is brought about in the physical properties of the mixture. In both cases the buttery or creamy mass will become very thin and fluid. The suspended colloid has been converted from the gel condition into the sol condition, and now all the particles are electrically charged, and to a very large extent have become dispersed and are in a much more finely divided state than was previously the case. Such a dispersed suspension is exceedingly difficult to filter by ordinary means, but use can be made of the fact that the clay particles are charged and can be deposited on an electrode of suitable polarity.

The anode of the machine used is a rotating drum made of an alloy to resist the corrosive action, whereas the cathode consists of copper strips placed round the anode, and distanced about $\frac{1}{4}$ in. therefrom. The bottom of the containing vessel contains paddles to keep the clay in suspension. The clay suspension consists of clay particles having

a comparatively strong negative charge and other substances having a very feeble negative or no charge at all.

In the electric field between the cathode and the anode the clay particles are strongly attracted to the anode. The moment a film of clay has been formed on the anode the water contained therein is violently ejected towards the cathode, and this stream of water tends to sweep away all but the particles having a high negative charge, which, in spite of the water current, are drawn to the anode. The lantern slides show a sample of raw clay, then the impurities which settle out of the clay after the addition of the electrolytes, then the impurities which are rejected by the Osmose machine, and finally the clay as taken up on the anode.

Although such a process cannot be considered as an ordinary filtration process, nevertheless it is, *inter alia*, a process for the continuous separation of suspended matter from suspending liquid with simultaneous partial drying of the solid product. Obviously such a continuous process can only be employed where large quantities of material have to be handled, but the same principle can be embodied in the Osmose filter press, which consists of an ordinary type of press made of insulating material, the filter cloths being placed upon insulated pieces of conducting material, such as wire gauze or the like. Into such a chamber the clay suspension can be passed under just sufficient pressure to ensure that the chamber keeps filled. Without the application of electricity under these conditions there will be no filtration, for the filter cloths would be choked from the first moment. By the application of a potential of 60 volts, however, the water is forced out through the cathode, and a firm, hard cake results very rapidly, and even in the case of plastic ball clay a 3-in. block can be made in an hour containing less than 30% of water. It will be impossible to produce a 3-in. block of ball clay in an ordinary filter press with 200 lb. per sq. in. in days, let alone hours.

The essential difference between the Osmose machine and the Osmose filter press lies not only in the continuous action of the Osmose machine, but in the fact that the machine brings about a certain selective purification or separation, whereas the filter press by its nature merely serves to collect the whole of the suspended product.

THE "STURGEON" SELF-DISCHARGING CENTRIFUGES FOR SEPARATING SOLIDS FROM LIQUIDS.

BY R. A. STURGEON.

Careful consideration of the various attempts which have been made to construct a self-discharging centrifuge led to the following basis for design being laid down:—

(1) The surface liquor and the lightest portion of the solids must be removed before the main deposit is discharged.

(2) There must be no attempt to overcome, by direct means, the centrifugal force of the deposit. The only forces to be overcome must be frictional resistances, while all the main forces must be balanced in some way.

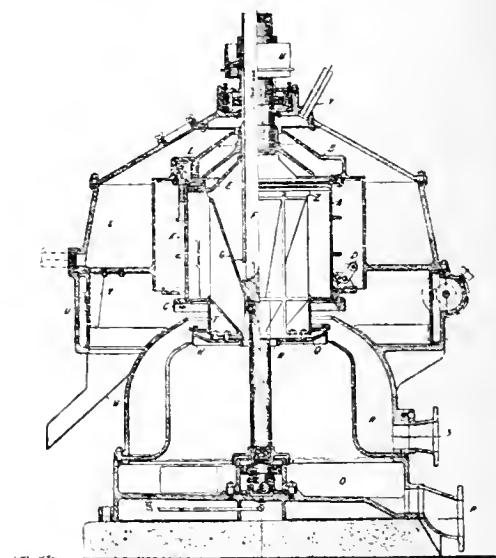
(3) The power for effecting the discharge must be obtained through the main drive to the centrifuge, and must not be dependent upon accessory machinery.

(4) The effective volume of the separating chamber must be the maximum obtainable for a given diameter of machine.

Several attempts were made to design a machine to meet these conditions, but the results were not very satisfactory until the use of the pressure produced in a liquid by centrifugal force was introduced to operate the discharging mechanism.

"Sturgeon" Centrifuge for Combined De-watering of Solids and Clarification of Liquors.

The type of separator designed along these lines for combined de-watering of solids and clarification of liquors is shown in the accompanying figure,



which consists of two half-sections—one through the valves in the top cover, and the other through the levers which hold the bottom cover up to its seating. The cylinder, A, has a fixed cover, B, at the top, and a movable cover, C, at the bottom. The movable cover is held on its seat by weights under centrifugal force acting through the bell-crank levers, D, which allow of a limited lift. The piston, E, which slides up and down the shaft, F, has a large hollow boss, G, through which the liquid under treatment may pass from the apertures in the hollow shaft. To the bottom portion of the boss is attached a disc or plate, H, which forms a joint with the inner edge of the movable bottom cover. Attached to the bottom cover are valve rods, K. When the piston reaches its bottom position it lifts the movable cover off its seat, thereby causing the valve rods to open the valves, L. The shaft is rotated by means of the pulley, M, and, of course, the cylinder and all parts connected thereto revolve with it.

The machine is started and the liquid to be treated enters the machine through the hollow shaft, F. It passes through the apertures into the hollow boss of the piston and thence into the cylinder. The solids are deposited on the walls of the cylinder as the liquid passes through, and the clarified liquid is discharged over the concentric weir, N, and into the chamber, O, whence it is carried away through the pipes, P. When it is time to discharge the deposited solids the supply of liquid is cut off, and water is admitted to the top side of the piston through the pipe, Y.

As soon as the piston commences to descend the joint near Q is broken, and the surplus liquid flows away into the trough, R, and is carried away by the pipe, S. As the piston descends the liquid continually overflows the weir, Q, until only solids are left. These are further squeezed, and much of their accompanying liquid is removed, flowing over the weir, Q. This liquid is necessarily very dirty, and is kept separate from the main effluent, which would otherwise be contaminated. Finally, the pressure under the piston becomes so great as to overcome the pull of the levers, D, and to lift the

cover, c. The solids are discharged on the revolving circular plate, v, whence they are scraped off by the knife, w, and fall down the chute, w. As the cover, c, lifts the valves, t, open, and the water above the piston is discharged into the trough, x. This discharge may be made tangentially in the opposite direction to that of rotation, so that the pressure which has been imparted to the water by the machine may to a great extent be restored during the discharge. Thus the horse power required to effect the discharge of the solids is reduced to a minimum.

As soon as the pressure of the liquid falls sufficiently, the cover, c, closes, and when all the water has been discharged the machine is ready for a fresh charge of the liquid under treatment. This forces the piston up to its top position, closing the valves, t, and coming to rest on the seating, the upward pressure being sufficient to maintain the joint. The machine then fills up, and the operation proceeds as before.

In this type of machine the effluent is discharged at the maximum distance from the deposit, and the main effluent, the liquor squeezed out of the solids during discharge, the solids, and the water used for discharging are all passed into separate compartments.

A simpler type of machine was also described to deal with cases in which clarification of liquor is the essential factor, the amount of moisture in the solids being of small importance; also another type of centrifuge for clarification and grease recovery.

THE SHARPLES "SUPER-CENTRIFUGE."

BY S. H. MENZIES.

The Sharples "super-centrifuge" applies the settling action of gravity highly intensified in two classes of operation, viz., that in which solids are settled out of liquids, which may be termed "clarification," and that in which one liquid heavier or of higher specific gravity is settled apart from another liquid of lower specific gravity. For the purpose of the paper this latter operation will be termed "separation."

With the Sharples "super-centrifuge," the action which takes place in the case of "clarification" of a liquid from suspended solids is a "subsidence" pure and simple and not in any sense a "filtration."

Since centrifugal force varies directly with increase of diameter, and as the square of the speed of revolution, the centrifuge in question has been constructed of tubular pattern.

The rotating member, or "bowl," is both supported and driven from the top, being otherwise entirely free to find its own axis. By these means the machine is operated with the minimum of wear and tear. One main ball-bearing constitutes the sole point in the machine at which friction has to be taken into account. The drive, arranged immediately above this main bearing, takes four alternative forms, viz.:—

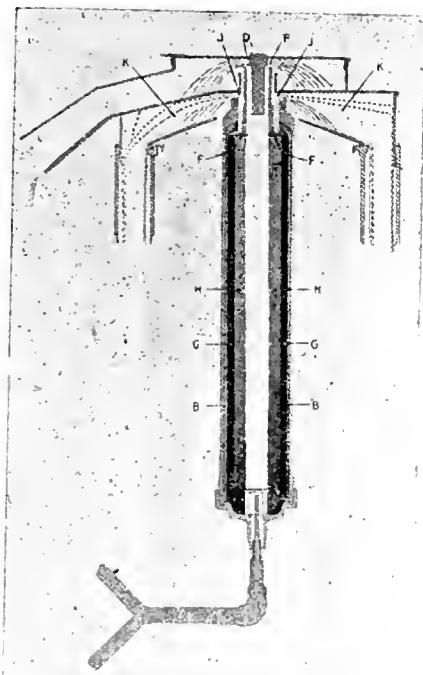
- (1) A pure belt drive from line shaft.
- (2) Direct steam drive by means of a turbine wheel fixed to the tube or bowl spindle.
- (3) Indirect electric drive, i.e., individual motor mounted on the centrifuge body and driven by short belt and idler pulley on to the head of the bowl spindle.
- (4) Direct electric drive in which the motor armature is mounted on and an integral part of the bowl spindle. Mechanical friction losses are thus limited solely to those of the one ball-bearing

which carries the rotating bowl. A considerable reduction in consumption of power is effected in this instance.

At the bottom of the bowl a short extension runs with a definite permitted clearance in a bushed socket, serving as a guide during the running up to speed of the bowl, when slight vibration or oscillation may occur. The liquid to be treated is admitted through the hollow centre of this extension of the bowl either under greater or less head, or in certain cases positive pressure, according as viscosity or other considerations demand. Settlement takes place during the upward travel of the liquid, which discharges at the top of the machine through one or two outlets, according as the process in hand is that of "clarification" or "separation." In the former case the suspended solids normally are deposited on the inside wall of the tube or bowl, and these solids are intermittently removed from the bowl as and when it becomes fully charged.

In certain industries a large diameter and slow-running centrifuge may be an economic primary stage to the final intensive treatment in the "super-centrifuge." By this means complete clarity would be secured in liquors heavily charged with solids which could not otherwise be economically handled in the "super-centrifuge." As an alternative to this liquors high in suspended solid content are given a preliminary gravity sedimentation before centrifuging.

Two typical processes in connexion with "clarification" which might be cited are those of dry cleaners' benzine recovery and the clarifying of lacquer or nitrated cellulose. In the former we have an instance of a heavy suspended material in an extremely light liquid, whilst in the latter very light solid material is suspended in a heavy and highly viscous liquid.



The process of centrifugal clarification with continuous discharge of solids.

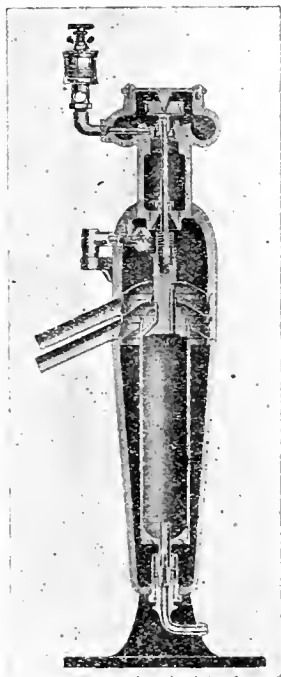
In both cases in the "super-centrifuge" clarification is complete and the suspended solids entirely eliminated. These two cases, however, afford good

examples of the manner in which physical characteristics affect capacity of the machine. In the case of benzine clarification the flow through a Sharples "super-centrifuge" can be taken comfortably to 250 galls. per hr., whilst in the case of the nitrated cellulose, where the process is one of eliminating fine vegetable fibres of cotton or pieces of cotton-husk, the flow through a Sharples machine to secure the best grade of lacquer entirely free of all solid suspension usually does not exceed 70 to 80 galls. per hr.

In one factory the introduction of a Sharples "super-centrifuge" completely revolutionised the process of manufacturing this nitrated cellulose. Prior to its installation the firm in question found it essential, in order to maintain the high character of their output, to purchase only the most expensive grade of cotton wool, whilst after the introduction of the clarifying process by means of the "super-centrifuge" it was unnecessary to pay any particular regard to the grade of cotton which was employed, since any impurities remaining after nitration were easily and completely removed, and the final product still maintained at its high excellence. A reduction in the contributory cost of this raw material by over 50% was thus effected.

In the "super-centrifuge" treatment of varnish one of two objects may be accomplished. Where ageing is not carried out to any great extent the complete removal of all suspension which would mar the coat or settle in the can is effected, and a brilliant product results. In the case of varnish which has to be aged, treatment by the intensive "super-centrifugal" force will secure a firm, thin layer of sediment not easily disturbed on decantation, as against the bulky sludge, which proves troublesome by reason of its ready disturbance during decantation.

Where "separation," as defined earlier in the paper, is alone in question two characteristics are essential in the constituent liquors, viz., immiscibility and difference of specific gravity.



The Sharples super centrifuge, laboratory type, turbine driven.

Specific gravity, viscosity and the globule character (in the case of fine emulsions), besides

other factors, all enter into the consideration of the type and design of bowl.

In a number of cases contributory processes to the central function of the "super-centrifuge" have been worked out by careful test and research, a prominent example being the separation and continuous discharge of amorphous wax from cylinder stock. This result, previously impossible in a centrifuge, has been achieved by carrying out the entire process at a very low temperature and employing a carrier liquid immiscible with the oil and of greater specific gravity than the heavy wax, upon which the wax is floated off in a continuous discharge. The diagram of action in the super-centrifuge shows very clearly what happens in the bowl.

The laboratory machine (see fig.) is a still further development of the sacrifice of diameter to revolutions, and emphasises the resultant gain mentioned at the outset, since in the commercial or full-sized Sharples super-centrifuge a separating force is developed at 17,000 r.p.m. of 16,950 times that of gravity, whilst in the laboratory machine a force over 40,000 times that of gravity is developed, and a means for effecting hitherto impossible separations, as also for facilitating qualitative and quantitative physical analyses, is afforded.

The opportunities presented by the machine include such processes as the separation of finely-divided wax from an alcoholic solution of shellac; the separation of bacteria from serums, for which the machine found a wide use during the recent war; separation of pigment from paint and enamel, and quantitative analysis of these.

Many complete processes have been worked out in which the super-centrifuge itself constitutes but one feature; amongst these may be mentioned the recovery of neutral wool fat from scouring effluent; the separation of vegetable oils from soap stock; the dehydration of water-gas tar; clarification of glue and of nitrocellulose liquors, and the separation of wax from mineral oil as before mentioned.

As an illustration of the action of the super-centrifugal force upon serum, a sample submitted for treatment from which all the corpuscles had been previously filtered off, and which appeared absolutely free of suspended matter clogged a Berkefeld filter after passing 200 c.c. Treatment through the super-centrifuge eliminated the bacteria thus evidenced to the extent that 20 litres did not clog the same Berkefeld filter.

DESIGN OF MECHANICAL FILTERS.

BY BALFOUR BRAMWELL.

The principal difference in filtration with the mechanical filter and the slow sand-bed is in the speed of filtering. In the mechanical filter this speed is approximately 40 times that of the slow sand bed, consequently the mechanical filters are often termed rapid filters to distinguish them from slow sand beds.

In the slow sand bed the straining action is confined almost entirely to a thin top layer of the filtering material, whereas in the rapid filter the whole depth of bed is made use of before the filter is thrown out of action for the cleaning process.

Slow sand beds generally work for months without scraping, but the rapid mechanical filters are usually designed so that the cleansing process is carried out in the case of waterworks once per 24 hours, or sometimes once every 12 hours, and in the case of filters for industrial purposes once per working day, or in some cases once every 6 hours.

The usual speed of filtration for slow sand beds is approximately $2\frac{1}{2}$ galls. per sq. ft. of superficial area, whereas the most economical and satisfactory speed for design of mechanical rapid filters is 100 galls. per sq. ft. when dealing with the

purification of ordinary average waters met with. With a better class of water this speed can be increased 20%, and it is not uncommon, but cannot be recommended, to have filters that are being cleansed every 6 hours working at a speed of 140 galls. per sq. ft. When the water to be purified is very good, and requires very little chemical treatment, the speed of filtration can be increased two or three times, but it is rare that such waters are met with except under exceptional circumstances.

Slow sand beds are much more sensitive to choking than rapid filters, since the straining action in the former is confined to a very thin layer of filtering material. With rapid filters agitation of the surface of the filter bed is often advantageous. In order to obtain the best results from rapid filters chemical and coagulant treatment must be used. Provided sufficient time is given for storage and for the chemical action, precipitation, and sedimentation to take place, there is no need for filtration.

In our days, however, it is a mistake to build huge reservoirs for the purpose of storing water to obtain purification when this purification can be achieved by quite a short storage and rapid filtration, for instead of months of time for the process only hours are required with subsequent rapid filtration. It is entirely wrong, however, to depend too much for purification on chemical treatment. Want of filtration or inefficient filtration often means overdosing with a germicide to obtain the necessary result.

The cleansing of the filter bed is a very important operation. At every periodic cleansing of a mechanical filter the whole bed should be disturbed and uniformly agitated and cleansed from top to bottom. In all mechanical filters, with one exception, this is effected by a voluminous reverse flow of water, which lifts and eases the bed before the agitating devices operate. The exception is the filter working on the "turn-over" principle, in which the whole chamber is made to revolve slowly.

In all the other filters the reverse flow has to be concentrated by means of restricted orifices, and without such reverse flow under a considerable pressure cleansing cannot be efficiently effected. In the case of air or steam injectors it has been found that a reverse flow of air or steam through the bed is useless without a voluminous supply of water.

In the "turn-over" filter, by revolving the whole of the filter chamber to upset the whole filtering material only a comparatively slight reverse flow of water is necessary to wash the impurities to the surface of the bed. The following are features affecting the efficiency of cleansing, for it cannot be disputed that the method adopted for the cleansing is really the crucial feature in the mechanical filter. With the "turn-over" principle there are no nozzles in the bottom of the filter. The bed rests merely on a double thickness of perforated plates with holes superposing, having sandwiched in between them a fine mesh of strong copper or bronze gauze. The area, therefore, through the bottom is nearly equal to the area through the perforated plates, and is very large. The gauze is well supported, and only exposed in the perforations, which are half an inch diameter. There is, therefore, no restriction to the passage of water, and practically no loss of head from this cause.

In rapid filters using chemical coagulant treatment the minimum depth of bed of one grade should not be less in the smallest sizes than 2 ft., and in large waterworks filters it should be from 2 ft. 6 in. to 3 ft.

The best filtering material is undoubtedly crushed quartz, because it is hard and sharp and less liable to trituration, and is practically non-absorbent. Natural sand gives the same filtering

efficiency, but it does not clean quite so well owing to its rounded particles, neither does it pack so well. It is absorbent, and most of it is composed of a soft, triturable material. Quartz is, however, very expensive and difficult to procure at the present time.

All material, whether mechanically crushed or natural, should be free from dust or very fine particles, and should, therefore, be riddled. The grade of filtering material should, of course, be made as uniform as it is possible to obtain, but in practice absolute uniformity cannot be expected; therefore, a system of cleansing which necessitates a reverse flow of water at such a high head and of so great a volume that it throws the finer particles of the filtering material to the surface is distinctly at a disadvantage.

The design of the "turn-over" filter was the outcome of trouble experienced in imperfect cleansing of the bed in Reeves' filters when dealing with a very bad water to be used for industrial purposes, especially water containing colloid matter, of which most rivers contain a certain proportion, and which is most difficult to deal with. Unless the bed is disturbed in every portion at each periodic cleansing the tendency is for the portions imperfectly cleansed to become gummed up in such a manner that the means provided for agitation, in order to loosen and wash out the impurities, are insufficient. Rakes could not reach all the parts of the filter bed. The result of this experience was that the "turn-over" principle was thought of, and the very first filter installed is still successfully at work to-day.

The sides of the chamber in the "turn-over" filter are curved or spherical, and have the same beneficial packing effect that cone sides have. The life of the copper gauze on which the filtering material rests averages five or six years. It costs only a few pounds to replace, and the work of replacing can be executed very quickly by turning the filter on its side, fixing one half in at a time without removing the filtering material. The bottom and gauze can also be washed with a hose at any time and inspected without removing the filtering material.

There is practically no loss of filtering material in the cleansing of the "turn-over" filter, because, unlike other filters in which the wash-water is being discharged at the same time as it is being allowed to flow into the filter chamber, the filter is specially arranged so that it is impossible to inject and eject the wash-water from the chamber at the same time. By this arrangement it is possible to place the discharge opening for the dirty wash-water close down to the surface of the bed, and, therefore, get rid of the dirt more thoroughly and rapidly by siphoning action.

Mr. E. A. ALLIOTT read a long paper on "Plate and Plate Frame Filter Presses," which will be printed in a subsequent issue of the Journal. An abstract of Mr. W. J. Gee's paper, "A New Process of Centrifugal Filtration," will also appear later.

ANNUAL DINNER.

The annual dinner of the Society was held in the King's Hall, Armstrong College, Newcastle, on Wednesday evening. Professor Henry Louis presiding.

After the usual loyal toast had been honoured, Mr. FRANCIS PRIESTMAN, Chairman of the Ashington Coal Company, Ltd., proposed "The Society of Chemical Industry." As a commercial man he was glad to say that he recognised what a great deal

depended upon such a Society as theirs. The coal trade was indebted to the chemist for a great deal in the way of fuel saving. He recalled how at his by-product works the German chemists had been replaced by British at the earliest possible moment, and the results had been equal to, and often better, than those obtained when Germans had been employed. The Society had since its inception done most valuable work both in disseminating knowledge and in bringing together a large number of scientific people; such gatherings were bound to be of benefit to the nation. He concluded by hoping that the good work of the Society would continue, and that the membership would increase and the Society generally become very prosperous. He coupled the names of the President and Prof. Louis with the toast.

PROF. LOUIS, replying to the toast, thanked Mr. Priestman on behalf of the Society, the President, and himself. Most of them were aware of the reason why he was presiding that evening. It was directly due to the effects of war stress on their President. Owing to his strenuous exertions on behalf of the nation Mr. John Gray was not able to be with them that night. His name was not widely known. He was one of those who had worked quietly, steadily, and unostentatiously, but without him and men like him the country would have been at a sorry pass to-day. Mr. Priestman had indicated that at last the British chemist had come into his own. If there were such a thing as a bright spot in the war it was that their industry and chemists had at last received national recognition. The academic chemist had been brought to apply his knowledge to factory work, with what great results they knew. They had succeeded in bridging over the gulf between abysmal ignorance on the one side and esoteric erudition on the other. Still, scientists and teachers of science did not appear to have got that recognition from the leaders of the nation that they were entitled to; they had received less than any other class from the State. He could wish that the Government was more fully alive to the importance of sustaining the scientific life of the community. It was obvious that so long as the purse strings of the nation were held by the politicians there would be little improvement. The politician and the scientist could never have very much in common. Their points of view were entirely different. After all, the principal task of the scientist was the search for truth. He did not think anyone would accuse politicians of wasting too much time in that quest, and it was natural to suppose that politicians did not see much use in the men who followed the practice of not only looking for facts but even publishing them! Their Society was in a flourishing condition, and its membership was increasing steadily; with its 5700 members it was one of the leading technical societies in the country. Obviously, the success of the Society was bound up in the success of the chemical industry. When he had addressed them a year ago, in the first flush of victory, he had looked forward to a time of peace which would enable them to repair the ravages of war. He was afraid those hopes had been disappointed. They were still looking for a true peace which would enable them to put aside all thought of discord and get on with their various tasks. During the war, in one respect, the task had been simple—they had had one common object in view, and the nation as a whole had worked together. It seemed that the common bond had now loosened, and men were pulling in different directions. Instead of patriotism they saw the discord of selfishness. Such a state of affairs was bound to react unfavourably on all industries. The time of false prosperity would cease, and the chemical industry was bound to feel the effects of the position more than any other. The chemical industry was a key industry in only a limited sense.

It certainly held the key that unlocked the door to failure, but it was by no means certain that it held the key that opened the door to success. The road to prosperity in this country lay through the heavy key industries—coal and iron—but the road to disaster might come through the neglect of the chemical industry. Dark as the immediate future might be, and he believed there were dark days before them, he did not despair of the future. He had always a belief in the sound common-sense of the average British citizen. When they did get back to habits of steady work he believed they would find that the chemical industry had played its full part with the other industries in re-establishing prosperity, and the prosperity of the chemical industry meant also that of their own Society.

DR. E. F. ARMSTRONG proposed the toast "Armstrong College," coupling with it the name of Sir Theodore Morison, K.C.M.G., K.C.S.I., D.C.L. Armstrong College was the centre of learning in that great city, the centre of thought; a centre whose influence was perhaps unseen, but it certainly pervaded the whole county of Northumberland. Referring to the growing recognition of the chemist, he said it behoved them to rebel against the label of "stinks" which had clung to the science for so long. He put forward a plea for associating the science with colour. In the attempt to create a great dye industry he thought the association of the industry with colour was very appropriate. In proposing the toast of that College he would like them to remember the great men it would assuredly turn out—men who would take up their work quietly and without seeking public advertisement. He would like them to cultivate the virtues of efficiency and tolerance and to adopt as their watchword the lines—

"To set the cause above renown;
To love the game beyond the prize."

SIR THEODORE MORISON, replying to the toast, said that Armstrong College had been connected with the Society of Chemical Industry for many years. It was the third time they had visited Newcastle, and he was glad to see that each time they came they had been associated with the College. The local section of the Society had also worked in intimate association with the College, to the great benefit of the College. The visit of such a Society to the College made them realise the enormous opportunities of work which were opening out to modern provincial universities; it was those visits of practical men engaged in practical work which took away from the provincial college that reproach of esoteric learning. He felt that it was by the connexion of the learning of the universities with the practical work of the world that the distinctive characteristics of the new universities would be founded. He thought they would always have a special advantage that the older universities could not have, and that was in living in close association with practical men engaged in industry. The occasions and opportunities for work were not only national, but international. He advocated the interchange of students between French and English colleges, believing that a real *Entente* was only possible by individuals of both countries getting to know each other better. That interchange of pupils was a very definite work for the universities.

COL. W. C. BLACKETT proposed the toast, "The City of Newcastle," in a humorous speech. As a Durham man he was glad to pay homage to Newcastle as a centre of learning. What training he had got for his profession as a mining engineer had largely been obtained from the professors and the technical societies of Newcastle.

THE LORD MAYOR OF NEWCASTLE (Councillor Walter Lee, J.P.), in reply to the toast, said that to welcome such a Society as theirs had given him exceptional pleasure. He realised, though not a chemist, how important the chemical industry was to such an area as the counties of Northumberland and Durham. He had learned something of the great achievements of the industrial chemist in the realms of by-product coking, and he was beginning to realise how greatly his district had benefited from the efforts of scientific men.

SIR WM. J. POPE (President-elect), proposing the toast of "Kindred Societies," coupled with the Société de Chimie Industrielle and M. Paul Kestner, said that the toast introduced a large subject because the Society of Chemical Industry claimed kinship with all associations which had for their object the advancement of science and technology. He would therefore confine his remarks to the youngest of the sister-societies. The French Society of Chemical Industry was not yet two years old, but, during that brief period, it had given evidence of remarkable vivacity, and had already taken a high position among the scientific societies of the world. The French Society had, of course, to some extent modelled its procedure upon that of our own; but, at the same time, it had introduced certain novelties of method which were worthy of their most serious consideration. The monthly Journal of the French Society of Chemical Industry was a literary production, and its contents exhibited all that precision and all that genius for expression so characteristic of our neighbours across the Channel; each number contained at least one article which dealt comprehensively with some great chemical question of industrial or economic importance. In spite of the recognised value of our English journal we could not but admit that we had much to learn in the art of presentation of important matter from the official organ of our French colleagues. One other point was perhaps of importance. During numerous recent visits to France he had occasion to observe that the Journal of the French Society of Chemical Industry was on sale at the railway bookstalls; that, together with the very large amount of lucrative advertisement matter contained in the French journal, should attract the earnest attention of those who devoted themselves, so much to our advantage, to the finance of our Society. No mention of the French Society of Chemical Industry could be dissociated from the name of the man who had founded that Society, whose initiative and genius had directed its early policy, whose organising ability had enabled him to gather together a band of collaborators who had made the new French Journal one of the ornaments of modern scientific literature, and who had been, in fact, the only possible first President of the young Society. Monsieur Paul Kestner was known to all as one of those great chemical technologists who had arisen from time to time, each of whom had, in his own unique fashion, assisted in establishing the chemical industry in its present commanding position; his inventions, and the driving force with which he had vitalised those inventions and made them powerful agents in the amelioration of many of the conditions of life, had made the name of the first President of the French Society of Chemical Industry a household word in the scientific community of every civilised country. He might speak at length on this theme, but actions spoke louder than words, and our Society, in conferring its medal on M. Kestner, had proclaimed more forcibly than he could ever hope to do the position which this great Frenchman had gained for himself as a pioneer in one of the most rapidly expanding branches of productive activity. In drinking to the success of the French Society of Chemical Industry and to the health of its first President, he should carry every-

one present with him in expressing an honest desire for the establishment of a closer relationship between their two societies; each of our nations had much to gain from a more intimate alliance of this kind. The casual observer might possibly at first sight think it remarkable that so much existed in common between the most exclusive of the Anglo-Saxon races, ourselves, and the French nation, which represented the most exquisite and the most artistically-minded product of the old Latin civilisation. He was sure, however, that if M. Kestner did him the honour of replying to these few words, they would carry away the conviction that his ideals in life were identical with our own. The events of the last six years had proved to demonstration that France and Great Britain were one and indivisible in their conception of the aims, the destinies, and the honour of the human race. He desired to propose the toast of "Kindred Societies," coupling the toast with the name of M. Paul Kestner, President of the French Society of Chemical Industry.

M. KESTNER replied as follows:—"I have the pleasant task to reply in the name of Kindred Societies. It is the youngest of these Societies I am representing here; it is also, I feel sure, the one which is nearest to your hearts. Established during the war, when the French and English were brothers-in-arms on the battlefield, it has been created in a pure spirit of brotherhood. But the sentiments which link us to your Society are at the same time filial. We always consider our Society as a daughter of yours. We wish it to be a daughter of which you will always be proud, and this will be our powerful stimulus for the future. This meeting comes on the morrow of the day when the International Union of Associations of Pure and Applied Chemistry has been definitely consecrated, in a grandiose frame in magnificent receptions, in Rome. The new Federation replaces the Union which existed before the war, and in which the different nations were represented only by their associations of pure chemistry. The fact that officially there is henceforward but one chemistry, that all associations of pure and applied chemistry are united in indissoluble links represents an event of incalculable importance. Certainly there has never been any need to plead the necessity of such fusion among us. Has not the Society of Chemical Industry in the past impartially chosen its presidents within the scientific glories and within the glories of the great English chemical industry? Is not its present President, Sir William Pope, illustrious scientist, succeeding a great technologist, John Gray? If there have been water-tight compartments between the associations of pure chemistry and of applied chemistry there has never been any partition between the two chemistries, as there exists only one. If there have been some misunderstandings sometimes our associations of applied chemistry have immediately set to work to dissipate them. We look upon our scientists as our chiefs, our masters; we venerate them, and our duty will always be to honour and help them under any circumstances. Gentlemen, during the journey I have just made in Italy in company with Sir William Pope, in witnessing this complete harmony between our Societies, this full and hearty agreement between the chemists of the nations represented, I could not fail to observe from the newspapers that there is in Italy an uneasiness—that public opinion expresses reproaches against the Allies. What is the cause of this uneasiness? In the great common enterprise of *entente* and of increasing intimacy between the Allied nations it seems that there are two distinct planes of thought which run parallel. The first plane is that of the diplomats, on whom devolves the burden to take decisions, the consequences of which are incalculable. The second plane is that of associations of scientists, thinkers,

economists, and manufacturers, who are so much interested in instituting firm relations between country and country. Since the Armistice the great international problems have been dealt with by the diplomats, nearly always without any knowledge of the public, although each one of us assumes a part of the responsibility of their decisions, and each of us has to bear the consequences. Yet the people of these nations as a whole and individually know nothing about these conflicts of interest, nothing of the solutions which are proposed or adopted, and those here are ignorant of the consequences these solutions may bring on the friendly relations of people to people and individual to individual. Less than ten years ago a French writer wrote about England, for the use of his fellow-countrymen, a book entitled "l'Île Inconnue." For many Frenchmen England is an unknown island, at least unknown in regard to that which is essential in the political, economical, social, and religious life of England. Without doubt the English have come to know France a little better since the war. But even to the English recently fighting in France there remains much to learn about our country, our ways, and our common aspirations. It is Societies such as ours, our Unions of Societies, that must establish from people to people throughout the civilized world collective and individual relationships which, if they are not able to prevent the mistakes that the diplomats sometimes commit, at least correct them and partially neutralise their consequences, through the fact that they establish permanent links of brotherhood. The Union of Associations of pure and applied chemistry in a single body has created to this end a remarkable and efficacious instrument. Let us hope that it will serve as a model to other Unions, which will be formed in other domains. The scientists, the thinkers, the economists and the manufacturers will in such Unions be united, and the result will be a deeper knowledge of each nation's qualities, and therefore a reciprocal esteem, which will dissipate misunderstanding. Sir William Pope, your interest in the Société de Chimie Industrielle, and the friendship with which you have honoured me, have transformed the present toast into one of the Société de Chimie Industrielle and of its President. In the name of that society, and on my own behalf, I thank you most cordially, and join with you in the earnest desire for the more and more intimate union of associations of chemistry, and particularly for the complete and indissoluble union of the French and English societies.

VISITS TO WORKS, EXCURSIONS, ETC.

On Wednesday afternoon and Thursday parties of members visited the works of the following:—Tharsis Copper and Sulphur Co., Palmers' Iron and Shipbuilding Co., Swan, Hunter and Wigham Richardson, Ashington Colliery, John Bowes and Partners, Newcastle Alloy Co., Newcastle Breweries, and Cookson and Co.

On Thursday evening a *Conversazione* was held at Armstrong College by invitation of the Newcastle Section.

Friday was devoted to a whole-day motor excursion up the Derwent Valley to Blanchland, thence to the Tyne Valley and Roman station at the Chesters, returning along the Roman Wall.

EXHIBITION.

During the week there was an interesting exhibition in Armstrong College of chemical and metallurgical products, apparatus, etc., mostly of local manufacture.

London Section.

Meeting held at Burlington House on May 3, 1920.

MR. JULIAN L. BAKER IN THE CHAIR.

THE THEORY OF GAS SCRUBBING TOWERS WITH INTERNAL PACKING.

BY F. G. DONNAN AND IRVINE MASSON.

The object of this paper is to obtain as simply as possible mathematical expressions which might serve as a preliminary basis for the experimental study of the factors which govern the design and operation of internally packed gas-scrubbing towers. It is hoped in later papers to present not only the results of this experimental investigation, but also a similar treatment of plate towers and other types of gas-washing plant.

In the relatively scanty literature on the theory of gas-scrubbing, the pioneering work of Dr. Ferdinand Hurter occupies a prominent position. In a series of papers communicated to this Society in the period 1885–1893¹ he stated an essential factor which governs the design of any apparatus wherein gas and liquid interact, and recorded many valuable experiments which afford fundamental data. The factor referred to is the area of gas-liquid interface offered per unit of volume of gas per unit of time; and on this basis he discussed the relative advantages of systems of bubbling, spraying, and tower packing. Thus Hurter's work is mainly concerned with the achievement of the maximum contact-time efficiency, irrespective of the solubilities and other specific properties of the gases and liquids which may be dealt with.²

Some time after the present paper had been written, we noticed that the theory of gas-absorption in packed towers had been dealt with by Partington and Parker,³ who applied it to the special case of the absorption of nitrous gases by dilute nitric acid. These authors took into account the fact that the dissolution of a gas in a liquid is not instantaneous, and they assumed that the rate of absorption is proportional to the concentration of the unabsorbed gas in the gas phase, which progressively diminishes from the bottom to the top of the tower. The equation deduced was:—

$$\text{Log}_e \frac{c_0}{c} = \frac{KkA}{V} x$$

where c_0 = initial concentration of the entering gas, c = its concentration at a height x in the tower, A = cross section of tower, V = rate of gas flow, k = area of packing surface exposed per unit of volume of tower, and K = a coefficient—not constant—depending on the rate of absorption of the gas by the liquid concerned, as found by experiment. The case is a special one, in that the change in absorption capacity and in rate of absorption of dilute nitric acid, due to nitrous gases which it has already absorbed, is slight and can be approximately allowed for. In other cases, however, this simplification is inadmissible, for the rate of solution of a gas in a liquid depends on the degree of unsaturation existing at the moment, as is well shown by the work of Adeney and Becker⁴ on the rate of solution of nitrogen and oxygen in water. The experimental results obtained by Adeney and Becker can be put into the following form. Let,

¹ J., 1885, 639; 1887, 707; 1889, 861; 1893, 227, 989.

² Some of the principles underlying the design of tower packing are set forth in an article published in "Chemical Engineering and The Works Chemist" (May, 1918).

³ J., 1919, 757.

⁴ Proc. Royal Dublin Soc., 1918-19, 15 (N.S.), 385, 609.

In order to integrate these equations, it is necessary to find the relation between m and n .

Since we may consider the level x as the bottom of a shorter tower, we have

$$V_g(m - M_2) = V_l(n - N_1) \quad \dots \quad (6)$$

and therefore

$$\begin{aligned} n &= \frac{V_g}{V_l} m - \left(\frac{V_g}{V_l} M_2 - N_1 \right) \quad \dots \quad (7) \\ &= f m - (f M_2 - N_1) \end{aligned}$$

substituting this value of n in equation (4), we have

$$\frac{dm}{dx} = \frac{k_1 k_2 S}{V_g} \left\{ (k-f)m + (f M_2 - N_1) \right\} \quad \dots \quad (8)$$

$$\text{or} \quad \frac{dm}{(k-f)m + (f M_2 - N_1)} = \frac{k_1 k_2 S}{V_g} dx$$

whence

$$\text{Log}_e \left\{ (k-f)m + (f M_2 - N_1) \right\} = \frac{k_1 k_2 S}{V_g} (k-f)x + \text{const.} \quad (9)$$

Now for $x=0$, $m=M_2$
and for $x=h$, $m=M_1$.

The first limiting condition shows that $\text{const.} = \text{Log} (k M_2 - N_1)$ so that:—

$$\text{Log}_e \frac{\{(k-f)m + (f M_2 - N_1)\}}{k M_2 - N_1} = \frac{k_1 k_2 S(k-f)}{V_g} x \quad \dots \quad (10)$$

The second limiting condition gives the equation:—

$$\text{Log}_e \frac{\{(k-f)M_1 + (f M_2 - N_1)\}}{k M_2 - N_1} = \frac{k_1 k_2 S(k-f)h}{V_g} \quad \dots \quad (11)$$

Subtracting (11) from (10) we have:—

$$\text{Log}_e \frac{\{(k-f)m + (f M_2 - N_1)\}}{(k-f)M_1 + (f M_2 - N_1)} = \frac{k_1 k_2 S(k-f)}{V_g} (x-h) \quad (12)$$

For given values of V_g , M_1 , M_2 , and N_1 equation (11) gives the relation between V_l , S , and k , or if V_g , M_1 , M_2 , N_1 , S , and h , are specified, we can calculate from this equation the value of V_l . Or again, if V_g , S , and h are specified (assuming $N_1=0$), we can determine the value of V_l required for any given value of r .

If, as will often be the case, $N_1=0$, we can write

$$\text{Log}_e \frac{(k-f) + f M_2}{k M_2} = \frac{k_1 k_2 S(k-f)}{V_g} x \quad \dots \quad (10')$$

$$\text{Log}_e \frac{(k-f)M_1 + f M_2}{k M_2} = \frac{k_1 k_2 S(k-f)h}{V_g} \quad \dots \quad (11')$$

and

$$\text{Log}_e \frac{(k-f)m + f M_2}{(k-f)M_1 + f M_2} = \frac{k_1 k_2 S(k-f)}{V_g} (x-h) \quad \dots \quad (12')$$

Equation (11') can be written in the form:—

$$\text{Log}_e \frac{k-f(1-r)}{kr} = \frac{k_1 k_2 S(k-f)h}{V_g} \quad \dots \quad (13)$$

If we also assume that M_2 is zero or negligibly small, equation (12') can be written in the simple form

$$\text{Log}_e \frac{m}{M_1} = \frac{k_1 k_2 S(k-f)}{V_g} (x-h) \quad \dots \quad (14)$$

$$\text{or} \quad m = M_1 e^{-\frac{k_1 k_2 S(k-f)}{V_g} (h-x)} \quad \dots \quad (15)$$

If the liquid issuing from the base of the tower be in equilibrium with the entering gas, then (if $N_1=0$)

$$\begin{aligned} V_g(M_1 - M_2) &= V_l k M_1 \\ \text{or} \quad \frac{V_l}{V_g} &= \frac{1-r}{k} \quad \dots \quad (16) \end{aligned}$$

Integration of equation (5) leads to similar equations relating m with x , namely:—

$$\text{Log}_e \frac{\frac{k-f}{f} n + k \left(M_2 - \frac{N_1}{f} \right)}{k M_2 - N_1} = \frac{k_1 k_2 S}{V_g} (k-f)x \quad \dots \quad (17)$$

$$\text{Log}_e \frac{\frac{k-f}{f} N_2 + k \left(M_2 - \frac{N_1}{f} \right)}{k M_2 - N_1} = \frac{k_1 k_2 S}{V_g} (k-f)h \quad \dots \quad (18)$$

If $N_1=0$, then equation (17) reduces to

$$\text{Log}_e \frac{\frac{k-f}{f} n + k M_2}{k M_2} = \frac{k_1 k_2 S}{V_g} (k-f)x \quad \dots \quad (19)$$

It will be observed that the equations given above reduce to identities if the factor $(k-f)$ be zero. The explanation of this is simple. Consider equation (2), i.e. $V_g \delta m = V_l \delta n$. If gas and liquid be in absorption-equilibrium at every level, then $n=kn$ and $\delta n=k \delta m$. Hence

$$V_g \delta m = k V_l \delta m, \text{ or } k = \frac{V_g}{V_l} = f.$$

But the supposition that gas and liquid are in absorption equilibrium at every level involves the conclusion that no interchange of the absorbable constituent occurs in the tower. For absorption to occur we must have $n < km$.

The aid which it is hoped these equations may ultimately afford in the design of towers to produce a predetermined effect may be briefly indicated by reference to equation (13), i.e.

$$\text{Log}_e \frac{k-f(1-r)}{kr} = \frac{k_1 k_2 S(k-f)h}{V_g}$$

Suppose we have to handle a given flow of gas V_g . Choice of temperature and solvent fixes k . If the tower is to be designed to effect a given ratio of absorption, say 0.01, then $r=0.01$. Choice of a certain flow of liquid and a certain type of packing leads to the fixing of f , k_1 , and k_2 . We are then left with S and h , which are now subject to the relation $Sh = a$ predetermined value. Practical considerations would then determine suitable values of S and h . We can certainly never expect to include all the varying conditions of practice in a simple set of algebraic equations.* But we may hope to employ such equations as a *guide* to practice as fully and as discreetly as the civil, mechanical, or electrical engineer employs his equations to guide him in the design of a bridge, a steam-turbine, or a dynamo. No attempt has been made in this paper to discuss different types of tower packing and their relative merits. Attention has been concentrated on the general physico-chemical principles governing the operation of scrubbing-towers in a specified simple case, with the object of obtaining a preliminary scientific basis for the subsequent treatment of questions of design.

It may suffice for the present to point out that the present paper shows in a quantitative manner how a high absorption efficiency depends on:—

- (a) High interfacial area between gas and liquid.
- (b) High relative motion of gas and liquid (within limits).
- (c) High degree of turbulent motion in one or both phases.
- (d) Sufficient rate of flooding to secure the maximum drip effect.

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DISCUSSION.

Dr. R. LESSING said that some means must be found for arriving at an expression for viscosity. In scrubbing coal gas with oil viscosity was of

*Frequently heat effects and temperature changes will play a prominent part. In this initial survey of the theory we have assumed that by suitable means the temperature is kept constant.

great importance, especially in its relation to subdivision and the turbulence to be effected. To make k , as large as possible a certain smallness of the filling body was necessary, particularly if the mass of the bodies was to be considered. It was essential that the total volume of the tower packing should be reduced to the minimum. That could only be done when the mass of the packing bodies was of small cubic capacity. For instance, if solid spheres of equal size were packed into a tower, 74% of the total capacity of the tower would be taken up by the solid material. In modern packings the idea was to reduce the amount of solid matter unapproachable by the liquid or gas and increase the surface between the packings. Raschig rings had the advantage that they occupied an exceedingly small proportion of the total volume of the tower; if made of ordinary sheet metal the standard 1-in. rings only occupied 7–9%, if of clay or acid-resisting material or cast iron 16–20%. They were disposed indiscriminately in the tower, and according to the angle formed between adjoining rings the cross-section of the open space between them varied. The descending liquid only had a certain small drainage space at its disposal without being stopped, and it was at that point that viscosity played a very considerable rôle. He had therefore designed another type of ring, which he had found to be very efficient both as regards total surface and drainage capacity. Unless the liquid running down the tower had a good drainage there would be stoppages, and back pressures would be set up which had a vital bearing on the gas stream outside the tower. To minimise back pressure on the gas he had designed a ring of circular shape with a centre partition which did not touch the opposite side of the circumference, nor was the circumference quite complete, so that there was at these places a considerable opportunity for drainage of the liquid. Had the authors considered that there might be a critical point at which turbulence might be excessive, i.e., that at that point the release of the dissolved gas from the liquid might be equally facilitated, as was the absorption of that same gas? To release the gas by heating, the same methods could be employed, and, supposing that a tower of this kind were designed which gave the highest possible efficiency at the top and at the bottom, then somewhere near the centre there must be equilibrium. He suggested that distillation or fractionation might be drawn within the sphere of calculation in connexion with solubility. He could not say whether that problem would be simpler or more complex. Using packed aggregates such as he had mentioned the results with fractionation were even more striking than with absorption of gases in liquids. He had been able to obtain fractionations with small rings which were better than the best obtained with Young's evaporative still-head. If it were possible to do this on the large scale, and to use these smaller bodies, the efficiency would be enormous and very small total tower capacities could be used. The 1-in. rings had 65–70 sq. ft. of surface per cu. ft., whereas the smaller rings had about 275 sq. ft. per cu. ft.

Prof. PARTINGTON said that the authors had attempted to take account not only of the differential rate of flow, which Dr. Parker and he had done in their paper, but of the change of concentration of the liquid throughout the tower. That had been introduced by Sorel before Dr. Parker and himself had done their work on the subject. They did not, however, take that into account, because in practice in nitric acid towers there was very little change of concentration of the liquid in passing through the tower. The rate of circulation was so high that the liquid had much the same composition throughout the tower. The most important factor in these equations was the absorption coefficient, i.e., the weight of soluble matter

absorbed from the gas in a given time by a given surface of liquid of given composition. The authors had attempted to bring that into line with the Henry coefficient. In practice, of course, the Henry coefficient did not apply to many cases, since the solubility of a gas was not proportional to its partial pressure, unless the gas was sparingly soluble. Dr. Parker and himself had considered that and had decided to abandon the use of Henry coefficients for that reason and to unite in one coefficient a number of these different separate variables. Theoretically it was very desirable to have these variables separated; but, as already pointed out, this absorption coefficient depended on turbulence, on the concentration of the gas and liquid, and so on, and his own case was complicated still further by the fact that chemical action took place in the liquid and an insoluble gas was again emitted. Nevertheless, by some work of Dr. E. K. Rideal, which had been suggested by Prof. Donnan, they had found that by suitable calculations it was possible to get a practical absorption coefficient which would apply to various fixed concentrations of liquid. All that was required to be known was the concentration of the acid in the tower, and that was fairly well fixed in a series of nitric acid towers. Then it became interesting to find by calculation if their simple equations would reproduce in any way the actual practical towers which were in use in various places, and, as they showed in their paper, it was possible to calculate the capacity of an absorption plant for the arc process such as was used in Italy with very great accuracy. Since that paper had been published, Dr. Parker had considerably extended the method of calculation and had obtained results which were quite close to those obtained in practice, including the new practice in French factories.

Captain C. J. GOODWIN pointed out that the authors had assumed throughout the paper that the rate of flow of liquid down the tower would be constant. In a great many cases that was not so. The modern tendency was not to have a steady rate of flow for the liquid, but to resort to periodical flushing, and he suggested that the authors should deduce from their formulæ the necessary coefficients for a tower in which it was assumed that the liquid retained on the surface of the packing was renewed or nearly renewed at each flushing, the quantity of liquid delivered each time corresponding approximately to that held on the wetted surface of the packing. In regard to direction of gas flow, it was generally assumed that contraflow must be more efficient than parallel flow. In some cases it was not so. With parallel flow of liquid and gas the time of contact was increased, and subject to considerations of the densities of the gases under treatment an improved efficiency was often obtainable by using parallel flow. The crux in the design of towers was the correct proportioning of scrubbing surface relatively to the maximum amount of free space economically permissible. Maximum scrubbing surface was not so important in reaction towers as distinct from absorption towers, because beyond certain limits an increase in scrubbing surface was useless if the rate of absorption exceeded the rate of formation of the substance to be absorbed.

Mr. W. MACNAE said that much work had been carried out during the war in various Government factories, and they had generally worked with 50% of free space in the absorption towers. The amount of nitrogen peroxide which was found to give the best results was 1 lb. per minute per 120 cu. ft. of free space when the surface area was 5000 sq. ft. Very uniform results were obtained over a long period of time in this way. That was working with 4000 Accrington earthenware rings of 3-in. size per tower. It was impracticable to fill up the large number of towers that had to be dealt with, with the

smaller rings mentioned by Dr. Lessing, on account of the expense.

Mr. H. GRIFFITHS observed that one of the fundamental assumptions in the paper was that the temperature remained constant throughout the tower. This was not always so in practice, and if the theory were applied in some cases the error would be very serious. In the case of hydrochloric acid it would easily be possible to feed cold water into the top of the tower and have a boiling solution issue at the bottom; in fact the boiling point would be reached even with as low a concentration as about 13% HCl. The authors had also assumed that the absorbing liquid was simply allowed to trickle through the tower, whereas in practice it was exceedingly common to employ a much larger circulation, and to pump the liquid from the bottom outlet N_2 and return to the tower at the top N_1 , a small portion only being short circuited to allow for the feed. Dr. Partington (*loc. cit.*) had taken this practice into account. Occasionally, in order to control the temperature within the tower, additional cooling surface would be added in the liquid circuit outside the tower. The constant k , had become a sort of accepted basis for judging the relative efficiencies of the various packings, but comparative tests showed that the various patent packings for which high values of surface per cubic foot could be secured, and for which in consequence high efficiencies were claimed, did not function so satisfactorily. This was probably because these packings consisted of a fairly dense arrangement of materials of low thermal conductivity, and the dissipation of heat of reaction from the tower was hindered. He had been told that in past years it was customary in the case of nitric acid towers to express the data for design in terms of lb. of nitric acid per hour per sq. ft. of radiating surface of the tower, therein taking account only of the thermal effect in the tower.

Dr. PARKER enlarged upon the point raised by Captain Goodwin with regard to the space left for reactions to take place in the tower which was unoccupied by the packing material. In the paper communicated by Dr. Partington and himself they were there only concerned with the reactions involving the formation of nitric acid in water absorption towers; one of the most important of these was the reaction between nitric oxide and atmospheric oxygen for the formation of nitrogen peroxide, in the absence of which no nitric acid could be formed in the absorbing liquid. From practical experience he could say that there were plants in operation in which there was from four to seven times more area exposed by the packing in the absorption towers than was necessary for the absorption of the NO_2 ; the result was that the efficiency of the absorption system was of the order of 70% instead of 90%, as claimed by the engineers in charge. It was very probable that the efficiency of absorption of such plants could be materially increased by a judicious unpacking of some of the towers, to leave more free space for the formation of nitrogen peroxide without decreasing the absorbing surface of the packing to such an extent as to inhibit the formation of nitric acid.

Mr. P. PARRISH said that for 20 years he had been designing towers in connexion with sulphuric acid manufacture, which offered very complex conditions, because the design of the Glover tower was dependent not only on the proportion of the chamber plant and the burners, but the capacity and effectiveness of the Gay-Lussac tower had a very important bearing on the operation of the Glover tower. Prof. Donnan decried the fact that there was not much data available with regard to the theory of absorption, but Hurter, 30 years ago, had given a really remarkable exposition of the theoretical considerations underlying absorption. Little practical data was available for use or appli-

cation by the practical chemical engineer. For 15 years the only data that the chemical construction engineer had had in connexion with the design of the Glover tower in connexion with sulphuric acid plants was that for every ton of sulphur burned daily in the form of pyrites it was necessary to have 550 cb. ft. of Glover tower capacity. That paid no regard to mass, to available surface area, or to the effective scrubbing surface per cubic foot. Some two years ago, as the result of designing some 50 towers in connexion with sulphuric acid plant, he had attempted to correlate the theoretical and the practical data which he had been able to amass. In regard to the design of Glover towers, in so far as they affected the manufacture of sulphuric acid, he had come to the conclusion that it was essential to afford 4½ sq. ft. of surface area per ton of monohydrate sulphuric acid made per day at a point in the tower where the lining of the tower was not more than 9 in. He had then developed in connexion with 10 different types of packing the free space per cb. ft., the superficial free space per sq. ft., the inlet area required, and the scrubbing surface in sq. ft. per cb. ft., and as a result of these considerations he had found that in many cases Glover towers had been over-designed to the extent of 300%, and in some cases towers were operating which were 500 and 600% more than was theoretically necessary. There were quite a number of other considerations in the functions of a Glover tower apart from absorption. In the first place, in the lower portion of the Glover tower concentration primarily occurred. Denitration also was effected, and such denitration took place within very narrow and well-defined limits. Assuming that the tower was over-designed, abnormal quantities of sulphuric acid were formed to the extent of from 10 to as much as 40% of the total make. It was very difficult to say precisely the point at which concentration ceased and denitration commenced, and the point at which denitration left off and making commenced, and the correlation of the various parts constituting a sulphuric acid plant must be taken into consideration both in connection with Glover towers and Gay-Lussac towers. The tendency in the future so far as absorption and reaction under the conditions he had outlined were concerned would be not so much reaction between gas and liquid *via* a medium which afforded a sort of skin friction, but to have a bubbling of the inter-phase—gas and liquid. He referred particularly to the Kessler type of plant. In this the material of which the calottes were made would not stand the stringent conditions to which they were subjected, and resort had to be had eventually to a filling medium. Experience of the working of the Kessler plant under the modified conditions suggested that the future tendency would be in the provision of some arrangement which admitted of actual bubbling of the gas through the liquid medium in order to reduce the limits and dimensions of the plant, concurrently reducing the capital expenditure thereon.

Dr. W. R. ORMANDY mentioned that he had recently designed a certain absorption tower in which the air carrying the small amount of soluble matter entered the bottom practically cold and in which no chemical reaction took place. He had no data to go upon, and so had allowed a large factor of safety and then doubled it. He believed that this tower would provide Prof. Donnan with exactly the data he required in order to build a theory.

Dr. IRVINE MASSON, in reply, said that the authors regretted they had had to leave aside for the moment those very interesting and perhaps more important kinds of tower where chemical reactions were performed. In these were included Glover towers, Gay-Lussac towers, and NO_2 towers. Even then, however, there were many applications of scrubbing towers where a simple physical kind of solution had to be brought about, and it was to

these that attention had first been directed. The simplifying assumptions, which had been justly commented on, were made with the idea he had mentioned. For instance, the thermal effect due to the condensation of any vapour or gas must always be taken into account in designing a tower, but as a beginning a theory for an isothermal process was needed. An equation was wanted to start with which could be elaborated factor by factor, and the thermal effect was one of these which would naturally be brought in as soon as possible, but it had not yet been done. The paper by Dr. Partington and Dr. Parker had given the first quantitative expression to be actually published, and as such it must receive great attention. It was to be noted that the assumptions made by Dr. Partington referred to the special case of nitric acid, which was not matched by any of the simple cases of physical solution which the authors of the present paper were considering. The essential difference, mathematically speaking, between the special case discussed by Dr. Partington and the one dealt with in this paper was the introduction of the factor $k-f$. It was necessary to take account of the fact that not only did the liquid absorb gas, but the gas tended to come out again. The rate of solution of the gas in a liquid surface per unit area per unit of time was proportional to the degree of unsaturation of that liquid, which depended not only on the concentration in the gas phase itself, but on the concentration of the gas already absorbed in the liquid. That was not the case in the investigation by Messrs. Partington and Parker.

Dr. PARTINGTON: We allowed for that in the calculation.

Dr. MASSON agreed that Messrs. Partington and Parker's factor k , obtained by experiments, empirically covered effects of this nature in the case dealt with; Professor Donnan and he had not done such experiments, but had attempted a generalised solution of the problem by the use of the Henry constant k and the variable factor k_2 .

Manchester Section.

Meeting held at Grand Hotel on May 7th, 1920.

MR. JOHN ALLAN IN THE CHAIR.

HYDROGENATION IN THE NAPHTHALENE SERIES.

BY F. M. ROWE, M.Sc., A.I.C.

In the course of the investigation of the reducing action of sodium and an alcohol carried out by Bamberger and his collaborators, hydro derivatives of naphthalene, the naphthylamines, naphthols, aminonaphthols, and naphthylenediamines were prepared. The products are invariably tetrahydro derivatives when sodium and amyl alcohol are used, but in the case of naphthalene itself the use of ethyl alcohol results in the formation of the dihydro derivative. The yield and character of the reduction product vary with the individual substance reduced, for α -derivatives of naphthalene give rise mainly to aromatic (ar.) compounds, whilst β -derivatives form mainly alicyclic (ac.) compounds. The aromatic compounds contain the additional four hydrogen atoms in the unsubstituted ring, and possess an accentuated benzenoid character, whilst the alicyclic compounds, which contain the additional four hydrogen atoms in the substituted ring, have lost their aromatic character and assumed the characteristics of an aliphatic compound.

The attention of subsequent investigators has been devoted mainly to the further examination of ar-tetrahydro- α -naphthylamine and its derivatives, for the action of sodium and amyl alcohol on α -naphthylamine results in the formation of this base more readily and in a better yield (70% of the theoretical) than is the case when other derivatives of naphthalene are submitted to the same reaction.

Ar-tetrahydro- α -naphthylamine has been shown to possess many interesting properties. It retains the naphthalenoid characteristic of reacting with diazonium salts under ordinary conditions, forming aminoazo compounds without the intermediate formation of diazoamines, in which respect it differs from ar-tetrahydro- β -naphthylamine, which forms intermediate diazoamines but does not form aminoazo compounds. In all other respects ar-tetrahydro- α -naphthylamine resembles a benzenoid amine, particularly γ -xylylene ($\text{CH}_3\text{:CH}_2\text{:NH}_2$ 1:2:3).

Of further interest is the fact that azo dyes derived from ar-tetrahydro- α -naphthylamine and its derivatives differ considerably in shade from similar azo dyes derived from α -naphthylamine and its derivatives (Bamberger, Ber., 1887, 20, 2915; Bamberger and Bordt, Ber., 1889, 22, 625; Morgan and Richards, J., 1905, 652; Green and Rowe, Chem. Soc. Trans., 1918, 113, 955; Rowe, J. Soc. Dyers and Col., 1919, 35, 128), approximating in this respect to dyes derived from benzenoid amines.

As a result of these investigations it is clear that ar-tetrahydro- α -naphthylamine and its derivatives might well prove useful intermediate products to the manufacturers of dyes if the production of the base on a technical scale were economically possible.

The conditions governing the formation of the base therefore were examined more closely. Bamberger refers to the influence of the particular alcohol used in the reduction on the yield and constitution of the reduction product, for tetrahydro-naphthalene is formed when an amyl alcoholic solution of naphthalene is reduced with sodium, but dihydronaphthalene is formed when amyl alcohol is replaced by ethyl alcohol. Further, in the case of β -naphthylamine, the replacement of amyl alcohol by ethyl alcohol results in a minimum yield of the tetrahydro derivative. It was concluded that the course taken by the reaction and the yield obtained was intimately connected with the boiling point of the solvent employed, but the later experiments of Bamberger and Muller (Ber., 1882, 21, 1112), using such solvents as hexadecyl alcohol, phenol, glycerol, and mixtures of amyl alcohol and vaseline, did not support this hypothesis, at least in the case of β -naphthylamine.

When a boiling solution of α -naphthylamine in ethyl or butyl alcohol is treated with sodium no tetrahydro base is formed, whilst the addition of butyl alcohol to amyl alcohol produces a diminution in the yield of ar-tetrahydro- α -naphthylamine proportional to the quantity of butyl alcohol in the mixture. Moreover, in an experiment in which butyl alcohol was employed under a pressure sufficient to raise the boiling point to that of amyl alcohol under normal pressure no tetrahydro base was formed, though as the apparatus used in this experiment burst, it is possible, in the light of later experiments, that the reaction had not proceeded for a sufficient length of time to produce the desired result.

At the time it appeared that this difference in the behaviour of amyl alcohol from that of other alcohols was not due merely to differences in boiling point, and as the reaction was always accompanied by a destruction of 10–15% of the amyl alcohol used, it seemed possible that the reduction was intimately connected with this oxidation of amyl alcohol, and was not simply a direct hydrogenation to the tetrahydro compound.

In the course of these experiments it was noted that when butyl alcohol was used the melting point of the base was lowered, but as no tetrahydro base

was formed, this point was not investigated further at the time. Recently, however, experiment has shown that this lowering of the melting point is due to a partial conversion of the α -naphthylamine into dihydro- α -naphthylamine.

The effect of the presence of various catalysts on the reaction with sodium and a butyl alcoholic solution of α -naphthylamine was studied also, but in no case could the presence of the tetrahydro base be detected after reduction.

According to the patents of F. Bayer und Co. (G.P. 305,347 and 306,724), alkali and alkaline-earth metals and alcohols, such as ethyl alcohol, reduce isocyclic and heterocyclic bases in the presence of "indifferent solvents." It is stated that ar-tetrahydro- α -naphthylamine is formed when an alcoholic solution of α -naphthylamine is added to a mixture of solvent naphtha and sodium at a temperature above 130° C., but that when an indifferent solvent of lower boiling point, such as toluene, is used, dihydro- α -naphthylamine is obtained. Consequently it would appear that ar-tetrahydro- α -naphthylamine is formed when α -naphthylamine, dissolved in any alcohol, is added to a boiling mixture of solvent naphtha and sodium, and that replacement of solvent naphtha by toluene results in the formation of dihydro- α -naphthylamine, irrespective of the alcohol used. If such were the case, the important factor in the reaction is obviously the temperature at which the reduction is carried out and the nature of the alcohol used is immaterial.

In order to test this point a series of comparative experiments was carried out, in which 30 g. of sodium was covered with 400 c.c. of an indifferent solvent, raised to the boiling point, and a solution of 34 g. of α -naphthylamine in sufficient alcohol to dissolve the sodium was added slowly, the mixture being worked up as soon as all the sodium had disappeared. Under these conditions ar-tetrahydro- α -naphthylamine was formed only when amyl alcohol was used, and was produced equally well at the boiling point of toluene as at the boiling point of solvent naphtha (139° C.), whilst when ethyl or butyl alcohol was substituted for amyl alcohol, dihydro- α -naphthylamine was formed even at the boiling point of solvent naphtha. Further, when dihydro- α -naphthylamine was substituted for α -naphthylamine and treated with sodium, ethyl alcohol, and solvent naphtha as before, it remained unaffected by the treatment. It is clear, therefore, that some other factor is concerned in the reaction besides the temperature at which it is carried out.

This method of preparation is, nevertheless, an improvement on Bamberger's method for the preparation of ar-tetrahydro- α -naphthylamine, for it requires less than one-third of the quantity of alcohol previously necessary; it is more convenient to cover sodium with an indifferent solvent and to add an alcoholic solution of α -naphthylamine to the boiling mixture than to add sodium in several portions to a boiling alcoholic solution of α -naphthylamine; and further, by replacing amyl alcohol by another alcohol, the previously unknown dihydro- α -naphthylamine is obtained. In fact, as far as the preparation of hydrogenated naphthalene derivatives by means of sodium and an alcohol is concerned, it is doubtful whether this method can be improved upon.

The constitution of the dihydro- α -naphthylamine obtained in these experiments was next investigated by removing the amino group so that the corresponding dihydronaphthalene could be examined and identified. The literature with regard to dihydronaphthalene is rather involved for the following reasons:—

(1) It has not always been recognised that two isomers exist, viz., 1,4-dihydronaphthalene and 1,2-dihydronaphthalene, the former yielding

phenylene-diacetic acid and the latter hydrocinnamic-*o*-carboxylic acid on oxidation.

(2) The melting points of the dibromides of the two isomers, which have been used for their identification, lie close together, being 71.5°–72° C. and 70°–71° C. respectively. (Actually when these compounds are compared with one another they may be readily distinguished, for the former crystallises in long, glittering, thin prisms, while the latter forms stout prismatic crystals; a methyl alcoholic solution of the former remains neutral after prolonged boiling, whilst with the latter the solution reacts strongly acid after short boiling; the melting point of either is lowered by admixture with the other.)

(3) The dihydronaphthalenes form mixed crystals with naphthalene which cannot be separated completely by physical means. These mixed crystals melt considerably higher than the pure dihydronaphthalene.

Experiments which were carried out with the dihydronaphthalenes in order to check their properties confirmed the work of Straus and Lemmel (Ber., 1913, 46, 232).

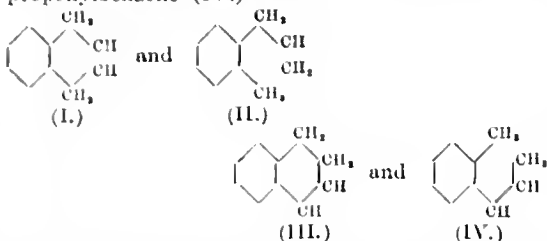
Crude 1,4-dihydronaphthalene, prepared by the method of Bamberger and Lodter by the action of sodium and ethyl alcohol on naphthalene (Annalen, 1895, 288, 75), was purified as described by Sand and Genssler (Ber., 1903, 36, 3705) by shaking an ethereal solution with an aqueous solution of mercuric acetate for 24 hrs. The crude mercury compound which separated was extracted with boiling benzene in a Soxhlet and crystallised in colourless needles, m.p. 121° C., which when decomposed with hydrochloric acid yielded pure 1,4-dihydronaphthalene, crystallising in plates, m.p. 24.5°–25° C.

The isomeric 1,2-dihydronaphthalene was first obtained by heating 1,4-dihydronaphthalene with five times its volume of 5% sodium ethoxide at 140°–150° C. for 8 hrs., but it was subsequently shown by Straus (Ber., 1913, 46, 1051) that both the temperature and concentration of the sodium ethoxide employed play a part in this reaction. When 1,4-dihydronaphthalene is heated with 5% sodium ethylate at its boiling point (87° C.) for 1 hr., there is no conversion, whilst a two-thirds conversion occurs at 105° C., and at 140° C. heating for $\frac{1}{2}$ hr. is sufficient for complete conversion. On the other hand, by using 10% sodium ethoxide the conversion is complete after heating at 100°–105° C. for 1 hr. The latter method was used for the preparation, except that it was found convenient to allow the alcohol to distil slowly during the reaction. In order to ensure purity, the product was converted into the mercury compound by shaking an ethereal solution with aqueous mercuric acetate, the product extracted with boiling benzene, in which the mercury compound of 1,2-dihydronaphthalene is insoluble, and decomposed with hydrochloric acid. Pure 1,2-dihydronaphthalene crystallises in colourless plates, m.p. –9° C.

There is no doubt that, in all investigations in which dihydronaphthalene has been prepared by the elimination of water, ammonia, hydrochloric acid, etc., from alicyclic derivatives of tetrahydronaphthalene and identified by means of the dibromide, the compound was actually 1,2-dihydronaphthalene and not 1,4-dihydronaphthalene as was supposed.

An important difference in properties between 1,4- (I.) and 1,2-dihydronaphthalene (III.) is that only the latter compound is reduced to tetrahydronaphthalene by sodium and ethyl alcohol. This fact disposed of the difficulty of reconciling the reduction of naphthalene through 1,4-dihydronaphthalene to tetrahydronaphthalene with the

behaviour of the analogous allylbenzene (II.) and propenylbenzene (IV.)—



only the latter of which is reducible by sodium and an alcohol to a saturated homologue of benzene, and consequently 1,4-dihydronaphthalene should not be directly reducible.

The preparation of tetrahydronaphthalene by Bamberger and Kitchelt by the action of sodium and amyl alcohol on naphthalene (Ber., 1890, 23 1561) is, therefore, explained by the initial formation of 1,4-dihydronaphthalene followed by isomerisation to the 1,2-dihydro compound and the formation of tetrahydronaphthalene under ordinary conditions only when amyl alcohol is used in the reduction and not when other alcohols are used, must be due to the fact that only in the former case are the concentration of sodium alkoxide and the temperature suitable for the isomerisation of the 1,4-dihydro derivative prior to further hydrogenation. The dihydronaphthalene obtained from the dihydro- α -naphthylamine previously referred to proved to be the 1,4- isomer and consequently the amino is 5,8-dihydro-1-naphthylamine. It crystallises in large colourless, rhombic plates or needles, m.p. 37°–38° C., and its hydrochloride crystallises in stout needles. The first stage in the reduction of α -naphthylamine, therefore, is similar to the first stage in the reduction of naphthalene, and if the two cases are completely analogous it is clear that this dihydro- α -naphthylamine would not be reduced to ar-tetrahydro- α -naphthylamine by a further treatment with sodium and ethyl alcohol, unless the conditions of the reaction were such as to bring about a preliminary conversion into the isomeric dihydro- α -naphthylamine by boiling with sodium ethoxide.

In order to test this hypothesis the reduction mixture employed for the preparation of 5,8-dihydro- α -naphthylamine was boiled for a number of hours after all the sodium had disappeared, instead of being worked up immediately. It was found that the reaction product was a mixture of two amines in this case and that by increasing the proportion of sodium employed or by using more concentrated sodium ethoxide and boiling for several hours, the whole of the 5,8-dihydro- α -naphthylamine was converted into this new amine. Further, this amine is readily reduced to ar-tetrahydro- α -naphthylamine by treatment with sodium and ethyl alcohol, even in the absence of a high boiling indifferent solvent. The hydrocarbon obtained from this base by removing the amino group proved to be 1,2-dihydronaphthalene, and consequently the amine is 5,6- dihydro- 1- naphthylamine or 7,8- dihydro-1-naphthylamine. It is a colourless oil, which does not solidify in a freezing mixture of ice and salt, and its hydrochloride crystallises in needles, whilst ar-tetrahydro- α -naphthylamine, which is also an oil, forms a hydrochloride which crystallises in pearly plates. It condenses with diazonium salts, forming aminoazo compounds, and when diazotised and coupled with amines and phenols it forms azo dyes.

The further investigation of the isomeric dihydro- α -naphthylamines is at present in progress.

The course of the reaction in the formation of ar-tetrahydro- α -naphthylamine from α -naphthylamine by the action of sodium and an alcohol is completely analogous to the conversion of

naphthalene into tetrahydronaphthalene, the first stage being the formation of 5,8-dihydro-1-naphthylamine, which is isomerised to the 7,8-compound, this latter then being reduced to the tetrahydro derivative.

Apparently the reason why ar-tetrahydro- α -naphthylamine has been obtained in the past only when amyl alcohol has been employed, and not when other alcohols have been used, is that only in the former case have the normal conditions of the reaction been suitable for the isomerisation of the intermediate 5,8-dihydro- α -naphthylamine.

It seemed desirable now to investigate more closely the direct hydrogenation of naphthalene and its derivatives by means of hydrogen in presence of a catalyst.

Sabatier and Sonderens, who prepared cyclohexane by passing a mixture of benzene and hydrogen over finely divided nickel at 180°–200° C. (Comptes rend., 1901, 132, 210), found that tetrahydronaphthalene was formed from naphthalene by a similar treatment at 200° C. (*ibid.*, 1901, 132, 1254). Ipatiev obtained successively tetrahydro- and decahydro-naphthalene by heating naphthalene with hydrogen in presence of nickel or its oxide at 250° C. under 120 atm. pressure, and α - and β -naphthol were converted in a similar manner into the corresponding decahydronaphthols. Willstätter and Hatt obtained perhydro- and decahydro-naphthalene by shaking an ethereal or glacial acetic acid solution of naphthalene with platinum black and hydrogen in a closed vessel (Ber., 1912, 45, 1474), whilst Willstätter and King, using the same method, found that naphthalene did not form tetrahydronaphthalene at any stage of the process, but that a mixture of naphthalene and perhydro-naphthalene was always produced until all the naphthalene was converted into perhydronaphthalene (*ibid.*, 1913, 46, 527). The same authors, however, obtained tetrahydronaphthalene from pure dihydronaphthalene—prepared by the exhaustive methylation of ac-tetrahydro- β -naphthylamine—which they believed to be 1,4-dihydronaphthalene, but which was actually the 1,2-isomer.

A method by which Lebeau and Pichon obtained tetrahydronaphthalene in a 90% yield consisted in enclosing a mixture of liquefied ammonia, sodium, and naphthalene in an autoclave, and allowing it slowly to attain the air temperature (Comptes rend., 1914, 158, 1514). In this reaction the sodamide formed must influence the course of the hydrogenation, as nascent hydrogen alone does not convert naphthalene into tetrahydronaphthalene.

In the case of the hydrogenation of aromatic amines, Sabatier found that the products obtained by passing a mixture of aniline and hydrogen over nickel at 190° C. were ammonia, benzene, cyclohexane, cyclohexylamine, dicyclohexylamine, and cyclohexylaniline (Comptes rend., 1904, 138, 457); a mixture of 40–50% cyclohexylamine, 10% dicyclohexylamine, and 10% cyclohexylaniline was obtained by Ipatiev by heating aniline and hydrogen in presence of nickel at 220°–230° C. under a high pressure for 50 hours; and Willstätter and Hatt obtained dicyclohexylamine as principal product with only 10% of cyclohexylamine by shaking a glacial acetic acid solution of aniline with hydrogen in presence of platinum black in a closed vessel at the ordinary temperature (*loc. cit.*).

The reduction of α -nitronaphthalene to α -naphthylamine with an excess of hydrogen in presence of a catalyst at 330°–350° C. was examined by Sabatier and Senderens (Comptes rend., 1902, 135, 225), who found that copper was the most suitable catalyst, as nickel gave rise to the elimination of ammonia and formation of tetrahydronaphthalene.

With regard to this elimination of ammonia during the reduction of amines, the reaction is reversible under suitable conditions, for Meyer and Tanzen found that traces of aniline are formed

when a mixture of benzene and ammonia is passed over nickel at 550° C. (Ber., 1913, 46, 3184), presumably as the result of a simultaneous dehydrogenation of the ammonia and hydrocarbon. In order to determine whether naphthalene and ammonia behaved in an analogous manner we carried out some experiments in which a mixture of ammonia and naphthalene was passed over nickel at 300° C., and obtained a yield of about 2% of base which appeared to be a mixture of α -naphthylamine and 5,8-dihydro- α -naphthylamine, and hydrogen was detected. The reaction is probably:—



The possibility of simultaneous dehydrogenation occurring must be taken into consideration in attempts to hydrogenate naphthalene derivatives, for the addition of hydrogen to an unsaturated aromatic compound in the presence of a catalyst may be reversed under suitable conditions, leading to a state of equilibrium between the hydrogenated and dehydrogenated compounds. In view of the results already obtained in the catalytic hydrogenation of aromatic compounds and amines in particular, it was decided to employ a nickel catalyst deposited on kieselguhr for our experiments on the hydrogenation of α -naphthylamine and to carry out similar experiments with naphthalene, which might serve as a guide in the former case. The hydrogen employed was purified by passing through 40% caustic soda solution, concentrated sulphuric acid, a heated copper tube containing a roll of electrolytic copper gauze, and a U-tube containing moistened caustic potash.

The process adopted for the experiments, which should be regarded as preliminary and of a qualitative nature only, consisted in prolonged bubbling of hydrogen at the ordinary pressure through the purified substance, either molten or dissolved in a suitable solvent, in presence of nickel deposited on kieselguhr, in a glass vessel fitted with a condenser and an agitator capable of being driven at a high speed.

Temperatures varying from 100° C. to the respective boiling points were tried with naphthalene and α -naphthylamine, in the absence of solvents, without result, except that there was an appreciable evolution of ammonia in the latter case, particularly at the higher temperatures used.

Negative results were obtained also in the case of α -naphthylamine dissolved in such solvents as petroleum ether, toluene or solvent naphtha, the experiments being carried out at the boiling point of the solvent. The first positive results were obtained by using a boiling ethyl alcoholic solution of naphthalene, when it was found that hydrogenation occurred. Under the conditions used, the products obtained were invariably mixtures which, however, could be separated readily. The first main product of hydrogenation proved to be 1,4-dihydronaphthalene, and this appears to be the first instance of the preparation of a dihydro derivative in the naphthalene series by direct hydrogenation in presence of a catalyst. A more prolonged reaction, or treatment of 1,4-dihydronaphthalene in boiling ethyl alcoholic solution, led to the formation of some tetrahydronaphthalene together with a mixture of lower boiling liquids, which were not examined closely, but were probably the more saturated derivatives.

Hydro derivatives of naphthalene were of considerable importance in Germany during the war. Tetrahydronaphthalene or "tetralin" and decahydronaphthalene or "dekalin" are valuable solvents and motor fuels which are now available in Germany in large quantities, and are manufactured by the direct hydrogenation of naphthalene (Vollmann, *Farben-Zeit.*, 1919, 24, 1689). It is not within the scope of this present paper to emphasise the importance of hydrogenated derivatives of

naphthalene as motor fuels, but it should be noted that these compounds, whilst having approximately the same calorific value as naphthalene, are mobile liquids, and that there must be some stage in the hydrogenation at which the compound will burn like a fatty compound without deposition of carbon in the cylinder.

An investigation of these compounds from this point of view is at present in progress in collaboration with Captain F. S. Sinnatt, the results of which will be presented to this Society on some future date.

With regard to α -naphthylamine positive results were obtained also when the treatment was carried out in a boiling ethyl alcoholic solution, but the hydrogenation was more satisfactory when boiling mixtures of ethyl alcohol and toluene or ethyl alcohol and solvent naphtha were used.

In the latter case, the α -naphthylamine was completely converted into dihydro- α -naphthylamine by prolonged bubbling of hydrogen, the catalyst being replaced by a fresh portion half way through the reaction. Examination proved this compound to be 5,8-dihydro- α -naphthylamine. Attempts were made to convert this compound into ar-tetrahydro- α -naphthylamine by further treatment, but whilst there was evidence of some reduction taking place, under the conditions used, no satisfactory conversion occurred. In view of these preliminary results, an apparatus is being constructed which will allow the experiments to be carried out under an increased pressure, and when complete this investigation will be continued.

In conclusion, I wish to express my thanks to Miss E. Levin and Mr. S. Loo, who have carried out a large number of the experiments connected with this work, and to Messrs. British Dyestuffs Corporation (Blackley), Ltd., who have kindly supplied me with the sodium and α -naphthylamine required in this investigation.

Dyestuffs Research Laboratory,
Municipal College of Technology,
Manchester.

DISCUSSION.

The CHAIRMAN said that the author's results indicated that a certain reaction proceeded much more efficiently in the presence of a copper than it did in the presence of a nickel catalyst, although, so far as was known, the mechanism in the two reactions was the same. If the mechanism were the same it would appear that the condition of the catalyst, the temperature at which it was operated, and the concentration in which it was used might play a very considerable part in the results. Very complex mixtures were obtained in the hydrogenation of naphthalene, and most of the products were obtained as the result of secondary reactions. By removing the desired product from the zone of reaction quickly it might be possible to control the results.

Professor PRYAN said that the naphthalene series was connected in a very simple manner with derivatives of isoquinoline. Now the reduction of isoquinoline and particularly of its derivative papaverine, had been studied pretty fully. Papaverine was an isoquinoline containing three substituents, methoxyl groups in the 6- and 7-positions, and a dimethoxybenzyl residue in the 1-position. Reduction of papaverine gave, to some extent, tetrahydropapaverine, and also a dihydropapaverine, but this was not analogous to anything Mr. Rowe had shown. It contained undoubtedly a bi-cyclic ring in the place of the pyridine ring, the 1- and 3-carbon atoms being united by a bridge, for when it was de-graded by suitable methods it gave derivatives of indene. This was perhaps interesting by way of contrast with the naphthalene series. In using sodium and alcohol in considerable quantities he found it con-

venient to employ the following method: The sodium was put in at the bottom of a large bolt head fitted with a reflux condenser and a dropping funnel and covered with a small proportion of the alcoholic solution, the whole being heated in an oil bath, and the bulk of the alcoholic solution was then introduced through the dropping funnel; by this means the reduction of considerable quantities was conveniently effected. Caro was necessary with amyl alcohol. Windans, who had worked a great deal with cholesterol, had shown that in this case the action of sodium and amyl alcohol did not simply effect reduction, but also addition of an amyl alcohol residue.

Mr. J. BADDILEY said that, within certain limits, there appeared to be an analogy between addition in the case of hydrogenation and substitution in the case of sulphonation of naphthalene. Apparently, in the hydrogenation of the naphthalene derivatives investigated, the reaction went through the *p*-dihydro compound to the *ortho*, just as in sulphonation the first-formed product was the α -acid which was then isomerised to the β -acid. In hydrogenation by means of sodium and alcohols the author had shown that only in the case of amyl alcohol did the hydrogenation pass beyond the dihydro stage. Further progress to the tetrahydro stage depended on the isomerisation of the 1,4-dihydro to the 1,2-dihydro compound. It was possible that whereas other alcohols had none, amyl alcohol had a specific isomerising effect, this difference being paralleled by the different isomerising effect of different strengths of sulphuric acid in the case of sulphonation. Had the author tried the effect of the presence of amyl alcohol in catalytic reductions? With regard to azo dyes he bore out what the author had said about the analogy between the behaviour of tetrahydro- α -naphthylamine and that of certain amines of the benzene series. In coupling with diazo compounds, however, *p*-xylylene presented a closer analogy than did *o*-xylylene.

Dr. T. CALLAN asked if the hydrogenated derivatives of the naphthylamines showed the same difference in their behaviour to nitrous acid, *e.g.* in the difficulty of obtaining clear diazo solutions and quantitative absorption of nitrous acid, as did the corresponding non-hydrogenated derivatives. Had the effect of traces of water on the reduction process been studied, and had other metals, such as calcium, been employed in place of sodium in the reduction process? Further, if the hydrogenated naphthylamine derivatives resembled the xylylenes in their characteristics, did they condense with benzaldehyde to form crystalline benzylidene compounds such as *p*-xylylene gave, or non-crystalline compounds such as the other xylylenes formed?

Captain F. S. SINNATT said that a compound of the nature of tetrahydronaphthalene which would have a fairly high calorific value and was a liquid should find a wide use as a fuel. The substance was no doubt easily soluble in alcohol, and if it would burn in the same manner as a fatty compound it could be added to alcohol to increase its calorific value.

Dr. J. A. R. HENDERSON remarked upon the difficulty in identifying the 1,2- and the 1,4-dihydronaphthalenes, and asked if the author had studied the action of chromyl chloride upon these two isomers. Chromyl chloride had been used with great success in the study of the constitution of the terpenes, and probably might serve to differentiate the dihydronaphthalenes.

Mr. W. A. SILVESTER said that in referring to the use of hydrogenated naphthalenes as motor-fuels the author had not stated the boiling-points of the compounds. They should be comparatively

high, and he had gathered that in Germany the compounds had been mainly applied as substitutes for lubricating oils. It was possible that the use of high temperatures in the Sabatier-Senderens process resulted in a cracking similar to that which occurred when petroleum hydrocarbons were heated.

Mr. Rowe, in reply, said that the secondary reactions which might take place in connexion with direct hydrogenation could not be better exemplified than in the case of the catalytic hydrogenation of aniline. Tetrahydroquinoline and tetrahydroisoquinoline were particularly interesting to him. The former closely resembled monomethylaniline in its properties and the latter benzylamine. The *N*-methyl derivative of tetrahydroquinoline resembled dimethylaniline, for it formed a *p*-nitroso derivative, and condensed with benzaldehyde to form an analogue of Malachite Green, whilst *p*-aminotetrahydroquinoline behaved like an alkylated *p*-phenylenediamine giving the Indamine, Safranin, and Methylene Blue reactions. As all his experiments on the direct catalytic hydrogenation of naphthalene had been qualitative only, up to the present time, no figures could be given as to the relative cost of tetrahydronaphthalene and other fuels. The analogy drawn by Mr. Baddiley between hydrogenation and sulphonation in the naphthalene series was interesting. Amyl alcohol had been tried in the experiments on catalytic hydrogenation, but as it took part in the reaction, resulting in a complex mixture of products very difficult to deal with, its use was abandoned. It was true that tetrahydro- α -naphthylamine, whilst resembling an *o*-xylylene in structure, more closely resembled a *p*-xylylene in properties. There was less difficulty in obtaining clear diazo solutions with tetrahydro- α -naphthylamine than with α -naphthylamine. He hoped to have more to say with regard to azo dyes derived from hydrogenated amines when work, at present in progress, was completed. The effect of the presence of traces of water on reduction with sodium and an alcohol had been studied. Dry alcohol was essential, for when naphthalene was reduced with 96% ethyl alcohol and sodium, a product was obtained melting at 37°–40° C. which was a mixture of 1,4-dihydronaphthalene with unaltered naphthalene. Bamberger and Lodter stated that such a product remained unaltered on further treatment with sodium and ethyl alcohol, but he had not found that to be the case. This mixture was readily separated by converting the naphthalene into the picrate, but by repeated crystallisation from alcohol the melting point was only raised to 59° C., at which point it remained constant. Bayer's patents, which he had mentioned, covered the action of alkaline-earth metals, which would no doubt give similar results, but he had only worked with sodium. The condensation products of hydrogenated amines with aldehydes had only been studied in the case of formaldehyde and α -tetrahydro- β -naphthylamine. C. Smith (Chem. Soc. Trans., 1904, 85, 732) prepared methylene-tetrahydro- β -naphthylamine, a product which on reduction was converted into methyl-tetrahydro- β -naphthylamine. The condensation products of dihydro- and tetrahydro- α -naphthylamine with aldehydes were being examined now. The benzylidene derivatives of these amines could be obtained in large, beautifully formed crystals—a further example of the amines resembling *p*- rather than *o*-xylylene. The hydrogenated naphthalene hydrocarbons were soluble in alcohol, and it appeared probable that if a study of the series of compounds, naphthalene, the isomeric dihydronaphthalenes, tetrahydro, perhydro, and decahydronaphthalene were made, as had been suggested, there would be some stage in the hydrogenation at which the compound would burn without the deposition of soot. He had not

tried chromyl chloride as a reagent for the identification of the isomeric dihydronaphthalenes. There was no real difficulty in identifying these compounds as the dibromides, provided pure specimens of these compounds were available for comparison.

Newcastle Section.

Meeting held at Armstrong College on April 21, 1920.

PROFESSOR F. P. BEDSON IN THE CHAIR.

PECTINS IN VARIOUS PLANTS.

BY ARTHUR J. W. HORNBY, B.SC., A.I.C.

In an investigation of the substances not usually determined in the analysis of cattle foods, the pectins were found to vary greatly for different foods. Examination of fresh plants by Fellenberg's method also showed that the methyl pectate content of different parts of plants varied greatly.

Fellenberg has shown that various pectins yield 35–46% arabinose, 6–10% methyl-pentosan, 50% galactose, and 7–11½% methyl alcohol on hydrolysis with soda, and his method of estimation depends upon the scission of the methyl alcohol on treatment with dilute caustic soda solution (see J., 1917, 1190; also Chem. Zentr., 1916, i., 530, and Biochem. Zeits., 1918, 85, 118).

Fellenberg's method not only affords a method of estimating small quantities of methyl alcohol in the presence of ethyl alcohol, but gives very good comparisons of the methyl pectate content of various plants and furnishes data as to part of the hitherto undetermined constituents of food substances.

Thus, in the analysis of turnips, after the percentages of water, protein, oil, soluble carbohydrates, fibre, and ash have been determined there always remains about 2% of undetermined matter, and, as can be seen from the following tables, the pectin substances go far to make this up.

As some of the methyl compound is destroyed in the process of drying, the fresh material was used in later investigations, and the amount of dry matter determined separately at the same time. As will be seen from the tables, there is in every case much more in the epidermal tissue than in the cortex. Also, when plant substances were affected by insects or by mechanical injury to the tissues, the subsequent wound tissue contained more methyl pectate than normally; for example, when living potato was scabbed by mechanical means, such as cutting with a knife, more of these pectins were formed in the wounded tissue. Also exposure of the potato to light, as in sprouting, developed more pectin in the green epidermis and inner cortex than is found in the normal tuber.

It is highly probable that this pectin has a protective effect, especially against insect attack. Methyl alcohol seems to be a strong irritant to insects, and the ease with which methyl alcohol is split off from the pectin seems to indicate that it would be liberated by the digestive juices of the insect. This may explain the immunity of the parsnip against insect attack when compared with carrots or potatoes.

Different parts of the same root vary in pectin content; e.g., parsnip near the crown contains more than other parts of the root, in both the epidermis and cortex, just as the dry matter and sugar content also vary.

In the tables the figures given for methyl pectate in the dry matter and fresh material are average percentages for the whole, whole peel, or whole flesh, as the case may be, although the moisture in every part of the root under investigation was determined separately.

Root and part of root.	Remarks.	Variety.	No. of determinations.	Methyl pectate.		Dry matter %
				In fresh material, %	In dry matter, %	
<i>Parsnip</i>						
Peel ..	"Hollow Crown"		4	4.14	15.15	34.4
Flesh ..	Normal		4	1.96	11.76	16.8
Flesh ..	Boiled		1	1.80	11.24	16.8
Flesh ..	Tissue attacked by fly		1	5	20	25
<i>Carrot</i>						
Peel ..	Normal		12	2.35	11.6	19.5
Flesh ..	Normal		12	1.05	7.96	13.25
Wholeroot	Small, ill-grown and attacked by fly.		4	1.75	12.0	14.5
<i>Potato</i>						
Peel ..	Normal	"Ally"	12	0.83	4.15	20
Flesh ..	"	"	12	0.17	0.58	19
Peel ..	Green (exposed to light)	"	1	1.21	5.04	24
Flesh ..	Exposed to light	"	1	0.32	1.60	19
Peel ..	Mechanically injured	"	12	1.16	6.10	20
Whole ..	"Up-to-date"	"	12	0.45	1.64	27.26
<i>Garden sward</i>						
Peel ..			3	3.20	16.67	19.2
Flesh ..			4	0.96	6.66	14.41
<i>Soft turnip</i>						
Whole ..	Mixed		12	0.58	6.85	8.5
<i>Swede</i>						
Whole ..	Mixed "Up-to-date" & "Caledonian"		12	0.82	6.70	12.2
Whole ..	"Up-to-date"		4	1.18	9.08	13
<i>Garden beet</i>						
Peel ..			12	3.8	14.75	25.75
Flesh ..			12	1.25	10	12.5

	Number of determinations.	Percentage of methyl pectate in dry matter.
<i>Fruit peels—</i>		
Orange	12	25.3
Apple	12	17.3
<i>Leguminous seeds—</i>		
Bean spermoderm	1	6.34
Embryo	1	1.40
Earthnut spermoderm	1	6.56
Embryo	1	3.20
<i>Cakes—</i>		
Linseed	12	2.61
Palm kernel	12	0.27
Linseed chaff	1	2.47
Oat straw	3	0.80
Meadow hay	4	1.20
Malt	12	1.34
Bran	12	0.12
Ordinary cork	12	2.49

In conclusion, it may be suggested that a methyl compound might form an efficient spray against insect attacks, although at present the expense of such compounds seems to be prohibitive. These investigations are proceeding.

I have to thank Mr. Collins, of the Agricultural Department, Armstrong College, for his kind assistance and supervision in this work.

Communications.

PROPERTIES AFFECTING STRENGTH IN WHEATEN FLOUR.

BY F. J. MARTIN, M.A.

Strength of flour.—One of the greatest drawbacks in the study of wheaten flour has been the

multiplicity of definitions of "strength": the most recent of these, that of Humphries and Biffen (J. Agric. Sci., 1906, 1, 1), defines "strength" as the capacity of a flour to produce a large and well-piled loaf; it is certainly a statement of what the miller means by the term, and this definition is used throughout this paper. Strength, therefore, is obviously a quality or a combination of qualities, the measure of which can only be judged empirically, unless and until it is possible to give accurate and quantitative significance to the words "large and well piled." Empirical judging in the form of "Bakers' marks" is at present the only way of obtaining a final judgment as to the strength of a flour, and the object of the work recorded in the present paper was to find out whether flours of different "strengths" possessed any qualities which could be determined by chemical or physical means without a baking trial.

Effect of gluten on "strength."—A large amount of experimental work on "strength" in the past has been concerned with the amount and properties of the gluten. One of the earliest methods suggested for estimating strength consisted of washing away the starch and drying and weighing the residual gluten. Experience of this method, however, showed that the amount of gluten did not determine the baking quality, and therefore the cause of strength should be sought in the quality rather than the quantity of the gluten. Considerable advance was made by the investigations of Osborne (cf. J., 1895, 293), who showed that gluten must be regarded as consisting of two proteins, gliadin and glutenin, which could be separated by their difference in solubility in dilute alcohol, in which glutenin is insoluble. Applying this fact to the problem of strength, Fleurent (Comptes rend., 1896, 123, 755) examined a number of flours, and stated that the proportions of gliadin to glutenin in good gluten (i.e., from a strong flour) was as 3:1, and that any departure from these proportions was accompanied by a lowering in quality.

Working on similar lines, Guess (J. Amer. Chem. Soc., 1900, 263) concluded that both a high gluten content and high gliadin-glutenin ratio were associated with strength. It was found, however, by experience gained in actual baking, that these results were not sufficient to assess the "strength" of a flour, and later work by Fleurent (Comptes rend., 1901, 132, 1421), in which he determined the gliadin by a new and rapid method, showed that variations in gliadin content of different flours did not affect the "strength" to an extent appreciable for the purposes of the baker.

Effect of gas-producing capacity on "strength."—Wood (J. Agric. Sci., 1907, 2, 139) divided the factors affecting strength into two classes, viz., those affecting the size, and those affecting the shape of the loaf. He considered that the capacity of a flour for giving off gas when incubated with yeast and water was the factor which in the first instance determines the size of the loaf. Further work by Humphries and Simpson (7th Int. Cong. Appl. Chem.) suggested that it is the gas evolved in the latter stages of fermentation which is the more important factor.

Effect of mineral constituents on "strength."—Continuing his researches on "strength," Wood (J. Agric. Sci., 1907, 2, 293) drew attention to the effect of varying concentrations of acids and salts on the physical condition of the gluten, and suggested that the relation between the concentrations of acid and soluble salts in the flour is the factor on which the shape of the loaf depends. Subsequently ("Technology of Breadmaking," Jago, p. 323) Wood found that the salts having the greatest influence were water-soluble phosphates, and

that a high percentage of soluble phosphates was associated with good gluten. Hardy (Brit. Assoc. Reports, 1909) also dealt with the influence of electrolytes on the cohesive power of gluten. It may be remarked here, however, that it is doubtful whether the concentration of electrolytes in flour can affect practical baking results, as in normal breadmaking it is customary for the baker to add over 1% of salt to the flour, an amount many times in excess of the soluble mineral constituents naturally present in flour.

Effect of enzymes on "strength."—A considerable amount of work has been carried out in recent years on the amylolytic and proteolytic enzymes of flour (Baker and Hulton, J., 1908, 368; Ford and Guthrie, *ibid.*, 389). The chief points established having a bearing on the present paper are (1) that the presence of an amylolytic enzyme is necessary to provide the sugar required for yeast fermentation, and (2) that the presence of a proteolytic enzyme has distinct deleterious effect on the baking qualities of the flour.

EXPERIMENTAL.

Scope of experiments.—From the above resumé it will be seen that the following have been suggested as factors determining strength:—

- (a) The total amount of gluten.
- (b) The gliadin-glutenin ratio.
- (c) The amount of gas which can be obtained by fermentation.
- (d) The concentration of electrolytes, especially phosphates.

The present paper records the experiments carried out on all these points with a number of flours, together with observations on the gas-retaining capacity of the dough, the water-soluble proteins, and the baking marks.

In selecting flours for experimental work care was taken to avoid those which might have been treated previously by the miller with soluble diastatic, phosphoric, or ammonium compounds.

Effect of the gas-producing capacity on the strength of flour.—Previous work has shown the effect of gas-making capacity on the size of the loaf, and some attempts have been made to correlate size and gas-producing capacity. As it did not appear that size invariably corresponded to the gas produced, the following experiments were carried out. Six flours were selected and baked under laboratory conditions, the following amounts of materials being used in each case:—Flour, 750g., water 400g., salt 1.2%, yeast 1.0%. The doughs were made up and allowed to ferment for 4 hours, at the end of which time they were handled, weighed and placed in tins to prove, the proving period being 45 mins. The flours were then baked and the resulting loaves measured three hours after being removed from the oven. At the time of making up the doughs each was sampled and aliquot portions, enclosed in tubes connected with a gas-measuring apparatus, were incubated at 29° C., readings being taken to correspond to the proving period, and the total gas at 24 hrs. The results in Table I. show that neither the total gas nor the amount liberated during the proving period determines the size of the loaf:—

It appeared that five of the flours used were good gas-producers, and the sixth fairly good; it was therefore decided to select a flour with poor gas-making capacity and by the addition of diastatic reagents to increase the amount of gas produced in order to determine if this increase was reflected in the size of the loaf. A sample of North Russian flour was selected and baked under the conditions described for the previous experiment, the

TABLE I.

Ref. no. of flour.	Total gas, c.c.	Gas produced from com- mencement to oven, c.c.	Gas produced during proving period, c.c.	Volume of loaf, c.c.
6 A	479	136	47	1825
3 C	426	143	48	2600
2 C	422	152	48	2650
1 C	417	148	47	2600
3 A	407	142	46	1875
7 A	335	159	38	3075

same proportions of flour, water, etc., being used. The diastatic reagents added were pure diastase, and the proprietary preparations known as "Diastafor" and "G.E.₁₀." It will be seen from the figures given in Table II. that there was an increase in size in the case of each of the diastatic reagents added

From these experiments it is seen that whereas the total amount of gas does not permit a value to be assigned to the size of the resultant loaf, yet in

on the first rise eliminates any effects due to the rate of gassing.

The effect of the quantity of gluten present was first examined. A strong flour, containing over 12% gluten, was selected and diluted with calculated quantities of wheat starch so that the percentage of gluten present in six mixtures varied from 7.5% to 12.02%. The doughs were made up in the following proportions:—Flour and starch 20g., water 11g., salt 1.2%, yeast 1.0%. The doughs were then rammed down into measuring cylinders and incubated at 29° C., the gas given off during fermentation being collected over brine in another set of cylinders, and the volumes of the doughs and the amounts of gas produced were read at frequent intervals (Table III). Curves plotted to show the relation between the amounts of gas generated and the volumes of the doughs were fairly regular for the first part of the experiment, but erratic for the latter part, the irregularity coinciding with the appearance of holes in the surface of the doughs (the equivalent of over-proving in baking practice). Consideration of the doughs was therefore limited to that period preceding the appearance of the holes.

TABLE III.

Ref. no. of flour.	Percent- age of gluten.		Times at which readings were taken (minutes).									
			Start.	45	55	65	80	90	100	110	120	130
7 A (a)	7.5	Volumes of gas produced in c.c.	0	14	19	25	30	35	40	44	48	68
(b)	8.5		0	15	21	26	32	36	41	45	60	67
(c)	9.5		0	14	19	24	29	34	39	43	47	56
(d)	10.5		0	14	19	25	31	35	40	45	50	56
(e)	12.0		0	13	18	24	29	34	39	43	49	54
7 A (a)	7.5	Volumes of the doughs in the cylinders in c.c.	28	42	45	50	54	55	56	57	57	68
(b)	8.5		28	42	40	51	55	57	58	59	59	60
(c)	9.5		28	41	45	50	55	58	59	61	61	63
(d)	10.5		28	42	46	52	57	61	63	63	63	65
(e)	12.0		28	42	46	52	57	62	68	68	68	70

order to obtain a large loaf an adequate supply of gas must be available: a deficiency of gas can, however, be rectified by the addition of suitable diastatic preparations.

TABLE II.

Treatment.	Vol. of loaf laboratory baking.	Increase in size.	Bakers' marks (baked in bakehouse).
	c.c.	%	
Untreated	2100	—	78
0.1% Diastafor ..	2550	21	84
0.02% Diastase ..	2675	27	88
0.15% G.E. ₁₀ ..	2725	28	88

Effect of the amount of gluten present on gas-retention.—It being generally accepted that the figures relating to gas production are not sufficient to indicate the size of the loaf, the next factor considered was the capacity of a flour, when in the state of dough, to retain the gas produced.

In the following method of estimating the gas-retaining power of a flour the volumes attained by the doughs during the first rise (i.e., during the first 2 or 3 hours) was considered, since it was found by experience that the rate of gas production during this period was approximately the same in all cases. At later stages during the fermentation the rates of gas-producing vary greatly for the different flours and in some cases the amount of gas produced is not sufficient to distend the dough to its maximum size. The consideration of the volumes

Table III. shows that the rate of gassing was very even throughout the series of doughs, but that the volumes of the doughs varied considerably. It will be seen that the amount of gas retained by the various doughs, after the evolution of 45 c.c. in each case, varies with the percentage of gluten in the dough.

Variations in power of gas-retention of the gluters from different flours.—Attention has frequently been called to the variations in the physical properties of the gluters derived from different flours, and experiments were therefore made to see if there were any marked differences between the gas-retaining power of these gluters when fermented under similar conditions, the percentage of gluten in the flours being the same. A series of flours was taken, and after determining the amount of gluten present, the flours were diluted to a common gluten content of 7.0% by the addition of pure wheat starch. Doughs were then made up in the following proportions:—Flour and starch 20 g., water 10.5 g., salt 1.2%, yeast 1.0%. The doughs were rammed down into measuring cylinders and fermented at 29° C. as in the previous experiment. The amount of gas evolved and the volumes of the doughs were read at frequent intervals; the readings are given in Table IV.

The gas-retaining powers of the doughs are given in Table V., from which it will be seen that there is marked difference in the gas-retaining powers of the gluters from various sources.

Comparison of the chemical and physical properties of flour with baking properties.—A number

of factors having been suggested as having some effect on the strength of flour, it seemed possible that if an examination of the flour, embracing all these points, were made, some combination of factors might be found that would indicate the baking values of the flour. Straight grade flours from single wheats were selected for examination: these flours differed widely in baking properties and in origin, including wheats from England, Russia, India, Australia, and North America. The following determinations were made in each case:—

(1) *Total gluten*, determined by estimating the total nitrogen and multiplying by 5.7, also by washing out the gluten and drying at 100° C.

25° C., the mixture being thoroughly shaken at half-hour intervals. At the end of 3 hrs. the mixture was filtered, the filtrate evaporated to dryness in the water bath, and the residue dried at 100° C.

(7) *Water-soluble nitrogen*.—The nitrogen in the filtrate from No. 6 was estimated.

(8) *Water-soluble phosphorus*.—The phosphorus in the filtrate from No. 6 was estimated and taken as the soluble phosphorus expressed as P_2O_5 .

(9) *Acidity*.—5 g. of flour was mixed with 50 c.c. of water and the mixture immediately titrated with sodium hydroxide, phenolphthalein being used

TABLE IV.

Ref. no. of flour.		Times at which readings were taken (minutes).							
		Start.	60	70	80	90	100	110	120
9 A	Vol. of gas produced, c.c.	0	23	28	33	39	44	48	53
8 A		0	23	28	33	38	43	48	53
7 A		0	23	28	33	37	43	47	51
6 A		0	21	25	30	34	39	43	48
5 A		0	21	25	30	35	40	44	48
9 A	Volume of doughs, c.c.	25	45	49	51	52	52	54	55
8 A		25	44	49	53	56	58	58	57
7 A		25	43	48	52	55	55	56	56
6 A		25	42	46	48	49	50	50	50
5 A		25	40	44	46	46	46	47	48

(2) *Gas-retaining power of gluten*.—This was estimated empirically as described above, the flour being diluted with starch to a gluten content of 7%. In order to give a numerical value to this factor the gluten from No. 1 Northern Manitoban Flour (Ref. No. 7 A L) was taken as the standard, and the gas-retaining capacity of the gluten under examination is expressed as the ratio of the volume of its dough to the volume attained by the same weight of the standard under the same conditions. The ratio is, of course, a measure of the total volume attained and not of the increased volume due to gas-retention, but it has proved convenient as it is more closely related to the observations made in baking trials.

TABLE V.

Ref. no. of flour.	Volume of dough.	Gluten, %
5 A	46.0	7% in each case.
6 A	49.5	
9 A	51.5	
7 A	55.0	
8 A	57.5	

(3) *Gas-retaining power of flour*, estimated by multiplying the percentage of gluten in the flour by the gas-retaining power of the gluten.

(4) *Crude gliadin*.—The flour was treated with 50% alcohol (by weight) in a Soxhlet extractor, using the minimum quantity of liquid. The extraction was allowed to proceed until no further protein was extracted. Using 1–2 g. of flour it was found that all the alcohol-soluble protein was removed in 3 hrs. The nitrogen in the extract multiplied by 5.7 is taken as “crude gliadin.”

(5) *Glutenin*.—The crude gliadin-nitrogen subtracted from the total nitrogen was taken as the glutenin-nitrogen. This figure multiplied by 5.7 gave the amount of glutenin.

(6) *Water-soluble extract*.—25 g. of flour was treated with 250 c.c. of water for 3 hrs. at 24°—

as indicator: the result was expressed as g. H_2SO_4 per 100 g. flour.

(10) *Gas-producing capacity* was measured by the method suggested by Wood (*loc. cit.*). A dough was made up of 10 g. of flour and 5 g. of water, with 1.2% of salt and 1.0% of yeast, and fermented at 29° C. for 24 hours. The amount of CO_2 produced in that period was taken as the gas-producing capacity.

(11) *Baking properties*.—The flours were baked by a short, straight dough process, the fermentation period in the troughs being four hours. Two series of trials were made; in one series nothing was added to the flour, while in the other series enough diastase was added, where required, to render the flour able to produce sufficient carbon dioxide on fermentation. Each trial consisted of baking about ten “cottage” loaves and two “tin” loaves. Bakers’ marks were awarded by Mr. A. E. Humphries, under whose direction these trials were made. The standard to which these marks refer is that of No. 1 North Manitoban flour 100 and Average English 65. Under this system of marking a good household flour has a mark of about 84.

The results of the examination of the flours are given in Table VI. Attention is called to the difference in Bakers’ marks due to the addition of diastase only.

The figures in Table VI. indicate that there is no connexion between the gas-retaining power of the gluten and either the amount of water-soluble extract or water-soluble phosphorus or acidity.

Also, there is no definite relation between either the crude gliadin or glutenin, or the ratio of these, and the gas-retaining power of the gluten.

Extraction of water-soluble protein by dilute alcohol.—The work of Chamberlain (J. Amer. Chem. Soc., 1906, 1657) demonstrated the presence of proteins other than gliadin in the alcohol-soluble extract, and experiments were accordingly carried out to see if the water-soluble proteins were also soluble in 50% alcohol.

A flour was first extracted with 50% alcohol in order to estimate what has previously been termed in this paper "crude gliadin." Another sample of the flour was extracted with water by shaking vigorously for 5 min., centrifuging, and decanting through a filter. The residue was then washed

be due to their action on the water-insoluble proteins, converting these into a soluble form. Experiments were therefore made to see if the action assigned to the proteolytic enzymes was directed to transforming the gliadin or the glutenin into water-soluble proteins during the earlier stages of their

TABLE VI.

Ref. no. of flour.	Dry gluten (by washing). %	Gluten as total N $\times 5.7$ (A). %	Crude gliadin. %	Glutenin. %	Water-soluble proteins. %	Water-soluble extract. %	Water-soluble phosphorus. %	Acidity %	Gas-producing figure. c.c.	Gas-retaining power of gluten (B). %	Gas-retaining capacity of flour (= B \times A)	Bakers' marks Nothing added.	Diastase added.
8 A	12.65	12.09	5.99	6.10	1.45	4.76	-0.87	-111	142	0.93	11.75	90	100
7 A L	12.95	11.41	5.99	5.43	1.40	4.63	-0.85	-111	140	1.0	11.41	90	100
103	10.20	11.40	6.04	5.36	2.07	5.03	-132	-073	106	0.98	11.17	79	88
104	12.80	12.54	5.93	6.61	1.97	5.45	-104	-098	169	0.87	10.71	82	85
9 A	12.60	11.41	5.43	5.99	1.57	6.80	-184	-166	over 250	0.88	10.41	85	85
102	11.10	11.23	5.87	5.36	1.96	5.14	-066	-051	110	0.93	10.44	72	84
101	11.55	11.19	5.97	5.19	2.07	5.04	-072	-052	100	0.94	10.49	72	84
107	12.60	11.81	5.11	6.71	1.45	4.35	-184	-117	101	0.83	9.80	72	82
2 A	8.36	8.33	4.06	4.32	1.74	7.00	-212	-100	193	0.83	6.95	65	65
5 A	7.34	7.53	3.98	3.60	2.02	6.60	-226	-119	220	0.83	6.29	65	65

with water by shaking for 2 min., centrifuged, and decanted through a filter and the nitrogen estimated in the mixed filtrates. The whole of the water extraction was carried out in 15 min., the flour being separated from the solution by centrifuging, so that practically no flour was in contact with the water in the filter paper. The water-extracted flour was then extracted with 50% alcohol, and the nitrogen in the extract estimated. The results (Table VII.) show that 50% alcohol dissolves a considerable proportion of the water-soluble proteins:—

TABLE VII.

Material extracted.	Solvent.	Nitrogen. %
1. Flour S.	50% alcohol.	1.00
2. Flour S.	water.	0.21
3. Residue from 2 ..	50% alcohol.	0.80

Effect of time in the estimation of the water-soluble proteins.—Difficulties experienced in estimating the amount of protein soluble in water indicated that the length of time the flour was in contact with the water had an appreciable effect on the amount found, and the following experiment was therefore made in order to ascertain the quantitative significance of the period of extraction. A sample of Sudan flour was extracted for periods varying from 15 min. to 4 hrs., and the results (Table VIII.) show that the amount of soluble protein found increases with the period of extraction. This increase is thought to be due to the action of proteolytic enzymes, the presence of which have previously been demonstrated in some flours (Ford and Guthrie, *loc. cit.*).

TABLE VIII.

Time of extraction	Soluble protein, %.
15 mins.	1.02
1 hour	1.30
2 hours	1.34
3 hours	1.40
4 hours	1.45

Source of increase of water-soluble proteins.—Amended gliadin figure.—Previous workers have noted the presence of proteolytic enzymes in flour, and the increase in soluble proteins is thought to

action. A sample of the flour used in the previous experiment was therefore extracted with water at 30° C. for 4½ hrs., and after removing the water-soluble protein the residue was extracted with 50% alcohol. The results recorded in Table IX. indicate that the water-soluble proteins have increased at the expense of the alcohol-soluble proteins:—

TABLE IX.

Material extracted.	Solvent.	Duration of extraction.	Nitrogen. %
1. Flour S.	50% alcohol.	—	1.00
2. Flour S.	water.	4½ hrs. at 30° C.	0.42
3. Residue from 2 ..	50% alcohol.	—	0.54
4. Flour S.	water.	15 minutes.	0.21
5. Residue from 4 ..	50% alcohol.	—	0.80

Since it appeared that the greater proportion of the water-soluble protein was also soluble in 50% alcohol, and that the increase in the water-soluble proteins, formed by incubating flour and water, was accompanied by a corresponding decrease in the alcohol-soluble proteins, it was decided to amend the "Crude gliadin" figure by deducting from it the amount of protein found soluble in water after a long extraction period. The result obtained was called the "Amended gliadin" figure, and in the method used for determining the water-soluble protein the extraction lasted 3 hrs.

Relation of "amended gliadin"—glutenin ratio to gas-retaining power of gluten.—The "amended gliadin" figure was estimated for the series of flours previously dealt with, and the ratio of the "amended gliadin" figure to the glutenin calculated. The results, compared with the figures obtained in the cylinder experiments for gas-retaining capacity (Table X), show that there is a fairly close agreement between the amended gliadin-glutenin ratio and the gas-retaining power of the gluten, a high ratio corresponding to a good gluten.

Comparison between "amended gliadin" figure and bakers' marks.—Since the gas-retaining capacity of a flour is correlated with the "amended gliadin"—glutenin ratio, and also with the amount of gluten present, it was thought that the "amended gliadin" figure would give a good indication of the size of the loaf. Table XI. shows that there is a close agreement between the percentage of "amended gliadin" and the bakers' marks (diastase added where required).

TABLE X.

Ref. no. of flour.	Alcohol-soluble nitrogen %	Water-soluble nitrogen %	Amended gliadin nitrogen %	Glutenin nitrogen %	Amended gliadin-glutenin ratio.	Gas-retaining power of gluten.
7 A L	1.050	0.246	0.804	0.952	0.843	1.00
8 A	1.051	0.255	0.796	1.071	0.744	0.98
103	1.060	0.365	0.695	0.910	0.739	0.98
101	1.049	0.361	0.685	0.911	0.751	0.91
102	1.030	0.344	0.686	0.910	0.720	0.93
9 A	0.952	0.275	0.677	1.050	0.644	0.88
104	1.040	0.346	0.694	1.160	0.598	0.87
107	0.896	0.254	0.642	1.176	0.515	0.83
2 A	0.712	0.309	0.403	0.748	0.539	0.83
5 A	0.698	0.355	0.343	0.632	0.543	0.83

TABLE XI.

Ref. no. of flour.	Amended gliadin figure.	Bakers' marks.
7 A L	4.58	100
8 A	4.54	100
103	3.96	88
104	3.96	85
9 A	3.86	85
102	3.92	84
101	3.91	84
107	3.66	82
2 A	2.30	65
5 A	1.95	65

Combination of gas-producing and gas-retaining capacity.—Both these factors exert a great influence on the resulting loaf, and an ordinary baking trial does not indicate either the capacity of gas-production or the power of gas-retention. It is, however, a simple matter to estimate the gas-producing capacity of a flour, and where deficient to increase it by the addition of a suitable diastatic reagent; when this is done, the baking of the resulting flour demonstrates the capacity of the flour to retain the gas liberated within the dough.

The series of flours already dealt with were baked under these conditions, and the gas-retaining capacity and "amended-gliadin" figure compared with the bakers' marks (Table XII.). It will be seen that these three sets of figures are closely correlated. In the same table the figures for gas-production, the amount of diastase added in the form of "diastafor," and the calculated gas-producing capacity as the result of adding diastase are given, in order to show how the absence of gas-producing power is overcome.

TABLE XII.

Ref. no. of flour.	Original gas-producing capacity.	Diastase added (= % diastafor).	Calc. gas-producing capacity. Diastase added (approx.)	Gas-retaining capacity of flour.	Amended gliadin %	Bakers' marks.
7 A L	140	0.1%	200	11.41	4.58	100
8 A	142	0.1%	200	11.75	4.54	100
103	106	0.2%	200	11.17	3.96	88
104	160	0.05%	200	10.71	3.96	85
9 A	over 250	nil	over 250	10.41	3.86	85
102	110	0.2%	200	10.44	3.92	84
101	109	0.2%	200	10.49	3.91	84
107	101	0.2%	200	9.8	3.66	82
2 A	193	nil	193	6.95	2.30	65
5 A	220	nil	220	6.20	1.95	65

SUMMARY.

1. An investigation has been made as to the relations between various properties of flour and "strength."

2. No correlation appears to exist between strength and the amounts of total soluble extract, soluble phosphorus, or acidity.

3. A strong flour must possess a *minimum gas-producing capacity* as measured by the amount of gas produced by fermentation during 24 hours. A deficiency in this respect can be rectified by the addition of an amylolytic enzyme, e.g., a diastase preparation.

4. A strong flour possesses a *high gas-retaining capacity*. This has been shown to be due to the amount and form of the proteins in the flour.

5. The water-soluble protein increases with the length of the period of extraction, probably due to proteolytic enzyme action, at the expense of the alcohol-soluble protein.

6. In estimating the gliadin present in flour it is necessary to make allowance for the water-soluble proteins, which are soluble to a great extent in dilute alcohol.

7. Flours with high gas-retaining capacities and high bakers' marks have been shown to be those in which the "amended gliadin" figure is also high.

8. For flours having a satisfactory gas-producing capacity (see 3), bakers' marks, gas-retaining capacity, and "amended gliadin" content are closely related, and it is considered that the estimation of either of the latter together with the determination of the gas-producing capacity will indicate the "strength" of the flour.

The author, in conclusion, wishes to express his indebtedness and thanks to Mr. A. E. Humphries for permission to make use of figures obtained while working at Coxes Lock Mill, Weybridge, and to Dr. A. F. Joseph for assistance in preparing this paper.

Wellcome Tropical Research Laboratories.
Khartoum, March, 1920.

THE PRESERVATION OF VULCANISED RUBBER.

BY HENRY P. STEVENS.

Some time ago I was informed that vulcanised rubber kept in a tin box over a layer of ordinary kerosene remains in a serviceable condition for a much longer period than if kept in air. I therefore determined to test the preservative effect of kerosene and other vapours. The results confirmed the claims made for kerosene vapour and also showed that water vapour has a similar or even more marked effect.

For the experiments I used various vulcanised mixings to hand, consisting of plantation crêpe or smoked sheet (90 parts) and sulphur (10 parts). I had previously investigated the effect of ageing in air on vulcanised rubber rings of this composition (J., 1916, 872; 1918, 305 T and 341 T), so that I was in a good position to note the effect of ageing under varying conditions.

Preliminary tests having shown that kerosene vapour had a marked preservative effect, experiments were made with a number of rings kept in small desiccators over kerosene in a dark cupboard. The controls were placed in an empty desiccator to protect them from laboratory fumes. The experiment was extended to include some rings placed in moist air with a layer of water instead of kerosene at the bottom of the desiccator, and other rings in dry air over calcium chloride. Rings were cut from two vulcanised rubbers, A and B; A with a coefficient of 3.02, that is to say, cured to give a

reasonably long life under ordinary conditions and likely to improve on keeping for a few months, and B with a coefficient of 4.50—that is to say, over-cured and likely to show an appreciable deterioration in a few months.

Rings from both rubbers serving as controls were also tested at the commencement of the ageing period (6½ months).

Sample.	Controls, tested 27/3/18. (1)	Aged specimens, tested 19/11/18, preserved in			
		(2) laboratory air.	(3) air saturated with moisture	(4) dry air.	(5) over kerosene.
A. Breaking strain, g. per sq. mm. Final length: original=1 ..	1450 10.69	1510 9.77	1670 9.79	1360 9.34	1670 10.30
B. Breaking strain, g. per sq. mm. Final length: original=1 ..	1740 9.51	1430 8.33	1600 8.71	180 2.79	1525 9.11

The specimens kept over kerosene were swollen and were therefore hung in air before testing. They still retained a little petroleum when tested, which probably accounts for the rather high figures for final length as compared with the remaining specimens.

The figures demonstrate the preservative action of water and kerosene vapours, both of which appear to be effective. With sample A improvement results with ageing; with sample B there is some falling off in breaking strain, but it is small. Dry air gives the lowest figures, and in sample B the rubber is perished. Laboratory air contains some moisture, and the figures are consequently intermediate between dry air and air saturated with moisture. The contrast between the supple rings aged over moist air or kerosene and the harsh feel of those aged in dry air is very striking.

The preservative effect of moisture in the air is a matter of great interest and not previously remarked on or demonstrated.* It is quite common to find rubber goods stored in a dry place, whereas a damp, cool cellar would be preferable. The results also have a bearing on physical tests made on vulcanised rubber in the East. I have already shown that temperature is an important factor in ageing (J., 1918, 281 r), and have suggested that the test rings should be kept in an incubator at the average temperature of the tropics during the period between vulcanising and testing, so as to enable tests to be made in Europe to correspond with conditions in the tropics. It now appears that the atmosphere surrounding the test pieces in the incubator should be kept moist. Similar con-

* Since the above was written I have been informed that rubber stoppers have been preserved in water for 30 years. Although the action may be the same, immersion in water is not so generally applicable to rubber goods as preservation in a moist atmosphere.

siderations apply to the so-called "accelerated ageing test," in which the specimens are heated in an oven to a temperature not exceeding 70° C. The results will differ from similar tests in a moist atmosphere or ageing tests at room temperature.

Further experiments were made in which the rings were suspended in stoppered bottles placed in an incubator at 30° C. to bring the temperature nearer that in the tropics. An attempt was made to substitute low-boiling petroleum for kerosene, as the vapour of the latter contains relatively high-boiling constituents which are difficult to remove from the rubber when once absorbed. Unfortunately, the more volatile petroleum vapour escaped from the stoppered bottle in a week or two at the elevated temperature. To remove the high-boiling constituents from the rubber the rings were soaked in low-boiling petroleum and air-dried before testing. The specimen of rubber taken was over-cured, having a coefficient of vulcanisation=4.79.

	Control tested 3/1/19.	Aged specimens tested 2/7/19.	
		in air saturated with moisture.	in kerosene vapour.
Breaking strain, g. per sq. mm.	1650	1330	840
Final length: original=1	9.89	8.45	9.08

In this experiment air saturated with moisture appears to have a greater preservative action than the kerosene vapour. As, however, the more volatile constituents of the kerosene vaporise and escape during the first few weeks of the treatment, the air in the later stages of the experiment will not be so fully charged with kerosene vapour as at the commencement. The kerosene-preserved rings were soft and supple although weak and partly perished. The kerosene vapours absorbed appear to counteract the harsh feel of rubber perished in the air. The soft and supple feel of kerosene-preserved rubber is not therefore to be taken as indicating that perishing has not taken place.

When vulcanised rubber ages only a small increase is shown in the coefficient, provided the rubber is not considerably over-cured.† It was of

† The present communication was written before the publication of Eaton and Day's paper entitled "Ageing experiments on vulcanised plantation rubber" (J., 1919, 339-347r). These authors have also noted the increase in combined sulphur which takes place on ageing and are apparently under the impression that they were the first to show definitely that such increase takes place. This, however, was already a well-ascertained fact (cf. Hinrichsen, Koll. Zeits., 1911, 8, 245; Speace, Koll. Zeits., 1912, 10, 290, and 1912, 11, 28; Van Rossem, Netherland Govt. Inst. Comm., Pt. 6, p. 219, as well as numerous determinations of my own, J., 1918, 305-306r and 340-342r, in the latter of which a correlation between the value of the coefficient and the amount of increase during ageing under uniform conditions is revealed). Eaton and Day also refer to the formation in one case of a volatile "sulphide." I had previously noted the formation of a volatile sulphur compound in all cases where a thoroughly perished acetone-extracted rubber was kept in a dry atmosphere (J., 1919, 192-196r) and I gave some account of its reactions and properties which are being further examined.

Control tested 12/12/18.				Aged specimens, tested 2/7/19.							
(1)				(2) Dry air.		(3) Air saturated with moisture.		(4) Kerosene.			
				C.	D.	C.	D.	C.	D.		
Breaking strain, g. per sq. mm.				1540	1610	quite perished	quite perished	1950	1600	Too weak to give a figure	60
Final length: original=1				9.21	9.08			8.60	8.90	2.34	4.18
Coefficient calc. on weight before extraction.				5.53	5.48	6.92	6.38	5.38	5.20	5.20	4.71
Do., on weight after extraction				—	—	7.49	6.85	5.41	5.24	6.13	5.27
Loss on acetone extraction, %				—	—	10.5	10.6	5.60	5.9	8.8	15.3

interest to ascertain whether this increase is influenced by the medium surrounding the specimen. As one could not be certain of removing the remains of petroleum from the specimens preserved over kerosene, all specimens were weighed both before and after acetone extraction, and the coefficient calculated on the weight of both the original and extracted rubbers. In the figures below the percentage loss on acetone extraction is also given. The vulcanised rubbers used were both appreciably over-cured, with coefficients C=5.53 and D=5.48. The rings were hung in bottles as before in an incubator at 30° C. The air in one bottle was kept dry by sticks of caustic potash.

It is probable that the deterioration in kerosene vapour resulted, as before, from the loss of the volatile constituents.

The loss on extraction of the moist air samples (3) only slightly exceeded the free sulphur, so that practically nothing but free sulphur and so-called resinous matter present in the original rubber was extracted by the acetone. On the other hand, both of the dry-air perished samples (2) lost about 10½%, which, after subtraction of the free sulphur, leaves 7–8% of vulcanised rubber extracted and dissolved out by the acetone.

In a previous paper (J., 1919, 1917) I showed that vulcanised rubber gradually becomes more soluble in benzene the more it is perished; eventually it becomes soluble in acetone also. As part of the perished vulcanised rubber specimens C and D under (2) dissolves in acetone, together with the sulphur with which it is combined, it follows that the coefficient, if calculated in the usual manner on the original weight, will be too low, and the correct figure will be a higher one, obtained by basing the calculation on the weight of the specimen after acetone extraction. This increase in the coefficient amounts to about ½% in the above examples. The following figures were obtained by analysing a number of specimens vulcanised to varying degrees, weighing the rubber both before and after acetone extraction.

Coefficient calc. on weight before acetone extraction.	Coefficient calc. on weight after acetone extraction.	Loss by acetone extraction, % by wt.	Free sulphur extracted (by difference), %.	Matter extracted other than free sulphur, %.
Specimens in good condition analysed shortly after vulcanisation.				
1.31	1.37	12.3	8.8	3.5
1.39	1.45	12.6	8.8	3.8
1.40	1.45	11.6	8.7	2.9
1.44	1.51	12.2	8.7	3.5
1.78	1.84	11.5	8.4	3.1
3.07	3.17	10.0	7.2	3.1
3.41	3.60	10.1	6.9	3.2
4.23	4.38	9.2	6.2	3.0
4.50	4.75	8.8	5.9	2.9
5.58	5.74	7.7	5.0	2.7
6.44	6.70	7.6	4.2	3.4
7.28	7.54	6.7	3.5	3.4
Specimens analysed when aged for varying periods and perished.				
5.43	12.60	56.3	5.1	51.2
6.37	9.32	32.7	4.3	28.4
6.38	6.85	10.6	4.3	6.3
6.02	7.49	10.5	3.8	6.7
7.29	9.04	20.8	3.7	17.1

The figures in the last column show that newly vulcanised rubber yields about 3% of acetone extract in addition to the free sulphur, whatever the coefficient may be, but that, after perishing, the extract may attain any figure. It may be concluded that previous published figures for the combined sulphur or coefficient of rubber after ageing, including my own (J, 1918, 305–6 T and 340 T), are too low for those specimens which were perished.

From the above results, particularly the constancy of the acetone extracts after ageing, it appears that the preservative action of water or kerosene vapour is due to an actual chemical preservation of the vulcanised rubber. These agencies inhibit or retard the chemical changes, including oxidation of the rubber, which normally take place when vulcanised rubber is aged in air, as shown by increase in weight and increase in acetone extract, but they do not retard the physical changes sometimes known as “after-vulcanisation,” as shown by the tensile strength and reduction of distensibility (reduced final strength) of the rings kept in air saturated with water vapour. We may therefore distinguish two changes which normally take place when vulcanised rubber ages:—Firstly, a physical change comprising an initial increase in tensile strength (if the specimen is not appreciably over-cured), and a gradual reduction in final length.

Secondly, a chemical deterioration, consisting mainly in an oxidation with a slight loss of sulphur in a volatile form. The extent of the chemical change is conditioned (1) by the coefficient of vulcanisation, the higher the coefficient the more rapid the oxidation, (2) by the atmosphere surrounding the specimen, and (3) by the temperature. I do not propose at this stage to put forward a theory to account for the preservative action of the water vapour, as further experiments to this end are in progress.

Conclusions.

1. The life of vulcanised rubber is prolonged by storing in air saturated with moisture or petroleum vapour. Even over-cured rubber can be preserved by this means for 6 or 7 months at tropical temperatures.
2. Preserved under these conditions, the acetone extract does not increase, showing that the rubber is protected from oxidation and decomposition. Nevertheless, the physical changes characteristic of “after-vulcanisation” proceed normally.
3. In dry air the chemical change (oxidation) takes place more rapidly than in air containing moisture, and an increase in the acetone extract takes place.
4. As the oxidation of soft vulcanised rubber takes place the more rapidly the higher the coefficient, and is accompanied by an increase in the coefficient, and as vulcanised rubber, when perished, tends to become soluble in acetone, the percentage of combined sulphur or coefficient in such cases should be based on the weight of the specimen after acetone extraction.

THE ESTIMATION OF NICKEL IN STEELS BY DIRECT TITRATION.

BY EDWARD H. HALL, A.I.C., B.SC.(LOND.).

The method to be described is a modification of the well-known one of the direct titration of the tartaric (or citric) acid solution of the steel by potassium cyanide.

It has long been recognised that provided chromium be absent (see Brearley and Ibbotson) the method will give results sufficiently accurate for routine work, but cannot be used where great accuracy is required. The reason usually given for this lack of precision is the difficulty of observing to a nicety, owing to the dark colour of the solutions being titrated, the point where the cloudiness produced by the silver iodide indicator is re-formed

by the silver nitrate after being first discharged by the standard potassium cyanide solution.

According to the author's experience of the method, a further difficulty is that of neutralising the tartaric acid solution of the steel before titration. The usual method of neutralising this solution is to add dilute ammonia until the solution smells faintly ammoniacal. The danger of this procedure is that the flask in which the neutralisation is made often smells ammoniacal before the solution is neutralised, and it is necessary, before smelling, to drive ammoniacal vapours from the flask. This is rather tedious and, moreover, still leaves a doubt as to the exact excess of ammonia added. The amount of this excess has a great influence on the result. It is therefore important when titrating a batch of steels against one of known nickel content or against a standard nickel solution, that precisely the same excess be used in each case. The actual magnitude of this excess, provided it be low, does not matter.

In order to make the method precise, therefore, it will be seen that the following conditions are necessary:—

(1) That a suitable means be found for viewing the appearance and disappearance of the cloudiness produced by the silver iodide indicator.

(2) That a means be found to control exactly the excess of ammonia added before titration.

The first condition is easily satisfied by transferring the tartaric acid solution of the steel before complete neutralisation to a Nessler tube (or a measuring cylinder), which is supported over a plane mirror by any suitable means. The angle of the mirror is so adjusted that light is reflected directly up the length of the tube. By looking vertically down the tube, the slightest cloudiness is apparent, even when the solution looks perfectly bright when viewed normally. The mirror can be conveniently mounted on a swivel (a microscope illuminating mirror answers the purpose admirably), so as to be able to adjust its angle, and can then be placed in a small wooden box, having one side removed to admit the light, and a hole in the top to let the reflected light through. The contents of the Nessler tube are stirred by a plunger made of thin glass rod. The second condition is met by making use of the fact that silver chloride is soluble in ammonia, and therefore a cloudiness produced by it will be discharged as soon as free ammonia is present. The application of this fact to the present problem will be apparent by following the complete method given below.

The procedure is as follows:—0.5 g. of the sample is dissolved in a 250 c.c. conical flask in 10 c.c. of concentrated hydrochloric acid, and when solution is complete 2 c.c. of concentrated nitric acid is added and the flask replaced on the water

bath to finish the oxidation. 35 c.c. of hot distilled water is then added, the flask placed on a hot plate, and the solution boiled vigorously for two minutes to expel nitrous fumes, and 12 c.c. of a solution containing 100 g. of tartaric acid per 200 c.c. is added, followed by 8 c.c. of 0.880 ammonia. The iron solution should still be acid after adding the ammonia. The flask is now cooled and the contents transferred to the Nessler cylinder or measuring cylinder, which should hold at least 150 c.c. From a burette about 2 c.c. of the standard silver nitrate solution (2 g. per l.) is run in; this will produce a turbidity of silver chloride. From a second burette dilute ammonia (sp. gr. 0.956) is run in, at the same time thoroughly stirring with the plunger, until this turbidity disappears. The neutral point is now reached. From this point an excess of 1.0 c.c. ammonia is added and then 2 c.c. of a 2% potassium iodide solution. This restores the turbidity, which is due in this case to silver iodide, which is not soluble in ammonia. The turbidity is now discharged by running from a third burette standard potassium cyanide solution (6.7 g. KCN and 1 g. KOH per l.) in slight excess, and again formed by cautiously adding the standard silver nitrate. The volumes of potassium cyanide and of silver nitrate added (including the original 2 c.c.) are noted, and from these figures the percentage of nickel in the sample is computed.

The potassium cyanide solution is best standardised by running the solutions against a steel of known nickel content; if this be not possible, after obtaining the end point of an actual estimation as described above, a known amount of a standard nickel solution is added, and the liquid again titrated. This nickel will have been estimated under almost the same conditions as that in the sample.

Having obtained the "end point" when working with a steel, a further 4 or 5 c.c. of potassium cyanide solution is run into the cylinder and the turbidity re-formed by adding silver nitrate. The quantities of cyanide and nitrate show the relationship under the same conditions as these operating in the original estimation.

The delivery stem of burette has an extension, bent twice at right angles, fused to it below the tap. This avoids the burette being directly over the Nessler tube, which would prevent it being viewed from above.

Duplicate results agree to three significant figures. For example, with one steel basic acetate separation followed by titration showed 3.42% Ni, whilst the new direct titration method gave 3.415, 3.417, 3.419%. With another steel the result with the basic acetate separation process was 3.93%, and with the direct titration process 3.924, 3.927, and 3.922%. The method embodying the modification described is very accurate and extremely rapid.

Annual Meeting.

CHEMICAL SHEET LEAD.

BY D. W. JONES.

(See J., 1920, 221—224 T.)

The author writes as follows in reply to Mr. Lancaster's communication to the discussion (*loc. cit.*, p. 224 T.):—

Mr. Lancaster questions the certainty that the additions of impurities existed in the quantities stated in the tables; and, secondly, mentions the difficulty encountered in introducing as much as 0.38% copper into a pure lead.

In reply I can state that in all cases the amounts of impurities contained in the lead were proved by careful analysis.

I am in agreement with Mr. Lancaster when he states that it is a simple matter to introduce a true equivalent of a metal of low melting point into lead, but variations arise when additions of copper are made to lead. In my experience the best results are obtained by preparing a mixture of 5% copper, 95% lead, by first melting the copper and afterwards adding the lead with constant stirring. This strong copper mixture is then added to a pure "chemical" or Pattinson lead, when a much lower temperature can be employed than in the first melting, thus obviating the liquation effect referred to by Mr. Lancaster. By this means it is possible to produce sheet lead containing 0.38% copper.

ERRATA.

Page 224 T, col. 2, lines 3—5, for "His experience . . . atmospheric corrosion," read "Lead stored in the open became severely carbonated in a short time after heavy rains, but this was not the case with lead containing perhaps 0.5% Cu."

BY-PRODUCTS FROM COKE-OVENS.

BY E. W. SMITH.

(J., 1920, 194—197 T.)

ERRATA.

p. 195 T, col. 1, line 41 from bottom, for "all cases" read "many cases."

p. 195 T, col. 2, line 19 from top, for "quality" read "gravity."

p. 195 T, col. 2, line 19 from bottom, for "40%" read "50%."

p. 196 T, col. 1, line 29 from bottom, for "100 cb. ft." read "1000 cb. ft."

p. 196 T, col. 2, line 34, after "gaseous, B.Th.U." insert "This may amount to as much as 1s. per gallon of motor spirit."

A NEW PROCESS OF CENTRIFUGAL FILTRATION.

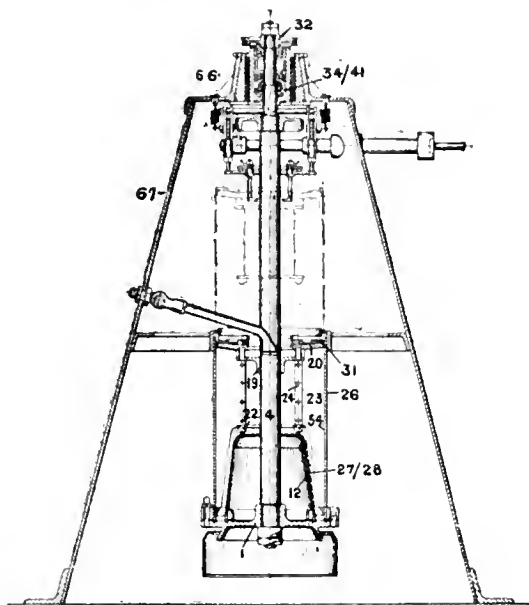
BY W. J. GEE.

(Abridged.)

It is possible completely to remove suspended solids from a liquid, however fine the particles, in a solid drum machine by centrifugal force exerted over a sufficient time. But in many cases the last few grains per gallon of exceptionally fine particles require so much time that the output of the apparatus is severely limited. I have myself found that some 98 to 99% of the total solids could be recovered at a very high rate of feed to the machine, but to recover the other 1 or 2% the feed had to be reduced beyond what would be a commercially prac-

ticable limit. In one case, to obtain a perfectly clear effluent, I had to cut down the feed to one-thirtieth of the rate at which 98.5% was recovered.

Consequently, in cases where the liquid is required to be completely clarified, it was necessary to evolve some method of dealing with the finer particles with an efficiency more nearly approaching that at which the larger solids are dealt with by centrifugal force. The possibility of using a filter for this purpose, using the pressure derived from centrifugal force, was obvious, but a peripheral filter would readily choke, and bring one back speedily to imperforate drum conditions. Fortunately I had some prior experience which led me to consider the possibility of an exit flow under a centrifugal head, if I may use the expression, and I found in this a complete solution of the choking difficulty. The final form of the apparatus, which combines centrifugal separation with filtration through a non-choking filter, is illustrated in the accompanying sectional diagram. The drum (26)



is secured to the base (1) by a bayonet-jointed locking-ring, the base being attached to a spindle (4) suspended from a patent ball-bearing. Rotation is imparted by the pulley (32). The bearing (34/41) is supported by a conical pedestal (67), to which is riveted a top casting of steel (66). The pulley (not shown) on the main driving shaft is fitted with a clutch of centrifugal friction type, which provides means for controlling the machine. The upper end of the drum (26) is closed by a top plate (20), which makes, during rotation, a watertight joint (31). This plate is provided with a central opening through which the machine is charged; it is also held centrally on the spindle by a distributing plate (19). Depending from the top plate (20) is the container, consisting of six vertical rods (24), with attached vanes or blades (23). These extend the whole length of the drum, being connected to the top plate (20), middle plate (22), and bottom plate (25). They serve the double purpose of dividing the drum into six longitudinal compartments, and compelling the liquid under treatment to rotate with the drum, so avoiding slip.

Each compartment is provided with a thin residuum plate (54), forming a removable lining,

curved to the radius of the drum. Upon these plates the recovered solids are deposited. When fully charged the container is lifted into the position shown in dotted lines. The recovered solids may then be removed without disturbance. A duplicate set of plates is supplied with each machine, and when the charged plates are removed the other set is inserted, and the machine put again into operation without delay. The charged plates are dealt with whilst the new charge is accumulating. Fitted to the base (1) is a perforated filter cone (12), projecting into the separator drum (26). A filter medium, cloth, paper, or other suitable substance, is wound round and secured to the outside of the filter cone by screwed wire clamps (27/28). The lifting of the container in order to remove the recovered solids is effected by a screw and bevel gear operated by cone clutches, connected to a continuously running counter-shaft, belt driven from the main power shaft. A nut carrying levers is connected by wire ropes to a ball-bearing turntable, provided with means of attachment to the top plate (20) of the container. This turntable allows the container, when lifted, to be easily rotated for access to the charge.

The new centrifugal filter has the following advantages over other filtering apparatus:—

1. The rate of filtration is constant throughout the charging period.

2. The filter, once made, is used over and over again; the removal of the recovered solids does not disturb it.

3. A very wide choice of filter material is available. Such materials as filter paper, asbestos, glass cloth, and so on can be used, as bursting of filter material is eliminated.

4. The removal of the recovered solids requires much less time than the cleaning of a filter press and the fitting of clean filter cloths.

5. The wear and tear of filter material is almost completely eliminated.

6. Materials which cannot at present be filtered at all commercially in filter presses, owing to choking, can be dealt with in the centrifugal apparatus without difficulty.

7. The apparatus lends itself to heating or cooling, so that materials may be treated at any required temperature.

8. The rotor can be readily enclosed completely for the filtration of volatile or dangerous liquids, which may be treated at any pressure or temperature. In recovering suspended solids from volatile solvents, loss of solvent and danger of fire or explosion are eliminated by using a totally enclosed machine.

9. The recovered solids are graded as to fineness of particle in a very exact manner, which is unvarying on repetition, so that standard qualities of materials are assured.

London Section.

Meeting held at Burlington House, on May 3, 1920.

MR. JULIAN L. BAKER IN THE CHAIR.

THE PREPARATION OF THIOCARBONYL TETRACHLORIDE.

BY PERCY FARADAY FRANKLAND, FREDERICK CHALLENGER, AND DOROTHY WEBSTER.

Some time ago it was found desirable to determine the most suitable methods for the preparation of thiocarbonyl tetrachloride, CSCl_4 , and thio-

phosgene, CSCl_2 , from carbon bisulphide. A thorough examination of the literature revealed the almost complete absence of reliable information concerning the yields to be expected, although both these substances have found application in the manufacture of dyestuffs, their use in this manner being protected by the German Patents 234,922, 232,791, 232,792, and 232,793 (Meister, Lucius, und Brünig).

As our experiments were intended as a preliminary to the manufacture of thiocarbonyl tetrachloride on a commercial scale, it was of first importance to determine what materials might safely be used in the construction of the chlorination vessel, as well as the effect on the reaction of traces of other substances. Our experiments were always carried out in glass vessels. A superficial examination of the chlorination of carbon bisulphide in the absence of any catalyst showed that no CSCl_4 and very little carbon tetrachloride was formed. In the presence of a very small amount of iron filings carbon tetrachloride was practically the only product, while with iodine as the only foreign substance present good yields of thiocarbonyl tetrachloride were obtained. The simultaneous presence of traces of iron filings, however, completely nullified the effect of the iodine, no CSCl_4 being formed. On the other hand, pieces of sheet lead exerted no harmful influence on the activity of the iodine.

Klason (G.P. 83,124) recommends the use of a lead-lined iron pan for the preparation of CSCl_4 , but does not explain the necessity for such an arrangement. Urbain (English Patent 13,733 of 1901) states that the addition of 0.5% of iron to the completely chlorinated mixture destroys any CSCl_4 , which is thereby converted into carbon tetrachloride and sulphur monochloride. In this connexion, Mr. F. H. Garner made several experiments which clearly showed that pure thiocarbonyl tetrachloride when kept in contact with metallic iron at the ordinary temperature is completely decomposed.

A few experiments were made in order to determine the effect of metallic tin and of "copper bronze" on the chlorination in presence of iodine. In the first case very little CSCl_4 was obtained, most of the product distilling below 80° and consisting of carbon tetrachloride. The effect of tin appears therefore to be similar to that of iron. "Copper bronze" seems to have much less effect on the activity of the iodine, a yield of CSCl_4 equal to 48% of the theoretical being obtained.

Klason (*loc. cit.*) has patented a method according to which a considerable improvement may be effected in the preparation of thiocarbonyl tetrachloride. This consists in treating carbon bisulphide in presence of iodine with six atomic proportions of chlorine instead of five, whereby the sulphur monochloride (b.p. 138°) is converted into the more volatile sulphur dichloride before the thiocarbonyl tetrachloride is attacked. The sulphur dichloride may then be removed by distillation. We have only carried out one experiment on these lines. The reaction mixture was distilled at atmospheric pressure and two main fractions obtained. The first (A) b.p. up to 80°C. , was found to contain large quantities of sulphur chlorides and practically no CS_2 , CSCl_2 , or CSCl_4 . The large fraction above 80°C. (B) contained about 26% of sulphur chlorides, judging from the loss when an aliquot part was submitted to distillation with steam.

On distillation of a further portion of B it was found that the fraction boiling above 140°C. still contained about 20% sulphur chloride. This was obviously due to decomposition of the SCl_2 during distillation into S_2Cl_2 and free chlorine and indicated the necessity for the distillation of the dichloride under diminished pressure. After removal of sulphur chloride from B the total yield of CSCl_4 was 50%.

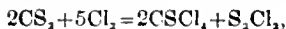
Klason gives no details concerning the yield obtained by this method, but our results certainly confirm his statement that preferential action of the extra atom of chlorine on the sulphur monochloride occurs.

Preparation of thiocarbonyl tetrachloride on the large scale.

After several preliminary experiments with smaller quantities it was found that the method now to be described could be relied upon to give a 60% yield of CSCl_4 calculated on the weight of the carbon bisulphide taken.

To about 2 kg. of carbon bisulphide, dried over calcium chloride, is added 4 g. of iodine. Chlorine (from a cylinder), dried over calcium chloride, is introduced at such a rate that the temperature is maintained at about 20°C . and almost the whole of the gas is absorbed. It is usually desirable to cool the glass vessel containing the bisulphide in cold water, except at the commencement of the reaction, when the temperature may be allowed to rise to about 25° before cooling is begun.

When the increase in weight corresponds to the absorption of five atomic proportions of chlorine, according to the equation



the introduction of chlorine is stopped and the reaction mixture is run in a thin stream into hot water contained in a large earthenware jar, into which a rapid current of steam is blown. By this means the sulphur chloride is decomposed, while the CSCl_4 distils over. Owing to the large quantity of SO_2 , which is evolved, it is necessary to employ a series of cooling vessels in which the CSCl_4 (after passing through a water-cooled condenser) may be deposited. We have usually employed four such vessels. In the first of these (which is air-cooled) the bulk of the desired compound collects. The second is cooled in ice, and the remaining two in ice and salt.

The CSCl_4 is then once again distilled in steam, in the usual way, in order to remove any unchanged sulphur chloride. It is then dried over calcium chloride and distilled (using a Young's 12-bulb column) until the temperature reaches 140°C . Experience has shown that the product then remaining in the distillation flask is pure enough to be used directly for the preparation of thiophosgene. A further quantity of CSCl_4 can be obtained by redistilling that portion boiling below 140°C . and again rejecting what has distilled below that temperature. Further small amounts of CSCl_4 can still be recovered from this low-boiling distillate, which contains in addition carbon bisulphide, carbon tetrachloride, and some thiophosgene, the presence of which is readily detected by its odour, the red colour of its vapour and the formation of the characteristic luminous, bluish fumes in presence of atmospheric oxygen. (In this connexion see *Bull. Soc. Chim.*, 1910 (4), 7, 722.)

The formation of thiophosgene was of particular interest since the ultimate object of our experiments was the conversion of the CSCl_4 to thiophosgene by means of a suitable reducing agent. The separation of this substance (b.p. 73.5°) from the fractions boiling from 70° — 80° was, however, impossible, owing to the presence of carbon tetrachloride (b.p. 78°).

Experiments in which carbon bisulphide, in presence of a trace of iodine, was treated with only two atomic proportions of chlorine in the hope of thereby reducing the formation of CSCl_4 and of CCl_4 were unsuccessful, no relative increase in the amount of the thiophosgene fraction being observed, which, indeed, under these conditions, was extremely small. These experiments are, however,

interesting inasmuch as very little carbon tetrachloride appears to be formed.

Although thiocarbonyl tetrachloride possesses a most irritating and objectionable odour, the whole of these experiments, except the large scale chlorinations, were carried out in a crowded laboratory, with careful utilisation of fume-cupboard accommodation, without occasioning serious inconvenience. When any of the product was spilt it was found convenient to cover it with sodium hydrogen sulphite, which reacts readily with substances containing the trichloromethyl group (Rathke, *Annalen*, 1872, 161, 149; 1873, 167, 202. Albrecht, *Annalen*, 1872, 161, 129).

THE PREPARATION OF PICRYL CHLORIDE. BY NITRATION OF 1-CHLORO-2,4-DINITRO- BENZENE.

BY PERCY FARADAY FRANKLAND AND FREDERIC
HORACE GARNER.

Picryl chloride was formerly obtained by the action of phosphorus pentachloride on picric acid. It can, however, be obtained by careful nitration of chlorobenzene or dinitrochlorobenzene. Once obtained, picryl chloride serves as the basis from which a large number of trinitro compounds of benzene and hexanitro compounds of diphenyl can be prepared.

The method of preparation of picryl chloride from 1,2,4-chlorodinitrobenzene was first described in G.P. 78,309, but in this patent the use of excessive quantities of nitric acid and oleum is recommended, and very few details of the method of manufacture are given. The purpose of the present investigation was to ascertain the exact conditions under which the nitration should be carried out, avoiding if possible the use of oleum and of temperatures above 130° .

The course of the nitration was followed by analysis of the nitrating mixture at short intervals. A mixture of sulphuric acid, nitric acid, and dinitrochlorobenzene was heated in a flask loosely stoppered with a plug of glass wool. The flask was immersed in an oil-bath maintained at the desired temperature. Small quantities of the acid mixture were removed at intervals for analysis. The liquor thus obtained was analysed for total nitric and nitrous acid in a nitrometer in the usual way, and for nitrous acid by means of sodium ferrocyanide and sodium citrate solutions.

In all the experiments described in this paper sufficient sulphuric acid was present to keep all the dinitrochlorobenzene in solution, and thus only one layer was present. In consequence of the large amount of sulphuric acid used no material increase in the concentration of water took place in the course of the nitration.

Crude dinitrochlorobenzene from British Dyes, Ltd., was used. When dried it melted at 43° — 44°C . (pure dinitrochlorobenzene melts at 50°C). The acids used were fuming nitric acid of sp. gr. 1.51, containing approximately 90% HNO_3 and 1% N_2O_4 , and sulphuric acid of the desired concentration made up from ordinary concentrated sulphuric acid and fuming sulphuric acid.

Setting points of mixtures of dinitrochlorobenzene and picryl chloride.

The setting points of mixtures of dinitrochlorobenzene and picryl chloride from 100% picryl chloride to 100% dinitrochlorobenzene were investigated. The dinitrochlorobenzene and picryl chloride used were pure, having been recrystallised several times from alcohol.

The curve plotted from these figures was used in the nitrations which follow to determine the degree of nitration which had taken place. There is a eutectic point in the curve at 56% dinitrochlorobenzene 44% picryl chloride and setting point 25.0° C. (corresponding approximately to 3 mols. dinitrochlorobenzene to 2 mols. picryl chloride)

Percentage of dinitrochlorobenzene.	Percentage of picryl chloride.	Setting point.
0	100	80.6°
4.7	95.3	76.6°
9.3	90.7	72.9°
13.6	86.4	70.0°
21.5	78.5	63.4°
28.8	71.2	56.8°
34.0	66.0	51.0°
37.5	62.5	46.7°
41.9	58.1	40.4°
47.2	52.8	33.1°
50.0	50.0	30.0°
52.4	47.6	27.3°
55.0	45.0	25.6°
57.1	42.9	26.1°
58.0	42.0	26.6°
60.5	39.5	34.0°
80.7	19.3	39.9°
89.7	10.3	43.8°
100.0	0	47.9°

Preliminary experiments had shown that, owing to the large amounts of nitrous acid produced during the nitration, it was impossible to obtain complete nitration without using a large excess of nitric acid over that theoretically required.

A series of experiments was carried out to determine the relative rates of nitration at different temperatures, using 100% sulphuric acid. From these experiments the most suitable temperature for the nitration was shown to be 100° or 110° C. as regards low production of nitrous acid compared with the rate of nitration; owing, however, to the low velocity of nitration at these temperatures, the temperature of 130° C. was chosen for carrying out the bulk of the nitrations which followed.

Further experiments carried out under such conditions that varying amounts of nitrous acid were present gave products exhibiting little difference in melting points after 12 hours at 130°; it therefore appears that nitrous acid has no great effect on the nitration.

It was also found that there is no advantage to be obtained by adding the nitric acid in stages, as regards the ultimate m.p. of the product. A greater loss of nitric and nitrous acids was observed when all the nitric acid was added at the commencement, but this was counterbalanced by the longer time required for the same degree of nitration.

To ascertain whether the production of nitrous acid had any direct relation to the amount of dinitrochlorobenzene present, three experiments (one with the nitrating liquid alone, another with the nitrating mixture and a certain amount of dinitrochlorobenzene, and a third with the same quantity of nitrating liquid and an amount of picryl chloride equal to the weight of dinitrochlorobenzene in the second) were carried out at 130° C. for periods up to 12 hours. The results indicated that by increasing the amount of dinitrochlorobenzene present in a given quantity of nitrating liquid the amount of nitrous acid produced is increased in the same proportion. This holds only up to three hours, as in the case of the longer experiments the amount of free nitric acid is diminished considerably after the initial three hours heating, and this diminution is greater the more dinitrochlorobenzene is present; therefore, since the amounts of nitric acid are greatly different, the experiments after three hours are no longer comparable. It was shown by experiments using pure dinitrochlorobenzene that the production of nitrous acid is not due to impurities present in the crude dinitrochlorobenzene used in these experiments.

In the case of picryl chloride there was a slight increase in the production of nitrous acid over the control; it was, however, much smaller than in the case of dinitrochlorobenzene. The decrease in nitric

and nitrous acids was less with picryl chloride than with the control, as would be expected. Similarly, the loss of nitric and nitrous acids by evaporation in the case of dinitrochlorobenzene would be less than that of the control.

The production of nitrous acid from nitric acid in the course of the nitration considerably diminishes the rate of nitration, since the amount of free nitric acid becomes less and less as the experiment proceeds; the diminution in nitric acid available for nitration is, in fact, more largely due to the production of nitrous acid than to its withdrawal by nitration.

Experiments on the nitration of pure dinitrochlorobenzene showed that the amount of nitrous acid formed is directly proportional to the quantity of dinitrochlorobenzene present and that nitrous acid is produced to the same extent whether crude or pure dinitrochlorobenzene is nitrated; also nitrous acid is produced with picryl chloride, but to a much smaller extent than with dinitrochlorobenzene.

Carbon dioxide produced along with the nitration.

Since, from the preceding experiments, it appeared that the nitrous acid produced along with the nitration is the result of oxidation of the dinitrochlorobenzene, the question arises as to the product of this oxidation; it is obvious that if dinitrochlorobenzene were oxidised to carbon dioxide, a larger amount of nitrous acid would be formed than if only a partial oxidation took place. It therefore seemed probable that carbon dioxide was the oxidation product, since no large amount of dinitrochlorobenzene disappeared during the nitration, and also since no substance other than picryl chloride was found in the fully nitrated product.

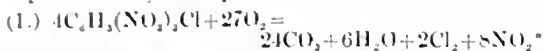
To settle this point a series of nitrations of dinitrochlorobenzene was carried out, and the amounts of nitrous acid and carbon dioxide formed were determined at intervals. When the results were plotted as graphs it was observed that for a short time nitrous acid was formed without any appreciable amount of carbon dioxide being evolved. After this initial lag, the production of nitrous acid was proportional to the amount of carbon dioxide given off up to about 6 hours; after this a slowly increasing amount of carbon dioxide was produced per unit amount of HNO_2 formed. This increase in the proportion of carbon dioxide to the nitrous acid may be explained as follows:—During the nitration in the latter stages a certain amount of nitrous acid is lost by evaporation, and this loss will not occur in the earlier stages; consequently the amount of nitrous acid formed at twelve or nine hours will be less than that actually found, and hence the ratio of carbon dioxide to nitrous acid is too high. Possibly another factor tending to make this ratio higher is the minute amount of substances not arrested by the apparatus. This amount will be the greater the longer the time, and, as comparatively little carbon dioxide is produced in the last three hours* as compared with the first, these will affect the later results much more than they do the former.

A similar series of experiments was carried out, using picryl chloride in place of dinitrochlorobenzene. The general results of these experiments were similar to those obtained in the case of dinitrochlorobenzene. The same decrease was observed in the amount of nitrous acid as compared with the amount of carbon dioxide evolved; the amounts of these formed in twelve hours were, however, less than one-tenth of the amounts formed in the case of dinitrochlorobenzene. There was no constancy in the ratio of the amounts of carbon dioxide

* The concentration of dinitrochlorobenzene is of course greatly diminished by this time through the formation of picryl chloride, and the latter is much less oxidisable. The concentration of HNO_2 is also much reduced.

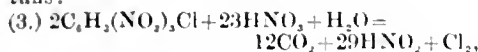
evolved to the nitrous acid in this case that was noticed with dinitrochlorobenzene (after the initial lag in this latter).

The oxidation of dinitrochlorobenzene may be represented by the equation:

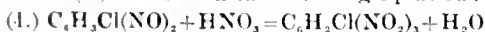


(oxygen being furnished by $2HNO_3 = 2HNO_2 + O_2$). Hence 1 g. HNO_3 expressed as HNO_2 is formed by the oxidation of 0.222 g. of dinitrochlorobenzene and gives 0.289 g. CO_2 . Actually the amount of CO_2 per unit of HNO_3 produced varied according to the length of the experiment. In one experiment 1 g. HNO_3 gave 0.241 g. CO_2 after three hours, 0.261 g. after six hours, and 0.282 g. after twelve hours. There is thus an increase in the amount of CO_2 produced per unit amount of nitric acid reduced; this may be explained as either due to the retention of some of the carbon dioxide in the liquid or to the formation of an intermediate compound which decomposed giving carbon dioxide. This would also account for the amount of carbon dioxide being less than that theoretically required.

In the case of picryl chloride a similar equation can be assumed for the production of carbon dioxide, thus:—



i.e., for 1 g. HNO_3 produced, 0.289 g. CO_2 is formed. Actually it was found that after 3 hrs. 1 g. HNO_3 gave 0.065 g. CO_2 , after 6 hrs. 0.092 g., and after 12 hrs. 0.142 g. Here a mere fraction of the amount of CO_2 theoretically required is evolved. The result is in accordance with the theory as regards the retention of the gas by the liquid; here the amount retained would presumably be about the same as in the case of dinitrochlorobenzene; but, as the total amount of carbon dioxide formed is much less in the case of picryl chloride this factor would influence the results more largely. The relative amounts of nitric acid used up in nitration and oxidation respectively were established by analyses of nitration mixtures using various proportions of dinitrochlorobenzene. In all nitration experiments where the loss of nitrous acid by evaporation was small in amount the decrease in $HNO_3 + HNO_2$ † as compared with the control was only about one-third of the corresponding increase in HNO_2 . On the assumption that this decrease is wholly due to the rate of nitration it is seen that of the nitric acid which is used up $\frac{1}{4}$ goes for nitration and $\frac{3}{4}$ for oxidation of the dinitrochlorobenzene. Correcting for the amount of nitrous and nitric acids produced on oxidation of the dinitrochlorobenzene, the ratio of the amount of nitric acid used in nitration to the amount used in oxidation is 1:1.8. By comparing equation (1.) above with the following equation:



it is seen that whereas 1 g. HNO_3 nitrates 3.21 g. dinitrochlorobenzene, 1.8 g. HNO_3 oxidises 0.469 g. Hence from 100 g. dinitrochlorobenzene 88 g. of nitrated product should be produced.

It will be shown that under the conditions given later a yield of 85% picryl chloride was obtained; the agreement is good considering that the calculation is only approximate.

Production of picryl chloride.

In the following experiments the proportion of 750 g. of 100% sulphuric acid to 100 g. of dinitro-

chlorobenzene was chosen because this is about the minimum amount (slightly over, in fact) which will dissolve the dinitrochlorobenzene; the picryl chloride formed separates out to some extent as the nitration proceeds.

Various quantities of nitric acid were added to these amounts of acid and dinitrochlorobenzene, and after 3, 6, 9, and 12 hrs. heating to 130° C. portions of the nitrating liquid were poured into excess of water, when the dinitrochlorobenzene and picryl chloride separated out either as liquid or solid; in the latter case the mixture of water and solid was warmed until the solid melted, when the supernatant water was poured off. The oil thus obtained was washed with hot water and a wet setting point determined. Picryl chloride washed with water in this way gave a wet setting point of 76° C. (dry setting point 80°–81°). A nitrated product with wet setting point of 72° C. was regarded as satisfactory.

These experiments established the following facts: Using 100% sulphuric acid, there is no advantage to be obtained by increasing the proportion of nitric acid beyond 125 g. of 90% acid and nitrating for 12 hrs. Other series of experiments showed that no advantage is obtained by adding the nitric acid in stages rather than the same quantity initially, or by subjecting the nitrating mixture to a lower temperature (100° C.) previous to the use of a temperature of 130° C. Using 96% sulphuric acid, a satisfactory product is obtained after 12 hrs. heating to 130° C. from a mixture of 175 g. of 90% nitric acid, 1500 g. of 96% sulphuric acid, and 100 g. of dinitrochlorobenzene. As before, no advantage was obtained by preliminary heating or the addition of nitric acids in stages. This large excess of sulphuric acid was found to be necessary, as when only 1000 g. was used a product of wet setting point 60° was obtained after 12 hrs. heating at 130°.

It was also found possible to nitrate dinitrochlorobenzene, using 94% sulphuric acid, but even with very large quantities of sulphuric acid and relatively large amounts of nitric acid the nitration was not complete in 12 hrs. at 130°. In one experiment a product of setting point 63° was obtained with 100 g. dinitrochlorobenzene, 3000 g. 94% sulphuric acid, and 200 g. 90% nitric acid.

If the nitration were carried out at a temperature of 140°–150° C. a saving in nitric acid and time could be effected on the results recorded above. Experiments were carried out by heating for three hours at 130°, raising the temperature to 140° in half an hour, then raising the temperature to 150° over a period of three hours, and finally heating to 145°–150° for a further period of three hours. Using 100 g. of dinitrochlorobenzene, 750 g. of 100% sulphuric acid, and 100 g. of 90% nitric acid, a product equal to that obtained in the former experiments with 125 g. nitric acid to the same quantities of acid and dinitrochlorobenzene was obtained in 9½ hrs. instead of 12 hrs.

From the above experiments the following proportions of acid and dinitrochlorobenzene are suggested as the most suitable for employment to produce good nitration after heating to 130° C. for 12 hrs.

Using 100% sulphuric acid, dinitrochlorobenzene, 100 g.; sulphuric acid (100%), 750 g.; nitric acid (90%), 125 g. (35 g. 90% HNO_3 is theoretically required for complete nitration).

The wet setting points of the product were as follows: After 3 hrs., 52° C.; 6 hrs., 63°; 9 hrs., 67°; 11 hrs., 69.5°; 12 hrs., 71°.

The yield of picryl chloride was 85%, m.p. 76° C., dry setting pt. 74°–75° C.

Using 96% sulphuric acid, dinitrochlorobenzene 100 g.; sulphuric acid (96%), 1500 g.; nitric acid (90%), 175 g. Wet setting points: After 3 hrs., 48°; 6 hrs., 61°; 9 hrs., 68.5°; 12 hrs., 72° C.

* A small amount of CO is produced, but this does not invalidate these conclusions.

† The nitrogen peroxide derived from the dinitrochlorobenzene is assumed to be dissolved in the nitrating mixture and therefore estimated at the same time as that derived from the nitric acid by oxidation of the dinitrochlorobenzene. The complete equation should therefore be:

(2.) $4C_6H_3(NO_2)_2Cl + 50HNO_3 = 24CO_2 + 2H_2O + 2Cl_2 + 58HNO_2$.
† This HNO_2 includes that formed from the NO_2 groups in the dinitrochlorobenzene suffering oxidation.

The yield of pieryl chloride was 85%, m.p. 77° C., dry setting pt. 75°–76° C.

The yield and quality of the product obtained are thus practically identical, irrespectively of whether 100% or 96% sulphuric acid is employed under the conditions indicated above.

Analyses of the products showed that with 100% sulphuric acid the loss in nitric and nitrous acids (expressed as HNO_3) was 9.6 g. per 100 c.c.; the loss in nitric and nitrous acids required for complete nitration 5.9 g per 100 c.c.; and the amount of nitrous acid formed 11.2 g. per 100 c.c. With 96% sulphuric acid the corresponding figures were 4.4 g., 3.2 g., and 7.8 g. per 100 c.c. respectively. Thus, with 100% acid 3.7 g. of nitrous acid, expressed as nitric acid, is lost by evaporation, and with 96% acid 1.2 g.; this difference is due no doubt to the greater concentration of the nitric acid when 100% sulphuric acid is used.

The Chemistry Department,
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Communication.

NOTE ON THE DETERMINATION OF TAR ACIDS IN DRAINAGE FROM TARRED ROADS.

BY J. J. FOX, D.SC., F.I.C., AND A. J. H. GAUGE, F.I.C.

As a result of experiments carried out by Mr. A. J. Mason-Jones, biologist to the Joint Fisheries Committee (Tar Pollution) of the Ministries of Transport and Agriculture, it is now known that water which has been polluted by tar and in which the amount of tar acids is as little as 1 part per 100,000 is fatal to fish life. It is therefore important to possess a ready means of detecting and estimating such minute quantities of tar acids. The method described in this note depends upon the formation of the azo-dyes obtained when sulphanilic acid is diazotised and poured into water containing tar acids after it has been rendered alkaline. The depth of colour produced is matched in a Nessler tube by the colour obtained under similar conditions with a mixture of the three cresols in the proportions specified below. The tar acids in the washings from tarred roads will not as a rule include much phenol, but consist of cresols and higher phenols. The mixture of cresols used by us has been found to be generally available as regards shade of dye produced, but occasionally the matching becomes a little troublesome on account of the red shade produced by the higher phenols.

The solutions required are (1) recrystallised colourless sulphanilic acid, 1.91 g. in 250 c.c. water, (2) pure sodium nitrite, 0.85 g. in 250 c.c., (3) sulphuric acid, 1 part of strong acid (sp. gr. 1.84) to 3 parts of water, (4) 8% sodium hydroxide solution, (5) standard cresol solution containing 1 g. mixed cresols per litre. The mixture of cresols for (5) consists of 35% *o*-cresol, 40% *m*-cresol, and 25% *p*-cresol.

The diazotised sulphanilic acid solution is prepared about 5 minutes before use, by mixing 5 volumes of sulphanilic acid solution with 1 volume of the sulphuric acid, adding 5 volumes of sodium nitrite solution, and cooling in a stream of cold water.

The cresol solution used for comparing the colour is a dilute solution prepared by mixing 5 c.c. of the strong solution (5) with 95 c.c. of distilled water, i.e., 1 c.c. of the dilute solution = 0.00005 g. cresol. Weak solutions of cresols are liable to change, and the standard dilute solution should therefore be prepared just before use. The strong solution (5) is stable if kept in a cool, dark place.

To make the test, 100 c.c. (or less of a strong tar extract) of the clear water or washings is placed in a Nessler cylinder and 5 c.c. of the sodium hydroxide solution added, followed by 10 c.c. of the diazotised sulphanilic acid. On stirring, an orange-coloured dye is formed when tar acids are present. The depth of colour is matched by adding to a similar quantity of distilled water in a second Nessler cylinder 2 c.c. of the dilute standard cresol solution, 5 c.c. of sodium hydroxide solution, and 10 c.c. of the diazotised sulphanilic acid solution. This is the trial assay, and determines the amount of cresol solution to be used to obtain an exact matching of the depth of colour due to tar acids in the water under test. The best results are obtained when 1 to 2 c.c. of the diluted standard cresol solution is required to obtain equal depths of colour. This can be secured as usual in this class of work by taking smaller or larger quantities of the sample under examination. The amount of tar acid is calculated from the quantity of cresol solution required to produce equal tints.

When the washings are coloured or opalescent the tar acids can be obtained by steam distillation, taking 250 to 500 c.c. of the water and testing successive portions of the distillate. This procedure is also advantageous when traces of tar acids are suspected, as it affords a means of concentration.

We have found that very dilute solutions of various tar products, *e.g.*, certain tar acids and tar bases and naphthalene, in non-sterile water rapidly undergo biological change. For this reason the examination of samples of water for tar acids must be carried out at the earliest possible moment after collection. A delay of 48 hours may result in the oxidation of a considerable proportion of the tar acids.

From Mr. Mason-Jones' results the presence of more than 1 part of tar acids per 100,000 in effluents or road washings must be regarded with suspicion where fish life is concerned.

The test for tar acids outlined above is very delicate, much less than 1 part per million being detectable. By this means phenols in minute quantities have been found to be very widely distributed. A fuller account of this will be given on a future occasion, together with a statement of the alteration undergone by dilute solutions of phenols and tar bases under the action of bacteria.

Government Laboratory,
July 24, 1920.

Annual Meeting.

Chemical Engineering Group Conference, Newcastle, July 14, 1920.

RECESSED PLATE AND PLATE AND FRAME FILTER-PRESSES: THEIR CONSTRUCTION AND USE.

BY EUSTACE A. ALLIOTT, B.SC. (ENG.), A.M.I.MECH.E.

Although much has been said and written about the filter-press, little has been mentioned concerning working conditions and performances. The object of the present paper is to make clear the construction and meaning of the numerous variations of type for those who have not had time to give special study to the matter, and an endeavour will be made to give helpful data and suggestions as to the use of this type of plant. In order that the various particulars may be as up-to-date as possible, a special effort has been made to obtain recent data from users which will represent their normal works practice. The figures given, therefore, are not test figures taken on a small scale, but may be assumed to apply to every-day large-scale operations. It should be understood, however, that the same material may vary widely in its filtering qualities, these variations being sometimes due to almost imperceptible variations in manufacturing processes, and in considering any particular problem due weight must be given to this point.

It is proposed to deal with the subject in the following order:—

- (1) Construction of the simple or "non-washing" filter press.
- (2) Choice and care of the filter cloth.
- (3) Theoretical and practical considerations concerning pressure, viscosity, rate of flow, cake thickness, size and shape of suspended particles, etc.
- (4) Methods of feeding the press.
- (5) Washing in the filter press, with details of construction, theoretical and practical considerations and results.
- (6) Air drying, steaming, hot and cold presses, etc.
- (7) Labour in discharging.

Owing to the familiarity of the filter-press it does not usually receive the attention it deserves, which would doubtless be accorded to it if it were a new piece of apparatus, or if the simplicity of its operation were less obvious. Nevertheless, even with such an apparently simple piece of plant, the application of a little intelligence and understanding will bring increased satisfaction, both in the choice of the press itself and in working it with minimum labour costs, maximum output, and best extraction of soluble matter from the cake.

Construction of the filter-press.

A filter-press may be described as a frame in which a number of loose slabs of filter surface may be clamped to form a series of hollow chambers capable of withstanding internal pressure. The filter surface is ribbed or grooved and covered with cloth. This arrangement gives a maximum of area in a minimum of space, and is capable of modification to meet the varying conditions called for by different grades of material, both as regards thickness of cake, suitable pressure, varying filter media and arrangements for washing and steaming the cake when formed. A large variety of constructional materials can be used, and the whole plant

is of high reliability and free from tendency to break down.

Two forms of press are in general use, namely, the recessed plate type, and the plate and frame type. These are often referred to as chamber presses and frame presses respectively. In the former type (fig. 1) the rims are raised, so that when two plates are brought together edge to edge a hollow chamber is formed between them. The feed passage is taken through the body of the plate, while the filter cloths are laid over each side of the plate, forming a pressure-tight joint at the rims when clamped up, and having holes for the feed passage, which are sewn together or clipped to make a joint at the corresponding hole in the plate. This type of press has the merit of being the least expensive and requiring less labour. It is very suitable for thin cakes, owing to the free communication of the feed passage with the chambers. The usual cake thicknesses are 1 in. on the smaller sizes, 1½ in. on the intermediate and large, and up to 2 in. on the largest plates. It is not generally desirable to use recessed plates for thicker cakes than these on account of the strain on the cloth imposed by the raised rim.

The liquor enters the chamber under pressure, the solid portions being retained by the cloth, while the fluid portions pass through and drain behind the cloths through the grooves or corrugations to the exit ports. They pass finally into the filtrate trough by simple spouts if rough filtration only is in question, but more usually taps are fitted by means of which any one chamber can be isolated if the filtrate from it is muddy, due to faulty cloth. Taps are also needed for certain arrangements of washing presses. Where a liquor must not come in contact with air, or it is desired to raise it to a higher level direct from the press, it can be taken away by an enclosed outlet passage.

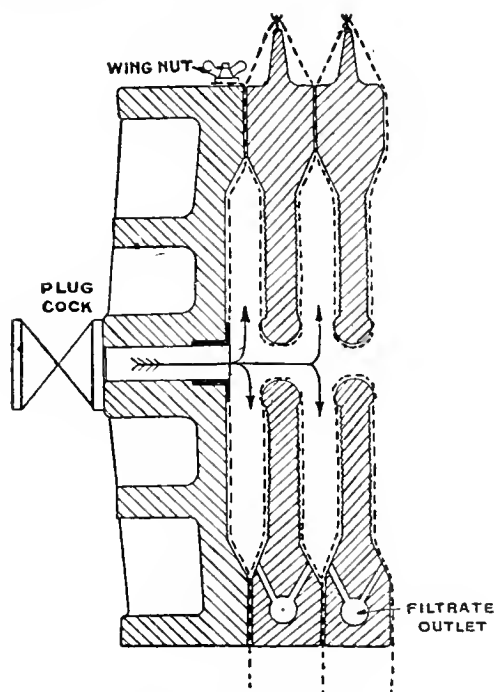
Recessed plates usually have a central feed, and for most purposes this is as suitable as any. It is sometimes preferred in the top corner in cases where the cake is apt to be slimy round the feed passage. By this arrangement the moist portion of the cake is readily isolated from the bulk, which is firmer and drier. Putting the passage in a corner in this manner, however, is apt to cause more strain on the cloth in a recessed type press. Bottom feed is useful if a solid cake is not to be formed, as the liquid contents of the cells can be run out before opening the press, but where the solids have a strong tendency to settle it may become choked, especially if the flow is slow.

When heavy suspensions are being dealt with, top feed is therefore valuable. In cases of this kind two feed passages are sometimes arranged at different heights so as to get the maximum consolidation of cake. It should be realised that the path of each particle is determined by definite stream lines, in conjunction with its own tendency to sink or float. Thus, with bottom feed, no large heavy particles may reach the top of the plates, unless the stream line flow is strong. There is no agitation worth speaking of in the press itself, as the small amount which may occur near each inlet port is quite powerless to cause redistribution if selective settling has been allowed to occur.

The plate and frame type of press (fig. 2) is suitable where very thick cakes are to be formed, as in this case the plates themselves are flat, and the chamber is formed by hollow frames being placed between them. This type of press is also suitable for materials which tend to rot the cloths, as the latter lie quite flat on the plate; it is a necessity if filter paper is to be employed. It must be used where very thick cakes are essential, but is not suitable for thin ones or materials of a lumpy or uneven nature, as the feed ports are generally taken in through the rim of the frames, and are

thus restricted in area. On a small press, say 25½ in. square, frames as thin as ¼ in. can be used if the feed is of a more or less liquid nature throughout the operation; ½ in. or 1 in. frames are usually quite thin enough, and where chambers exceed 36 in. square it is preferable that these should be thicker if possible. For this reason it is sometimes desirable when handling very free filtering materials, having a tendency to deposit a very hard, thick cake, to use an internal feed. Passages arranged externally to the cake are highly satisfactory for less freely filtering substances, as they isolate the moist unfiltered liquor from the drier cake.

Further, the feed holes in the cloths are clipped automatically by the joint surface of the plates and frames, and so need no sewing or special clips. The usual position for the passage is at the top centre of the press, which allows little opportunity for displacement of the feed holes in the cloths either by accidents or shrinkage. Another position in general use is in the side of the press at a little above or somewhat below the centre. Such passage is not so liable to get blocked by settlement as one in the bottom of the press, and is in a favourable position for consolidating the cake.



Enlarged sectional elevation.

passages or taps for this purpose, but except for washing presses it is rarely that these are necessary.

Press plates are usually made in cast iron for neutral or alkaline materials, or in wood for acid liquors, or for dealing with such materials as colours, which might be spoilt by contact with

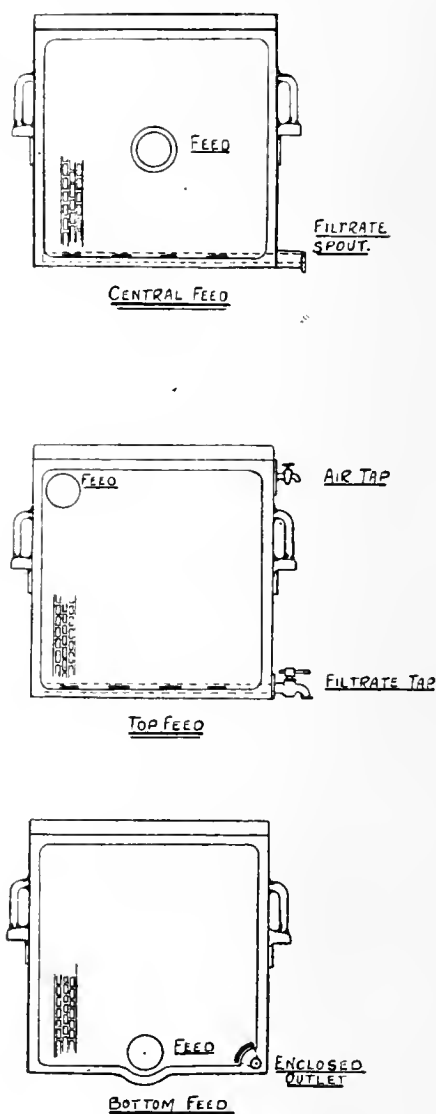


Fig. 1.

Cast-iron recessed plates. Types of feed inlets and filtrate outlets.

For varnishes and gummy liquors which flow with difficulty these presses are sometimes made with four passages, one at each corner of the chamber; one of the lower passages serves as feed inlet and drain while the other three act as filtrate outlets, being joined together at the head of the press. By this arrangement the liquor has a minimum distance to travel through the grooved surface to get to the outlet passage. Further, the upper passages ensure a free escape of the air from the chamber, whereas it might otherwise have difficulty in passing through the joints when these are under heavy pressure and the cloths heavily gummed up. Sometimes presses are provided with special air

iron. They can, however, be made in bronze or aluminium, or may be covered with hard lead or vulcanite. In the latter case the vulcanite is usually supported by thin steel sheets and the joint surfaces are made of softer rubber. Such a press will deal with strong hydrochloric acid.

Wood plates have been made of a variety of materials, but on the whole the coniferous woods are the best, as these have the greatest uniformity of structure, suffer the least disturbance from medullary or transverse rays, and have less sapwood and small shrinkage. For strength, cheapness, and service generally good pitch pine is difficult to equal. Presses of this material have been

reported as withstanding very satisfactorily 8% hydrochloric and 30% sulphuric acid. The author has reports where even 10% or stronger hydrochloric acid is being used, but it is preferable not to go above 4% if at all possible. Timber has to be carefully selected for such work. After the best quality wood has been bought some 20% or 30% has usually to be rejected on first inspection, with further rejections after it has been worked up. Oak makes good press plates, and is especially good for withstanding acetic acid, while teak is sometimes reported as being more resistant to acids than pitch pine, though it is more expensive. Beech, maple, or sycamore are used as substitutes, but the previously mentioned woods are preferable. Cypress is also used, more especially in the United States. Very much depends on the quality of the wood, straightness of grain, freedom from knots and shakes, and specially freedom from sap on the filter surface. Impregnation is of doubtful value for increasing resistance to acids. Of preservative mediums paraffin wax (or coresin) is perhaps the best. It is of importance to keep wood presses

must be adequately supported with a minimum amount of dead space, though, providing the channels are free, the percentage of dead space within reasonable limits seems to make no appreciable difference in practice. Finally, smoothness and absence of sharp corners is a factor of no little importance. Covering the surface with perforated plates is only to be recommended in special cases where the adequate supporting of the cloth has to take precedence over every other consideration. Coarse-mesh gauze makes a good support. But any covering adds to the work of keeping the filter surface clean. For very viscous liquors the supporting of the cloth may have to give way to the necessity for providing very free flow at the back of the cloth, with consequent wider gaps between the supports.

In large presses, say over 36 in. square, where very high pressure or uneven filling of the chambers may be encountered, it is necessary to provide the surfaces of the plates with bosses to act as additional supports. Such bosses may be cast upon the face of the plates in the first instance, though

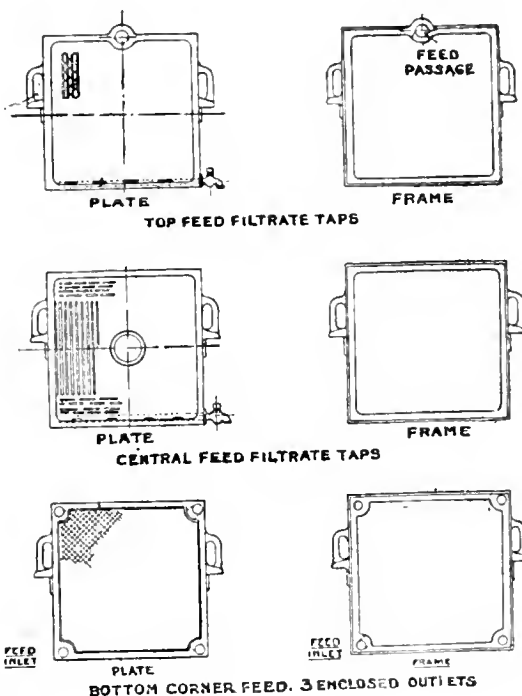
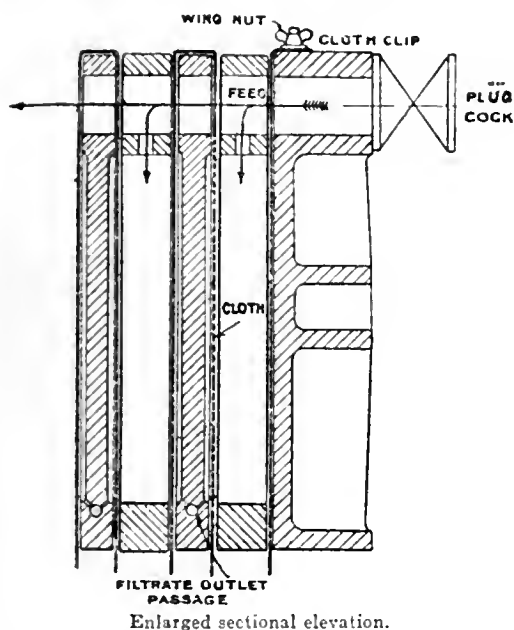


Fig. 2.

Flush plates and frames.

closed and moist when out of service. They should be sluiced down before dead periods and kept filled with water. Shrinking is thus avoided, and there is no opportunity for weak liquors left on the plate surfaces to concentrate by evaporation and attack them. Further, in starting up a new press the stay rods should not be too tightly screwed up but a little allowance made for the necessary expansion of the wood when first moistened. These rods can gradually be tightened up after a little use, but should not be allowed to cut into the wood.

There are various types of filter surface, ranging from very fine straight grooves to comparatively coarse pyramids or lozenges. The first and most important point is that the channels should be sufficiently deep and narrow not to be closed by the sag of the cloth. In the second place the cloth

in some cases it is preferred to add specially made bosses screwed on to the face. The great essential is that the height shall be accurate and the business surface carefully rounded off. Further, the slope must be gentle to avoid injuring the cloths. Unless absolutely necessary, such bosses are preferably dispensed with.

The design of handles requires more care than is often given. There should be ample room to get the hand in easily without any trapping or fouling. In the case of heavy plates rollers are usually fitted, and in some cases a special carriage is even arranged on which the plates can be run along the press. Some users prefer the handles to be kept very low (*cf.* fig. 40), as they state that the plates are then drawn back more evenly, and there is less tendency to tilt, and cut the cloth between the edge

of one plate and the face of another. It is doubtful, however, whether there is any real difference in actual practice, as the ordinary type of handles are very convenient to grip.

For feeding the press and for washing purposes cocks are better than the ordinary type of valve. A useful valve, however, has the screw entirely clear of the stuffing-box, and the valve itself is carried through the stuffing-box so that nothing can get at the back of it to jam. Occasionally presses are fitted with a rail running right along one side, a little below the bottom of the plates. This and the gutter serve as supports for draining trays which catch any drip, especially in the case of old wood presses. All the liquor then runs to the trough, and the floor is kept clean.

A "dead" plate, or "cutting-off plate," is a desirable fitting if batches of varying size are to be dealt with, some of which may be insufficient to fill the whole press. Such a plate has one working face only, and is sufficiently strong to withstand the unbalanced end pressure. It can be placed anywhere in the press, permitting a few chambers to be used while the rest are isolated. In default of this sufficient space may be made for the tailings of a batch by emptying a few chambers at the feed end only, leaving the remaining cakes to be removed when these have been filled again.

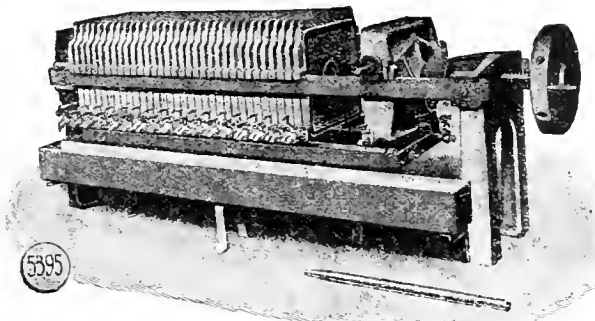


Fig. 3.

The question of side rods has had some attention from all makers, but it is difficult to say where the greatest advantage lies. Flat rods may be used on somewhat longer presses without support, but the plates slide more readily on the round bars, and these can be conveniently trussed in some types of press. It is important that sufficient space be left between bars and plates to give convenient room for the edges of the cloths. The plates are kept in position by distance pieces, though in the case of flat bars the handles on one side are sometimes provided with a notch for this purpose. This, however, is liable to jam if the plates are not moved back perfectly evenly.

After the plates themselves, perhaps the most conspicuous feature is the closing gear. There are

a great variety of designs, both hand and power operated, but on the whole the simplest types are preferable so long as they will give the required pressure.

The usual type of gear for small presses is the centre-screw pattern (fig. 3). This is in use for wood presses up to 36 in. or more in size, working at pressures up to 30 or 40 lb., but is preferably limited to the 25½ in. size and under. Such a gear will readily close an ordinary 25½ in. cast-iron press against 100 lb. pressure. A swinging distance piece on the head enables the latter to be drawn back after the tightening screw has been released by a couple of turns. It is sometimes arranged to run on wheels, and in the case of larger presses these are often on a common axle, fitted with a handle to give quick movement. The centre of the head is also recessed so that it can be pushed right up over the tightening screw and further play obtained. In some forms, instead of having a distance piece, the end standard is fitted with a pivot which carries the screw, so that as soon as the latter is released it can be swung up right out of the way. A common means of tightening is by a bar inserted into holes on the spindle head, a method which gives a very sure grip. Ratchet devices are, however, in common use. In a somewhat special variant of this form the screw is stationary, and a nut which revolves on it transmits the pressure through levers to half a dozen arms or rods which bear on the press head. These arms can be released and shot right back when it is desired to empty the press.

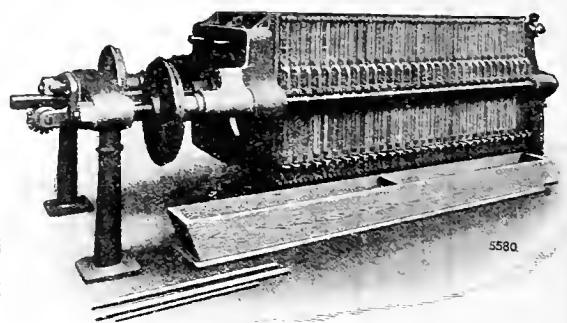


Fig. 4.

Another gear suitable for larger presses consists of a rack, fixed to the loose head, engaging a pinion on the end standard (fig. 4). By this means the head is rapidly moved backwards and forwards, while the side rods are screwed and carry wheel nuts which are tightened by levers against swinging distance pieces. Such a device will adequately close a 38 in. cast-iron press against 100 lb. per square inch or even more. It is necessary to attach the rack to the loose head by a joint, as it is found that there is slightly more accumulation of dirt at

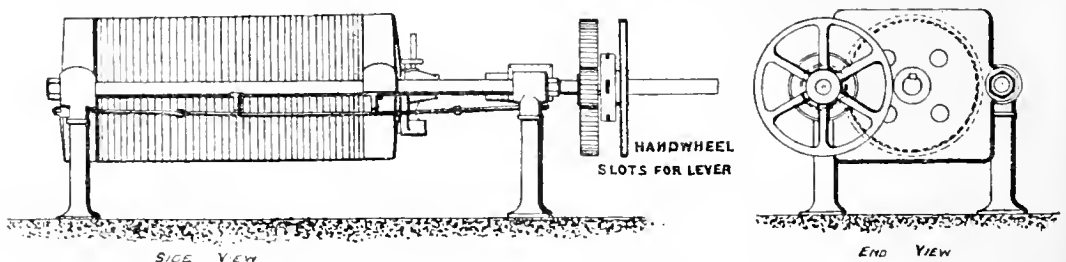


Fig. 5.

38 in. plate and frame filter press. Spur wheel and pinion closing; and trussed side rods.

the bottom edges of the plates, and this causes the head to tilt.

Another form which is often employed is the spur wheel and pinion gear (fig. 5). This is generally similar to the centre-screw type, but the screw is driven by a large spur wheel engaging with a pinion, giving an additional multiplication of from four to eight times according to the size of the press. The pinion is fitted with a large hand-wheel for rapid manipulation, and final tightening is accomplished by levers. Sometimes both spur wheel and pinion are fitted with elaborate devices for slow and quick movement, but according to the author's observation the simpler arrangements are better, as the more elaborate are apt to consume time without giving any better closing. Such a spur wheel and pinion device permits the use of trussed side bars in the case of a long press. This arrangement obviates the necessity for supporting pillars, which may interfere somewhat with the removal of the cake from under the press. Such a gear is suitable for most duties on either wood or iron presses, but a more powerful one is the toggle

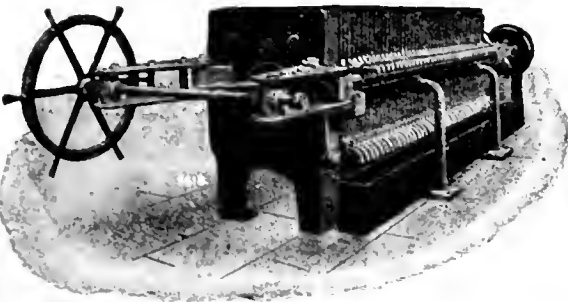


Fig. 6.

lever pattern (fig. 6). This is suitable for large wood presses up to about 48 in. The arrangement of the loose head is like the rack and pinion closing gear except that the pinion has its own spindle and hand wheel for ease in operation. The side rods, however, are not fixed to the heads but are attached to the short arms of a pair of levers which have a hinged connection to the fast head with a screw and hand wheel, by means of which their long extremities are drawn together, a very large mechanical advantage being thus obtained. The merit of the device is the ease by which one man can put on the final tightening pressure simply

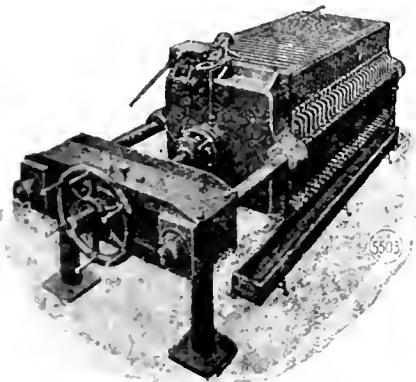


Fig. 7.

by operating the hand wheel without the use of levers. The arrangement of the gear is such that perfectly even pressures are applied to both sides of the press.

Yet another form of hand-closing gear utilizes a small hydraulic pump mounted on the loose head (fig. 7). This is suitable for very high pressures or large presses. It is somewhat costly, and takes longer to operate, but puts on a perfectly definite amount of pressure, which can be regulated exactly by the gauge. In the instance illustrated the head is moved up and down by a centre screw and large hand wheel, the hydraulic ram being housed in the centre of the loose head and engaging the end of the screw. The ram carries a lock nut, which is screwed up when the pressure has been put on, preventing the press blowing or leaking when the pump pressure drops. Pneumatic closing (fig. 8) saves labour, in so far that the head can be drawn back simply by manipulating a valve, and by the

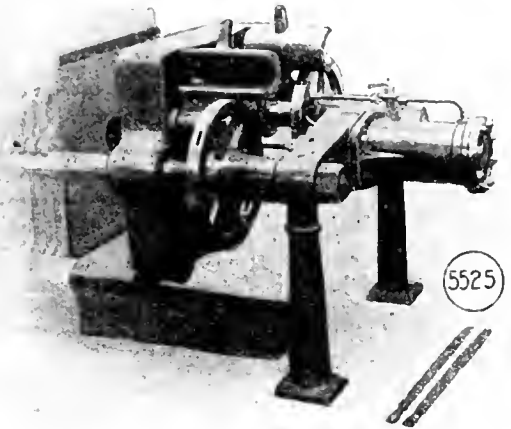


Fig. 8.

same means may be pushed back again, carrying all the empty plates with it. This is a very valuable feature in large installations having simple recessed plates in which the cloths and passages require no attention during the replacing of the empty plates. Large numbers are in use for sewage sludge. It should be remembered, however, that the actual pressure is not put on by the ram but by the wheel nuts on the side rods.

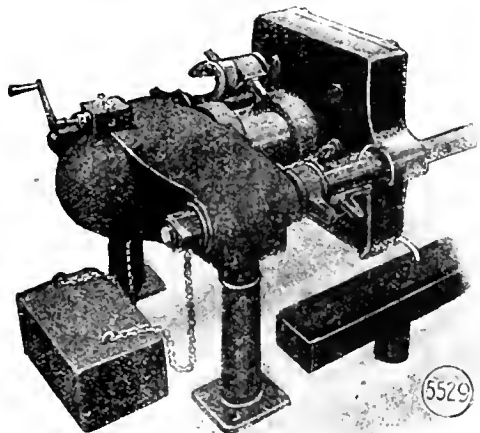


Fig. 9.

The hydraulic closing gear shown in fig. 9 has an additional advantage in that the final tightening is done mechanically, the only handwork being the operation of the locking device in case the water pressure drops. In the instance shown this

locking gear is very similar to the previous tightening gear, except that only hand pressure has to be put on the lock nuts. The loose head may be drawn back either by a special hydraulic cylinder or by weights. Where the space is small, or it is not possible to cut through the floor to accommodate the chains carrying the weights, special devices can be arranged on the top of the end standard so that the weights are kept above the floor.

division and muddy to another. Separate run-off valves may be also provided for washings. In the case of washing presses it is usual to make the trough larger and provide a plug or cock, so that it can be used as a measuring tank for the wash water.

Where presses are installed on an upper floor, discharging to the lower one, steel shoots are fitted to the fast and loose heads. If volatile liquors or those liable to give out dangerous vapours are

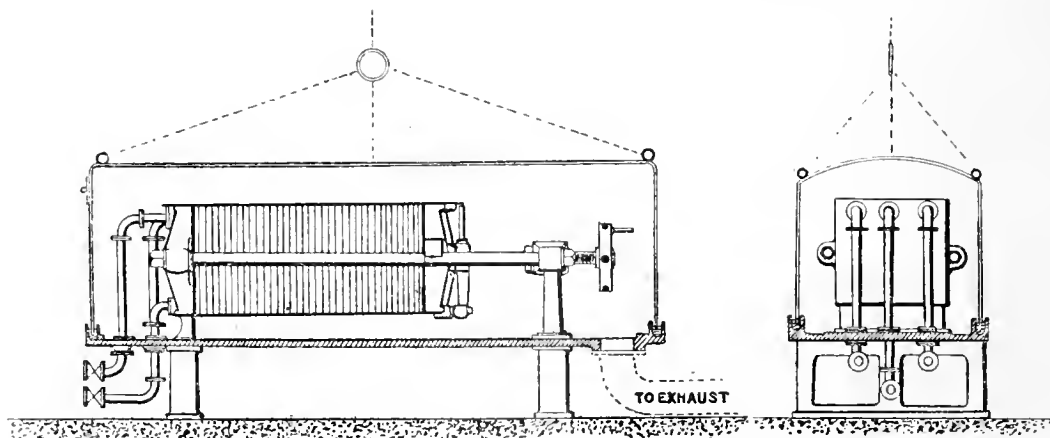
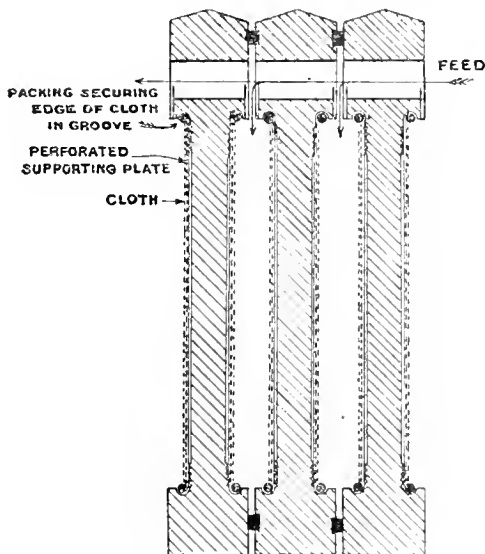


Fig. 10.

Enclosed filter press.

25½ in. square; washing type; enclosed outlet; C.I. tray and sheet iron hood; feed and wash pipes taken through tray with device for air flushing at end of operation.



Enlarged sectional elevation.

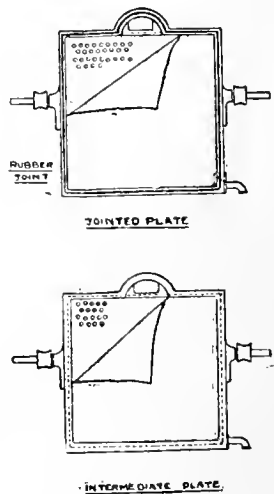


Fig. 11.

Plates with inserted cloths.

Cake trays are often provided to go under the press, thus saving labour in this direction. At times the whole press is arranged to stand in a special trough which completely surrounds it, and serves to isolate the cake from the rest of the floor.

The filtrate trough is sometimes arranged with a longitudinal division plate. The taps are then fitted with rubber extensions or swivel nose-pieces, by means of which clear filtrate can be run to one

being dealt with the press can be fitted with a base on to which a cover fits, the joint being made by means of a water seal or clamps (fig. 10). In this case the press is arranged with a continuous outlet passage, and the feed and any other passages are controlled by external valves.

For strong caustic liquors, which cause ordinary cotton cloths to perish rapidly, or for delicate cloths, the plates can be arranged so that the cloths

do not pass over the joints, but the latter are made by rubber or other rings let into grooves all round each rim in alternate plates (fig. 11). All the plates are then provided with a recess all round the inner side of the rim, into which the edge of the cloth is laid and made fast by a packing ring being caulked in. Alternatively the cloth may be secured by being clipped in by a joint plate which is held down by screws. Further, in such instances a perforated plate is laid over the usual corrugations, thus still more reducing the strain on the cloth, which has then a perfectly flat surface on which to rest. This arrangement effectively lengthens the life of the cloths, as the greatest wear usually takes place at the nip of the plates, and, further, if the cloth is kept well supported at every point, so that a spud cannot readily be pushed through it when the cake is being cleaned off, it may readily last, even when apparently quite rotten. A perforated plate or grid is sometimes laid over the top of the cloth. This protects it from the spud, and prevents a rotten cloth from being torn away by the weight of adhering cake. It will be realised that recessed presses of this type cannot be constructed to make thin cakes, but are suitable for thick ones from, say, $1\frac{1}{2}$ in. upwards.

Where quantities of non-viscous liquor containing very small amounts of very fine matter in suspension are to be filtered special presses are in use. The plates are not provided with a filtering surface of the usual type, but are filled with cakes of finely divided cotton pulp or other suitable material. Such a press would filter many liquors at the rate of about 4 galls. per sq. ft. per hour, and when it became clogged the pulp would be knocked out of the chambers, washed in a special washing machine, and then pressed firmly into position again by means of a special hydraulic or screw press. A usual method is to prepare the pulp cakes during the filtration period and then lay or press them in position in the specially designed plates at the end of the run, when the old pulp has been knocked out for washing.

Choice and Care of the Filter Cloth.

In recessed plate presses double cloths (fig. 12) are often used. These cloths are stitched together at the edges of the feed opening, and one cloth is passed through the feed-hole in the plate, both halves being then spread over the filter surface and



Fig. 12.

fastened at the top by clips or pins, or they may be stitched. In such cases the top of the plate carries a projection or fin for convenience in fastening the cloth. This method of fixing is suitable for most purposes, and is strongly recommended where cloths are thin or have often to be taken off and washed. For certainty in obtaining clear filtrates with fine precipitates there is nothing to equal cloths with sewn openings.

Another way of clothing the press is to take a strip of cloth, long enough to cover both sides of the plate, with two holes cut in it corresponding to the feed passage. This is hung over the top of the plate, and the joint round the feed passage is

made by clips. A very usual form of clip, the "bayonet" pattern (fig. 13), is in halves, one of which is provided with inclined projections, which engage with lugs on the other one, so that on giving a slight rotation the two halves are pressed firmly on to the cloth, making the joint. This form is the quickest to apply, but the screw pattern (fig. 14) is to be preferred as the most secure. It

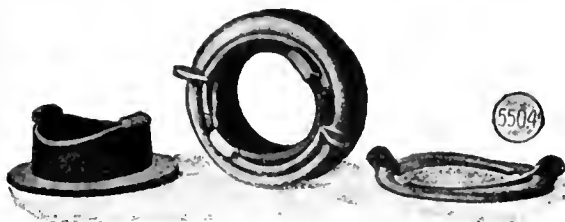


Fig. 13.

is however, rather more costly. Such clips save a good deal of time and expense in stitching round the feed opening, and are useful where the cloths have to stay in the press till they are worn out. They are, nevertheless, apt to be misused. For instance, where cloths have to be taken frequently from the press for washing purposes it will be found that the double cloths previously described are much more quickly removed and replaced. In one case of a 38-in. recessed plate press having 26 chambers, using thin cloths, it was found that the use of cloth clips made a difference of three-quarters of an hour in stripping and re-clothing as between the use of double cloths with sewn centres and "bayonet" clips. Then again with thin cloths it is sometimes difficult to get clips to make a secure joint.



Fig. 14.

In frame presses cloths of double length may be hung over the plate without any special attachment or stitching, but it is best to sew them on if there are many passages in the plate rim and the cloth is to remain for some time.

Still another method is to use single cloths, caught by clips or pins at the top of the plate. The cloth top is sometimes eyeletted and engages with hooks of adjustable height. This permits ease in fixing when replacements are frequent, but sewing is the best if well done.

Fig. 15 shows a comparatively coarse precipitate of calcium sulphate (magnified 18 diameters), while fig. 16 shows to the same scale a tightly woven 18-oz. twill cloth, with no spaces between the threads, such as is used for filtering substances of a fine uniform nature, which would not be perceptible except at a much higher magnification. Such a tightly woven cloth is a necessity in some cases, say for many dyestuffs, but would be quite unnecessary for such a precipitate as calcium tartrate (fig. 17), in which the crystals range from comparatively large, say 0.1 mm. side, to very small. An open jute cloth (fig. 18) served very well for this

purpose, though even after careful shrinking many holes of 0.25 mm. side are apparent. In such a case the first fine particles pass through the cloth, while a layer of the coarser ones is rapidly built up on it, the spaces between them being so fine that even the smallest are caught. The first runnings are muddy, but soon brighten, and can be readily returned to the press, while the cloths retain their free-flowing qualities for longer than if an attempt had been made to get clear liquor at the start. The spaces between the threads in the shrunk cloth may be larger than any of the particles, but apparently the latter tend to jam, and are assisted in this by stray fibres from the threads.

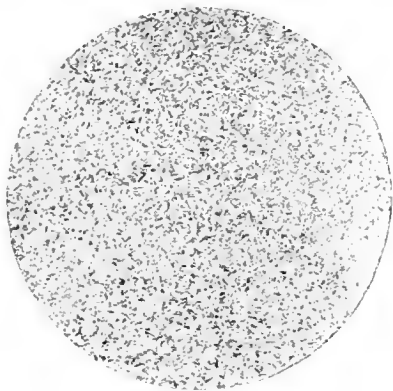


Fig. 15.
Sulphate of lime ($\times 18$).

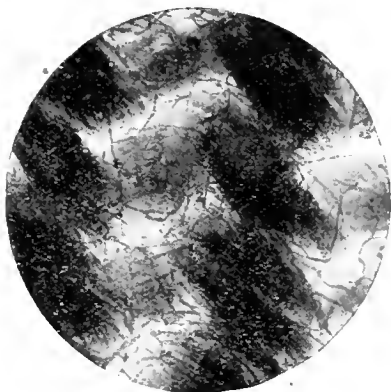


Fig. 16.
Twill cloth ($\times 18$).

In dealing with caustic soda, about 5% free alkali is as much as a cotton cloth will stand to give a reasonable life in ordinary types of press. Somewhat stronger liquors may be filtered if the cloth is treated with heavy naphtha or green oil, especially if care is taken not to allow the caustic liquor to dry into the cloths. In one case which came under the author's notice good results were reported consequent on the removal and soaking of the cloths every week-end. This was additional to the use of an oil preservative. Some users report having employed twill cloths with 10% free alkali, the life being about three weeks, but it is desirable, if possible, to keep to 3% or 4%. A similar percentage of sulphuric acid should be satisfactory, though some users report that cotton cloths withstand 7% to 8% economically. Twill cloths are perhaps in greater favour than the chain weave, though some

users say the latter are stronger and give finer filtration. As far as the author can judge there is not much difference between the two, providing the cloths are properly chosen. The mercerising effect of alkali should not be forgotten, and if a cloth is to be used for alkaline liquors it is best not too tightly woven, as otherwise it may become relatively impervious. Further, for corrosive liquors those with the thicker threads seem to give the best results, as they will hold together in the press even after they have become quite rotten.

Linen is employed for the more open-mesh straining cloths, which are sometimes useful in the filter press when dealing with comparatively coarse sus-

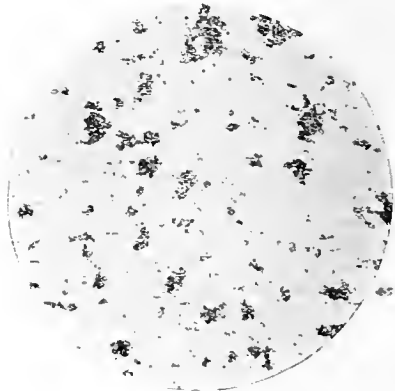


Fig. 17.
Calcium tartrate ($\times 18$).



Fig. 18.
Jute cloth ($\times 18$).

pensions in liquors containing casein or other substances, which would rapidly render a mere compact cloth impervious. Jute and hessian cloths or sacking are often used instead of cotton, and are quite good with weak alkaline liquors, providing a very fine, tight weave is not requisite.

These cloths are also used as under-cloths, a strong coarse cloth being used to support a thin, tightly woven twill. This helps to protect it from damage at the joints, especially where there is likely to be trouble with rusting. Further, in such an arrangement the upper cloth may be sufficiently closely woven to stop very fine particles, and yet its thinness prevents its becoming too rapidly impervious. It is readily removed and washed, while the under-cloth, though strong and heavy, can be coarse enough to run freely for a considerable period. Filter paper may be used in-

stead of a fine upper cloth. It must be well supported, and should be of the crinkled variety, as this can give a little to the sag of the cloth. Paper has unfortunately the disadvantage that it may come away with the cake, but this is of no consequence if only the clear liquor is required.

Cloths with a swansdown surface are used for such purposes as the filtration of yeast, and are then protected from clogging by a superposed cloth of strong twill. It is, however, open to question whether they are any better than good twill cloths.

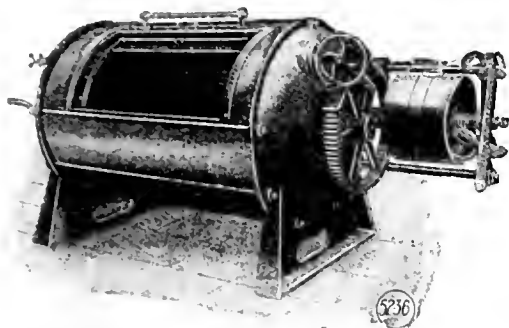


Fig. 19.

For strongly acid liquors there are a variety of cloths to choose from. One firm reports that camel's hair is satisfactory up to 30% sulphuric acid and 10% hydrochloric acid, while horsehair would stand 10% hydrochloric acid, but is not good for sulphuric acid. The same firm have used woven copper wire for 12% to 14% hydrochloric acid with a life of 4½ months, while the same wire cloth was satisfactory with 30% sulphuric acid. Wool or felt cloths will generally resist from 15% to 20% sulphuric acid, but are of little use for hydrochloric acid.

Asbestos will withstand any strength of acid or alkali likely to be put into a filter press, but is apt not to resist handling, and, further, is difficult to obtain in a sufficiently finely woven form. The qualities vary a great deal, and it is well to make careful tests before buying in quantity. On the whole, for strong acids, a good camel's hair cloth, if treated properly, has much to recommend it. A great deal, however, is sold as camel's hair which is of quite other origin.

With regard to wire cloth, in one case which came under the author's notice a twill nickel cloth was used for removing sodium sulphite from mother liquor up to 50% causticity. A sample of such cloth, 360-mesh, appears quite impervious to light, except when held at certain angles.

Cloths should not be allowed to dry on the press when out of use, but should be rinsed free of corrosive liquors and retained solids and kept moist. The former will cause rotting if allowed to dry in, while the solids will become hard and impervious. Repeated drying and wetting accentuates shrinkage. In a well-woven filter cloth for dealing with materials such as dyes, the threads, though stout, should be packed so closely that they touch one another at every point. Then again the cloth should be as free as possible from parallel dark streaks or "reed" marks or lighter portions indicating missed or broken threads. Occasional dark specks on a cotton cloth do not, however, signify.

Some users prefer to have the cloths shrunk before they are put on the press. With a well-woven cloth with small shrinkage this is not necessary with centre feed, top feed, or passages in external lugs. Where, however, there are ports near the bottom of the cloth it is usually wise to shrink the cloths before use. It should be noted that

the holes can be cut and the press clothed while the cloths are wet. In some cases boiling water should be used, but this, however, is not usually necessary, although it would have the effect of causing a cloth to filter clearly when it otherwise might not do so. The question of shrinkage is a difficult one, and depends on the weaving, though it may be said that the less compact cloths may shrink anything up to 7 or 8%, while more closely woven ones may shrink as little as 2½%. Something depends, too, on the quality of the cotton and the quality of the weaving, and instances are not unknown where two cloths of apparently precisely the same quality, texture, and weave may shrink very differently. On the whole, such valuable cloths as camel's hair are best soaked in mother liquor at the correct temperature before being put on the press, as if shrinkage occurs the strain on the cloth the first time it is used may seriously impair its life.

In dealing with many food products, and also in cases where the cloths become rapidly clogged, it is necessary to remove them at regular intervals for washing. The ordinary type of washing machine (fig. 19) consists of a vessel or outer casing within which revolves a perforated drum in which the cloths are placed. This drum is fitted with beaters, and revolves three times in one direction and then three times in the other to prevent roping of the cloths. The washing medium may be a weak solution of soda which can be heated by live steam, or a solvent may be employed. Another form of this machine has an independently driven squeegee inside the drum which acts as a shelf or lifter for part of the time and during the remainder compresses the cloths against a beater fixed to the drum shell. Another type of washer (fig. 20) consists of a tank constructed in wood or iron. A

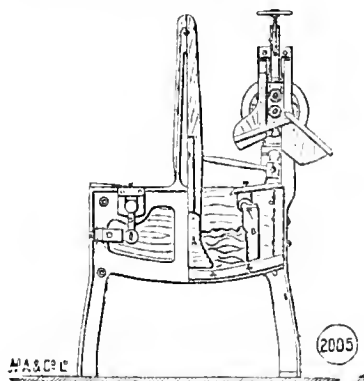


Fig. 20.

squeezing block runs the whole length of the tank, and is suspended from hinges fixed on pillars attached to the machine. There are false sides to the trough which are sprung in such a way that they can give a little to the action of the squeegee as it rocks from side to side. The cloths are placed concertina fashion on either side of the squeegee and are first compressed and squeezed against the side of the trough and then released, being allowed to fall over and open out, after which the process is repeated. Such a washer is excellent for squeezing out obstinate dirt with a minimum of wear on the cloths.

The simplest device is a tank fitted with a steam circulating injector. This is satisfactory with light cloths and readily soluble precipitates.

Some theoretical and practical considerations in regard to rate of filtration, etc.

The design and efficient working of a filter-press installation depends on the material, the treatment

desired, and various factors of a mechanical nature.

The questions relating to the material comprise:

1. Quantity per hour.
2. Chemical properties.
3. Suitable temperature for filtration, and viscosity of liquor at that temperature.
4. Percentage of solids.
5. Size, shape of grain, and hardness of suspended particles.
6. The permeability of the deposit as affected by pressure, time, and flow.
7. Nature of first deposit on cloth.
8. Does deposit bind on to cloth, or will it fall readily away?

In regard to treatment we must consider:—

9. What extent of clarification is desired.
10. Is the cake or the filtrate of most value, and is a firm cake to be formed?
11. Must the material be washed, and, if so, how thoroughly?
12. Is it to be air dried in the press?
13. Are any special steaming, heating, or cooling arrangements needed?

The mechanical and other factors include:—

14. Practical chamber thickness.
15. Practical maximum pumping capacity.
16. Effect of various influences on the length of the discharging period.
17. Relative cost of various arrangements.
18. General layout in relation to remainder of plant.

It will be seen that it is not usually practicable to obtain exact information on the whole of the physical data involved. Fortunately trials with a gallon or two of liquor will enable the following data to be settled:—(a) Suitable cake thickness; (b) working pressure; (c) cloth; (d) time to make a cake, or rate of filtration; (e) nature of aids to filtration, if required.

Usually one or two tests will elucidate these sufficiently to allow those with proper experience to approximate to the most perfect scheme.

We will now consider various factors in detail, commencing with a mathematical treatment of such as are amenable to it. This is helpful to clear thinking, for while nothing can be a substitute for experience, it is not always realised what influence such factors as cake thickness and length of press have on output and economical results.

Where the solids build up with a perfectly rigid granular structure, the rate of flow is proportional to the pressure. It also varies inversely as the resistance of the cloth etc. and that of the deposited cake. The former is fixed, while the latter increases throughout the operation, being proportional to the total flow and the percentage of suspended solids. Sperry defined unit resistance as that which gives a flow of one gallon per sq. ft. per hour under a pressure of 1 lb. per sq. in. In the case of the solids a deposit of 1 in. was assumed in fixing the unit. The rate of deposition was settled as being unity when one gallon of filtrate flowing through 1 sq. ft. left a deposit of 1 in., there being 1% of solids in suspension. On these assumptions he derived the formulæ:—

$$\frac{dQ}{dT} = \frac{P}{R_m + R_t} = \frac{P}{R_m + \frac{RQX}{K}} \quad \dots \quad (1)$$

$$T = \frac{RX}{2PK} + \frac{R_m Q}{P} \quad \dots \quad (2)$$

$$Q = \sqrt{\frac{2PKT}{RX} + \left(\frac{KR_m}{RX}\right)^2} - \frac{KR_m}{RX} \quad \dots \quad (3)$$

where P=pressure. Q=gallons discharged liquor.
T=time in hours. dQ/dT =instantaneous rate

of flow. X=percentage of solids in liquor. t' =thickness of deposit in inches. K=rate of deposition. R=resistance of solids. R_m is resistance of cloth. (Cf. Chem. and Met. Eng., 1916, 15, p. 198—203.)

These formulæ all apply to constant pressure conditions, and do not include the period of diminished flow during consolidation of the cake. Since, for any series of such tests P, R, and R_m are readily kept more or less constant, while X is apt to vary, we may write $A=R/2P$, $B=R_m/P$, and $N=K/2X$ where N is the number of gallons to form 1 in. of cake; i.e., to deposit a layer $\frac{1}{2}$ in. thick on each cloth. A, B, and N are very readily found by a trial filtration in an experimental press, and we may simplify the original formulæ as below:—

$$\frac{dQ}{dT} = \frac{1}{AQ + B} \quad \dots \quad (1a)$$

$$T = \frac{AQ^2}{2N} + BQ \quad \dots \quad (2a)$$

$$Q = \sqrt{\frac{2NT}{A} + \left(\frac{NB}{A}\right)^2} + \frac{NB}{A} \quad \dots \quad (3a)$$

Such formulæ apply with very fair exactness over the greater part of many time discharge curves, providing these are taken under constant pressure conditions. We shall be mainly concerned with number (2a), as (3a) is only required when the ques-

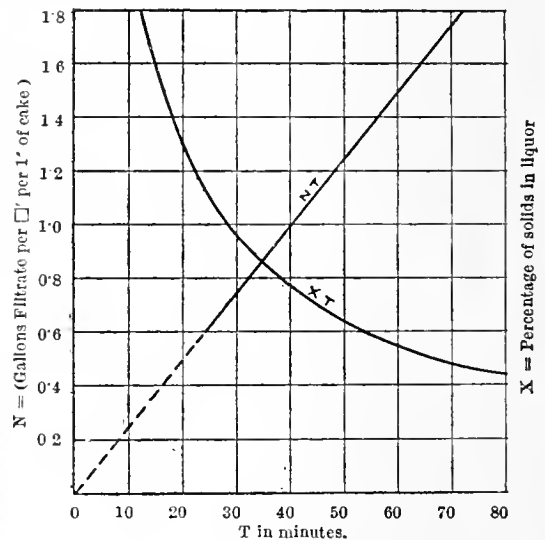


Fig. 21.

Relation between time to form a cake and the strength of the mixture.

tion of cake formation is of no importance. The rate of flow dQ/dT diminishes with increased flow, as is well shown by the diminishing curve (fig. 26) described later.

We are now in a position to develop the formulæ with a view to applying them to the solution of the relation of the average rate of flow to the dilution of the mixture; of that of cake thickness to output; and of length of press to capacity—both absolute and in relation to prime cost. For the sake of simplicity we shall not consider closely the effect of the time required for final consolidation, or for filling the press initially, where the pump is of limited capacity. Nevertheless, the simple outline below will illustrate sufficiently the general tendencies.

Let F, be the time to form a 1 in. cake, the amount of filtrate being N gallons by definition. If a cake

t inches thick is to be obtained, the amount of filtrate is $Q=Nt$, and substituting this in (2a), the time F_t to form it is given by—

$$F_t = \frac{AN^2t^3}{2N} + Nt$$
$$= N\left(\frac{At^3}{2} + Bt\right) \quad \dots \quad (4)$$

From equation 4 we see that the time to get a cake is proportional to N (or the volume of filtrate to make a cake) providing the thickness remains

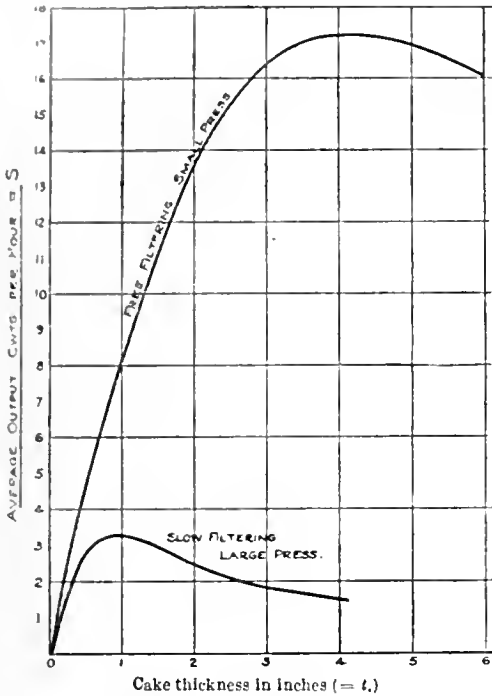


Fig. 22.

Relation between cake thickness and output for two extreme cases.

constant. It is not quite inversely proportional to the percentage strength of the mixture (although in many cases it may be nearly so) since a certain amount of liquid remains in the cake. Fig. 21 shows the theoretical relationship for a freely filtering material building a thick cake. The relation $N \cdot T$ is a straight line, while that of $X \cdot T$ is a curve giving values of T nearly inversely proportional to the value of X . The proportionality would be exact if X , in Sperry's formulæ, were calculated on filtrate, and not on total liquor. In practice, however, both these relationships may be somewhat altered by the greater consolidation of the cake when formed from weak mixtures, which may cause the time to form it to be lengthened out of proportion (*cf.* fig. 25 described later).

We will now consider the question of best cake thickness.

Substituting $M=AN/2$ and

$$m = NB, \text{ then}$$
$$F_t = Mt^3 + mt$$
$$F_1 = M + m$$

Let E be the time for emptying, which may be taken as constant for a given size of press, within reasonable limits of thickness. C is the weight of the cakes when 1 in. thick, and S the rate of

output over the whole operation, both measured in lb. per sq. ft. of area. Then

$$S = \frac{Ct}{F_t + E}$$
$$= \frac{Ct}{Mt^3 + mt + E} \quad \dots \quad (5)$$

S is a maximum with regard to t when $dS/dt=0$ (see fig. 22).

We have therefore

$$\frac{d}{dt} \left\{ \frac{Ct}{Mt^3 + mt + E} \right\} = 0$$

$$\therefore \frac{C(Mt^3 + mt + E) - Ct(2Mt + m)}{(Mt^3 + mt + E)^2} = 0$$

$$\therefore Mt^3 + mt + E - t(2Mt + m) = 0$$

$$t = \sqrt{\frac{E}{M}} = \sqrt{\frac{E}{F_1 - m}} \quad \dots \quad (7)$$

$$t = \sqrt{\frac{2E}{AN}} \quad \dots \quad (7a)$$

$$t = \sqrt{\frac{8EPX}{RK}} \quad \dots \quad (7b)$$

If we assume that the press is filled by a pump big enough to give the maximum initial rate of discharge, then the time taken to pump an amount of liquor equal to the volume of the press is proportional to t . In the above calculation all terms in t cancel out, and expressions (7) and (7a) are unaffected. The case is similar if we assume the cakes are washed at a fixed rate of flow. It may however be argued that there is a maximum washing pressure which should not be exceeded. In this case the time W_t for washing will be

$$W_t = t^2 W_1$$

where W_1 is the time to wash 1 in. of cake. Similarly it may be held that the time for final consolidation is approximately

$$k_t = t^2 k_1$$

where W_1 is the time to wash 1 in. of cake. Equation (5) now becomes

$$S = \frac{Ct}{(M + W_1 + k_1) \cdot t^2 + mt + E}$$

and the best thickness is

$$t = \sqrt{\frac{E}{M + W_1 + k_1}} = \sqrt{\frac{E}{(F_1 + W_1 + k_1) - m}} \quad (8)$$

$$\text{or } t = \sqrt{\frac{2E}{AN + 2(W_1 + k_1)}} \quad \dots \quad (8a)$$

Fig. 22 shows two curves drawn on the basis of equation (5) for certain conditions commonly met with. The existence of a best thickness for maximum output is clearly shown. In practice the use of this is limited on the one hand by the fact that if the cake is above a certain thickness it may consolidate and become impervious before it is fully formed. This will call for more work in emptying and drying. On the other hand, thin cakes will require more labour per ton, and so a balance of advantage must be struck.

Nevertheless the fact remains that for a material dealt with in a large press taking some time to discharge, thicker cakes are best if they can be formed. A small, rapidly discharged press will give most output with thin cakes if the filtration is fairly slow; if a thick cake can be formed rapidly it may however be satisfactory. This is entirely apart from any question whether thick cakes can or cannot be made. It is purely a matter of securing a proper balance between the filtering and discharging periods. It is also clear that if it is required

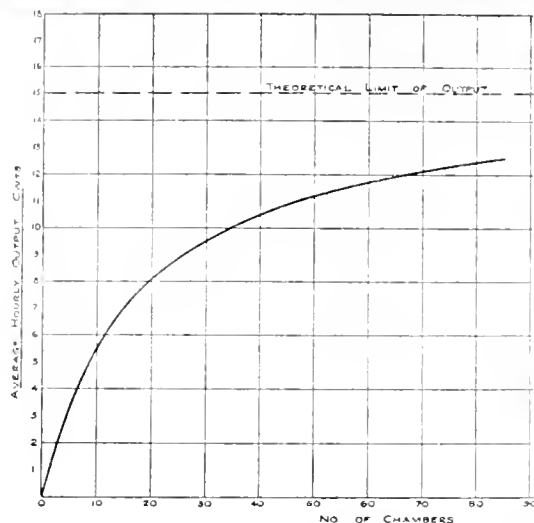


Fig. 23.

Relation of average output to length of press.

to wash the cakes at a fixed pressure, or if much consolidation is necessary, then thinner cakes are again desirable.

The cake thickness, however, is usually settled on practical grounds, determined solely by the thickest cake that can be made of suitable dryness, with a view to reducing labour costs. It is then of importance to know what length of press will give the best results. Let S_z be the average hourly capacity of the whole press consisting of Z chambers; C' the total capacity per chamber in lb. of cake; U the time in operating the closing gear per pressing, and V the time for emptying and re-closing one chamber. Then

$$S_z = \frac{CZ}{F_t + U + ZV} \quad \dots \quad (9)$$

Fig. 23 is plotted from this formula, and shows that after a certain length has been reached the addition of further chambers only slightly increases the output. This tends to reach a fixed limit, on account of the longer period the press is at a stand-still during each shift. If the cake is built quickly the press must be short or the discharging period will not be in balance. This will not increase seriously the labour cost, since this in the main is taken up in emptying and replacing the plates, only a small proportion being required for working the closing gear and manipulating the valves. It is only where the period of filtration (which in the present case may include washing and air drying) is very protracted, or the material can be emptied very rapidly, that it is economical to use a very large number of chambers. These considerations are strictly borne out in practice.

A further important question is that maximum output should be obtained per unit of prime cost. Let \mathcal{L}_z be the total cost of the press; \mathcal{L}_F the cost of the frame and heads, which includes the first chamber; \mathcal{L}_c the cost per additional chamber. Then

$$\mathcal{L}_z = \mathcal{L}_F + (Z-1) \mathcal{L}_c$$

The ratio of output to cost is therefore

$$\frac{S}{\mathcal{L}_z} = \frac{CZ}{(F_t + U + ZV)(\mathcal{L}_F + (Z-1)\mathcal{L}_c)} \quad \dots \quad (10)$$

Obtaining the maximum value in the usual way, we get

$$Z = \sqrt{\frac{(F_t + U)(\mathcal{L}_F - \mathcal{L}_c)}{V \mathcal{L}_c}} \quad \dots \quad (11)$$

Where

S =hourly output in lb.

\mathcal{L} =total cost of press. \mathcal{L}_F =cost of framework.

\mathcal{L}_c =cost of each additional chamber.

C' =lb. of cake per chamber. Z =number of chambers.

F_t =length of filtration period, including washing etc.

U =time in hours to work gear and valves per operation.

V =time in hours to empty and replace one chamber, including an average allowance for clothing.

Taking a 38 in. plate and frame press, we may assume that the time for working the closing gear, operating valves, pumps, etc., is 20 mins.; and for emptying, say 2½ mins. per chamber. Assuming 3 hours is occupied in getting a cake and substituting current values for \mathcal{L}_F and \mathcal{L}_c , we find the best number of chambers is 32. Hence large units would best be made up of this size press, though for installations of only a single press it might not be worth while to limit the size.

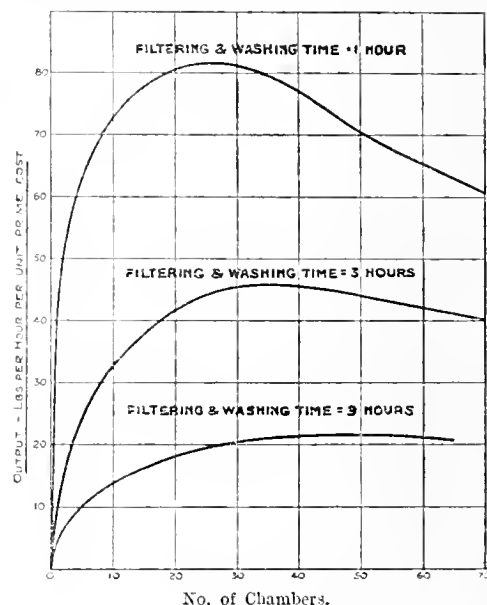


Fig. 24.

Relation of length of press to output per unit prime cost for a 38 in. sq. P. & F. cast-iron press.

Fig. 24 has been drawn on the above basis for three varying periods for filtration and washing. For the slower ones more chambers are seen to be economical, but the slope of the curve varies very gradually for the longer periods. Even when 9 hours are required for filtration we see that it is hardly worth while going over 40 chambers, though the best figure is given by calculation as 54. If discharging were slower, then shorter presses still would be indicated. This would also be the case with more quickly filtering liquors. Recessed presses, or those with wooden plates, would preferably be rather longer, as the cost per chamber is proportionally less. Should the closing gear be elaborate and slow to work, the presses would be best if slightly longer in the case of rapid filtration.

It is difficult to obtain sufficiently exact figures on which to base a similar calculation in regard to the most economical size of plate. The laws would, however, almost certainly follow the lines of figs. 22 and 24, and there would be a definite size for maximum average output, and for maximum output in relation to prime cost. The latter consideration would almost certainly indicate a somewhat smaller size than the former. The labour in discharging one chamber may be divided into (a) mov-

ing plate, (b) dropping the cake, (c) cleaning the cloths, (d) renewing cloths, (e) cleaning passages and ports.

Now (b) and (c) are fairly independent of the size of plate, (a) is more or less so up to a point, after which it increases very rapidly, (e) is directly proportional to the area, while (d) is more or less so, depending on the type of plate.

If the filtering period is long, larger plates will be economical, but in this case one would not expect to find the line of demarcation very strongly defined. If the cleaning of the cloth is the major part of the operation, this will tell against any

Nottingham. Fig. 25 shows a family of curves relating to spent lime suspensions of various strengths. The total discharge is plotted against time, each test being made with a frame 4 in. deep, at a constant air pressure of 70 lb. per square inch. Readings were taken every minute, and filtration continued till three constant ones were obtained. The law connecting time and flow is well illustrated. In the case of the weakest suspension this is $T = 1.19Q^2 + 0.8Q$ (or $T = 3.57Q^2/2N + 0.8Q$) where T is in minutes. The effect of the consolidation period is shown by the difference between the upper part of the actual curve, and the dotted line repre-

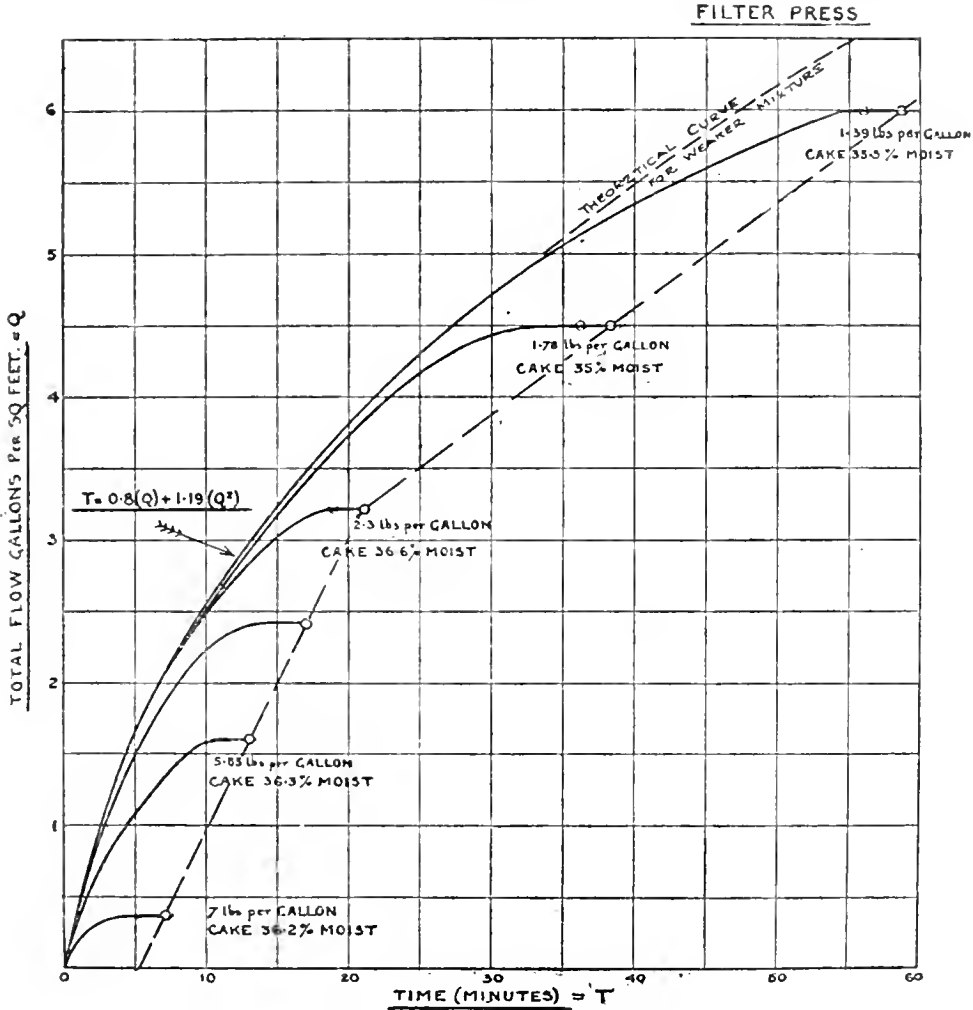


Fig. 25.

Filtration of caustic liquor containing various amounts of spent lime.
4-in. cakes, 70 lb. air pressure.

marked economy in the use of big plates, but if the cakes drop cleanly away, then the best results are likely to be obtained with large sizes. Wood plates, on account of their lightness are economical in larger sizes than iron ones. As a general rule 38-in. or 40-in. plates are quite large enough, and even these do not always show any marked reduction of labour as compared with the 32-in. size. The chief advantage of very large sizes usually lies rather in the saving of space.

We may now pass to a consideration of one or two test curves, taken in the small scale plant department of Messrs. Manlove Alliott and Co., Ltd.,

senting the theoretical one. The maximum reading in gallons for each case is, of course, proportional to N , and on reference to equation (4) we see that the time to get a cake should also be proportional. If this holds, all the curves should end on the same straight line passing through the origin. The four tests with stronger suspensions agree well with this, except that the line is displaced a little to the right. This is no doubt due to some residual consolidation effect. The curves for the weaker suspensions vary markedly from this line, taking longer in proportion. On inspection the cakes were distinctly dryer in appearance, and the percentage

moisture was found to be slightly less. Apparently the slower filtration was due to the fact that the lime particles had time to settle down more compactly; and it is probable that the smaller ones were gradually washed from between the larger (cf. fig. 28) to form a more compact and impervious layer near the cloth. The distinction between the two rates of flow is very marked, but further trials would probably show that they merge more or less gradually.

The tests on both the lime and the dye were made with the same cloth, and exhibit clearly how different are the resistances of the cakes, and how markedly the resistance of the cloth varies when the latter is used under altered conditions.

The permeability and dryness of a filter cake depend on the size and shape of the particles, and especially on their texture, whether firm or otherwise. Comparatively coarse, hard grains are favourable, as the capillary passages are large,

FILTER PRESS.

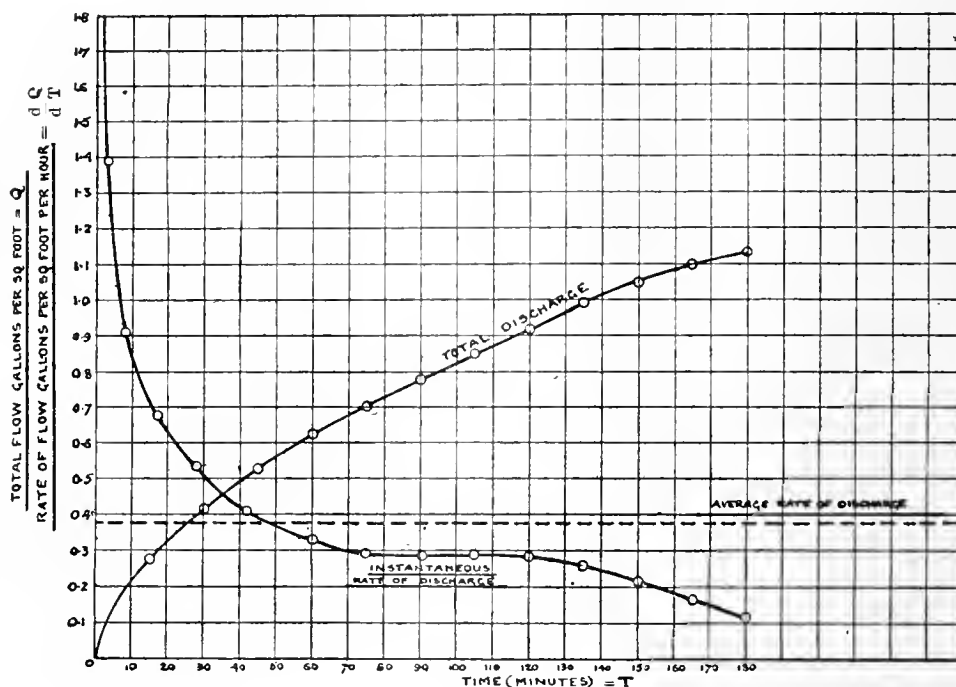


Fig. 26.

Time discharge curves for Direct Yellow.

Fig. 26 shows a time discharge curve for a sample of Direct Yellow recently tested by the writer. In this case a complete 1 in. cake was formed at a very early stage, though, of course, it was extremely soft. This was tested and confirmed by two separate trials, at the end of which the press was opened while still draining freely. The curve itself is the result of a third trial carried on as long as seemed worth while. In this case, although the conditions under which the solids are deposited are so very different from the former instance, the curve agrees well with $T = 115.4Q^2 + 25Q$ (or $T = 255Q^2/2N + 25Q$) right up to about 80% of its total flow. Curiously enough, where it departs from the formula, the curve shows a quicker rate of filtration, and this divergence may be due to some slight subsidence of the suspended solid matter, so that during the latter portion a comparatively weak liquor might have been fed to the press. The mixture was, however, kept agitated during the whole test.

On the same chart has been plotted a curve showing the rate of filtration, at every moment during the test, as compared with the horizontal line giving the average rate of flow over the whole period. Nearly half of the substance is fed in at a considerably higher rate than the average, while it will be seen how slowly the flow from the taps comes to an end as the cake is gradually consolidated.

and high pressure can be used. It is not, however, desirable that the grain should be so coarse as to cause rapid settling. If this should happen in the press a cake of uneven quality would be formed, which would be unfavourable for washing or air drying. It might be difficult also to secure a clear effluent, as is explained later.

A great part of the art of filtration lies in the control of the nature of the suspension. In this the microscope is a useful aid. Higher powers than $\frac{1}{2}$ in. objective, and 9 times eyepiece are not usually necessary, but for substances like anthracene or β -naphthol a polariscope is useful in rendering the crystals visible. If the particles are mounted in liquor they can be made to separate or turn over by pressing with a sharp point on the cover glass, and thus their real shape and the compactness of their grouping may be judged. By this method, for instance, it can be seen quite clearly that β -naphthol particles consist of large, though very thin, flat plates, while particles of anthracene in suspension in oil, having very much the same appearance while still, are really somewhat irregular nodules which are more or less of equal shape when viewed from any angle.

Fig. 27 shows the previous sample of calcium sulphate magnified to 250 diameters. It will at once be seen what a coarse open meshwork will be built up in the cake. Such a shape and size of crystal is, of course, extremely free filtering, and

is very favourable to good washing results. The needles shewn run 0.001 in. in length, or even larger, and the breadth is about 0.0001 in.

Fig. 28 shows on the same scale a suspension of calcium carbonate. Many of the crystals have

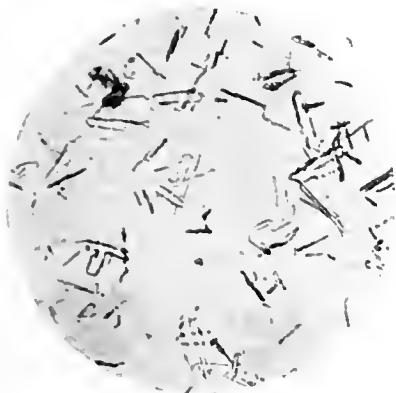


Fig. 27.
Sulphate of lime ($\times 250$).

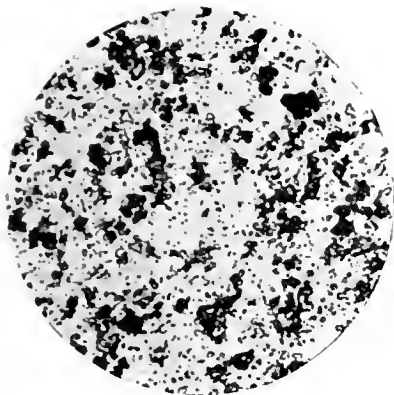


Fig. 28.
Spent lime ($\times 250$).

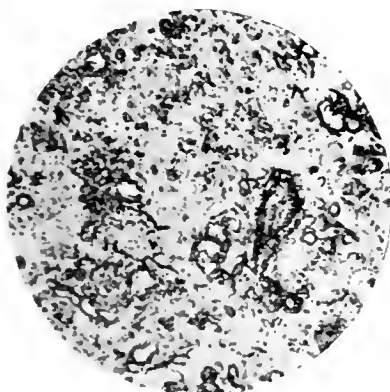


Fig. 29.
Sludge from soap lye ($\times 250$).

aggregated to form comparatively large firm lumps, but the scattered particles will give a fairly good idea of the size and shape of the individual unit. Numbers of these are only 0.0001 in. in diameter, while some are even less. This material

is, however, quite free filtering, as will have been gathered already. This is due to the fact that the particles are hard and angular, with no trace of sliminess or gummy matter, and the aggregated clusters are sufficiently firm and numerous to keep



Fig. 30.
Benzopurpurin ($\times 250$).



Fig. 31.
Direct Yellow ($\times 250$).



Fig. 32.
Grey filter paper ($\times 250$).

the cake open. The precipitate from soap lye presents quite a different appearance (fig. 29). Even in the photograph the shapeless outline and scummy nature of the particles are indicated, suggesting good results would be secured by not too

high pressures. Fairly rapid filtration may, nevertheless, be obtained, and probably this is due to the comparatively large size of the particles concerned. A somewhat different appearance is presented by the various aniline dyes. In the case of the substances previously mentioned, all the particles appear to be at rest, but in the case of dye-stuffs a number are often to be seen in motion. If, for instance, Benzopurpurine is examined in suspension in water, assuming the preparation has been freshly made up or recently agitated, each particle would seem to be separate, assuming a thread-like form 0.0002–0.0001 in. long by about one-tenth of this in diameter. Some preparations are even smaller, while the threads are not rigid but curl and uncurl in addition to having a distinct oscillatory or Brownian movement. After a long period of rest all movement will have temporarily subsided and the separate particles will have massed themselves together in such a manner as shown in fig. 30. Filtration at comparatively low pressures is clearly indicated.

The agglomerated and motionless condition is the more favourable to good working. The particles may also be seen to be of somewhat larger dimensions than when they are scattered and freely moving. Since this state can be brought about by the presence of an electrolyte such as salt, it is good to retain a suitable proportion of this if market conditions, etc., will permit. Naturally cake formation is apt to be slow at best with deposits of this type, and closely woven cloths are necessary. Washing is usually out of the question. This is partly due to the high resistance of the cake, and to the fact that as soon as water penetrates the mass it removes any electrolyte present, and the dye particles then assume a still more intractable nature.

Fig. 31 shows a sample of Direct Yellow. This gives a freer flow and is more readily retained by the cloth, as is indicated by the large size of the particles and the way in which they are massed. Fig. 32 shows a sample of filter paper on the same scale for comparison.

Before leaving the subject of quality of grain, it may be of interest to mention a curious property of many filter cakes, due to the shape and hardness of the particles. Such cakes appear firm and comparatively dry and free from surface moisture when first discharged. If a small piece is shaken in the hand it becomes sloppy, and a distinct amount of free surface moisture appears. This is due to the fact that the particles are deposited initially in any order, but when shaken assume their most compact grouping, causing the moisture in the interstices to appear at the surface. Further, as was shown by Osborne Reynolds (Scient. Papers Nos. 50–51), such hard material does not necessarily assume its most compact grouping under the application of pressure, providing this is not sufficient to crush the grain. This supposes the existence of an elastic boundary, which is provided by the cloth. This property Reynolds termed "dilatancy" (fig. 33). It is so marked that if a football bladder is filled with fine sand and water, and fitted with a glass tube in the neck, excess water will not be forced out, but is actually seen to be drawn into the bag when the latter is squeezed.

Some liquors contain particles so fine that they cannot be retained on the usual filter cloth, unless of a very close texture, while in other cases, such as sugar scums and some varnishes the liquor is so slimy that an impenetrable layer is speedily formed on the cloth and it becomes impervious. In such cases various aids to filtration can be used. The requirements for such an aid are as follows:—

- (1) It must form a readily permeable deposit.
- (2) It must not be difficult to keep in suspension.

(3) The capillary pores between the particles must be sufficiently fine.

(4) If very fine scummy particles are to be removed, it must have a maximum of surface, and its nature must be such as to favour adsorption.

(5) It must have no injurious action on the material being filtered.

Among such substances may be mentioned calcined fuller's earth, diatomite, and various other refined fossil earths, large crystals of calcium sulphate, charcoal, wood meal, cotton meal, etc. In some cases it is actually possible to produce a lime precipitate in the liquor before filtering, with the result that the slimy particles are covered with a lime deposit and the cake rendered more open and permeable.

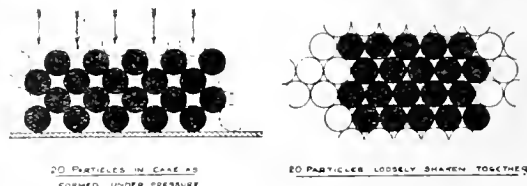


Fig. 33.

In this connexion it should not be overlooked that the coarser particles in a precipitate may assume the nature of a filter aid towards the smaller ones, which would not be retained by the cloth if the whole precipitate were fine. Hence it may be difficult to get clear filtrates if the larger particles are allowed to settle out in the filter press owing to insufficient pump capacity, etc.

For fine clarification the cloth may be given a very thin coat of the filter aid by pumping in a suspension of the latter before commencing to filter. It is also useful when mixed with the liquor, as it not only helps to give a clear filtrate, but builds up an open structure in the cake, thus permitting a more rapid flow where slimy matter has to be removed.

The following weights of dry solids per cubic foot of press capacity—together with the moisture in the cake—may be of interest. They are all taken from ordinary large scale practice. They should, however, be regarded only as a guide, as very different results are sometimes obtained between one factory and another. Those moistures marked with a * have been reduced by means of blowing air through the press:—

Material.	Dry weight per cb. ft.	Moisture, %.
Orange chrome	340	5
Zinc chrome	200	7
Lemon yellow chrome	46	55
Para Red	53	30
Prussian blue	15	60
Magenta lake	20	60
Wolfram tin fines	136–160	10–17
Tungstic oxide	104	14–21
Spent lime	57	38
Calcium sulphate	42–55	40
Anthracene	29	46
<i>β</i> -Naphthol	47	30–40
Sulphur black	42	40
Sulphur blue	59	32
Wool red	19	60
Direct brown	35	50
Benzopurpurine	38	35–40
Congo red	42	50
Triacid violet	23	30–35
Orange II	20	40–45
Sewage sludge	30	60
Sludge from soap lye	30	60

Cake thicknesses vary from $\frac{1}{8}$ in. up to 4 in. or even 6 in. in the case of very permeable deposits. Some samples of calcium sulphate and carbonate will build up to the higher figures. In many cases 2 in. is considered a good thickness, even for the above substances. Colours and dyestuffs are usually

filtered in presses having chambers 1 in. to 1½ in. thick. Materials are not unknown which will build a layer only a few hundredths of an inch in depth. In such cases cake formation is impossible, and the excess liquor has to be drained out of the press.

It is well worth while for users to study and test the best pressures for feeding their particular grade of material. Too many operators are satisfied that things are right so long as a clear effluent is obtained, and it is not realised that better results are often obtained by relatively low pressures, except perhaps in the last stages of the run. In relatively few cases does high pressure produce proportionate results either in rate of flow or weight and moisture of filter cake. In fact, it may reduce the rate of flow or stop it altogether by destroying the open structure of the deposit.

½ gall. per sq. ft. per hour or even less. Some vegetable oils have rates of flow varying from 1 to 8 galls. per sq. ft. per hour, but in other cases these figures may be halved. In one case soap lye after acidification filtered at the rate of 4 galls. per sq. ft. per hour, and the same liquor after neutralisation gave a flow of about double this amount. This filtration was carried out at a pressure of about 20 lb. per sq. in., never exceeding 30 lb., whereas it is sometimes considered that pressures of 100–150 lb. are necessary for this material. In another case the lye was filtered only after neutralisation, and the rate of flow was 5½ galls. per sq. ft. per hour under a gravity head of only 20 ft.

In the case of sewage, rates will vary from 3 galls. down to ¾ gall. per sq. ft. per hour, the usual figure being about 1½, and the pressure

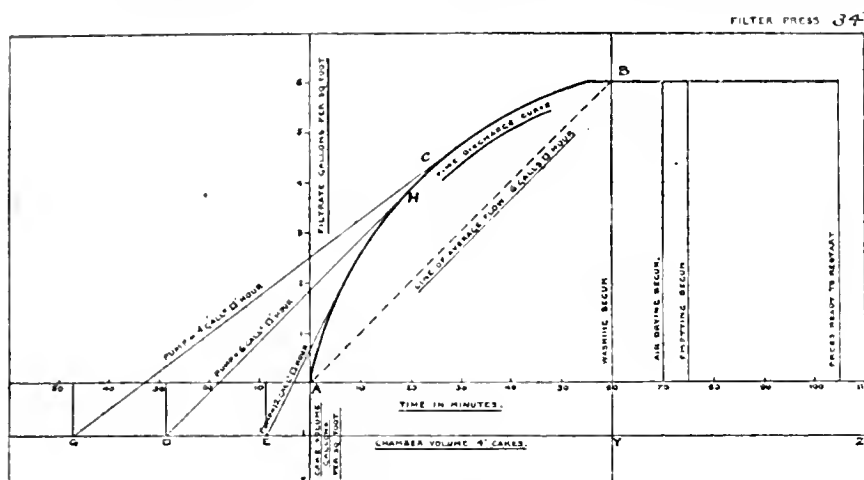


Fig. 34.

Correction of time discharge curve for cake volume and pump capacity. Rapid filtration.

In ordinary filter press work pressures may range from, say, 150 lb. per sq. in. down to 20 ft. gravity head or even less. The higher pressures are only suitable for very viscous liquors containing hard solids of a hard granular nature. Where viscosity is small pressures of 70 to 100 lb. are usually ample for granular or fibrous suspensions, while many materials of only a tolerably open texture are dealt with very satisfactorily at 40 to 60 lb. Dye-stuffs, colours, etc., and other materials of a very fine nature are often advantageously handled at half this pressure or even less. Prussian blue, for instance, is generally dealt with at 30 ft. gravity head.

Even when higher pressures are justifiable it is well to start the operation at a comparatively low pressure in order that the first layer of the cake may be open and porous. For slimy materials the preliminary feed may well be under a low gravity head. Once a good layer has been formed there is a tendency for the particles to interlock, and they are then less liable to be forced through the cloths by a higher finishing pressure.

Rates of filtration are very varied. They are highest where large quantities of liquor containing small amounts of solids are being handled, providing that it is not necessary to form a cake of high solidity. In such cases it pays to clean the press out as soon as the rate of flow falls off. In clearing a watery liquor from impurities and dirt, rates of from 15 to 30 galls. per sq. ft. per hour are often obtainable. On the other hand, viscous materials, such as varnish, may only give a flow of

80–100 lb. per sq. in. For the majority of dyestuffs the range is about 2 galls. per sq. ft. per hour down to ½ gall., and in very difficult cases one gets down to a flow as little as one-tenth of the latter amount.

Methods of Feeding the Press.

Fig. 34 illustrates one method of fixing a suitable pump capacity. A time-discharge curve, ACB, is obtained in the usual way, the dotted line, AB, representing the total average discharge. It is presumed that the original curve is obtained experimentally from a small-scale test, in which case the rate of flow is generally determined entirely by the press and not by the pump. It is further assumed that the substance is one in which it is permissible to use pressures rapidly approaching the maximum in the early stages of the test. A curve obtained in this manner needs to be corrected for the volume of the cake itself and for the employment of a pump of a practical size. In the instance given, AB corresponds to the steady discharge of a pump having a capacity of 6 galls. per hour per sq. ft. of filter area. The volume of cake is 1.04 galls. per sq. ft., and a line GY is drawn to represent this at a suitable distance below the horizontal axis. The line DU is parallel to AB and tangential to the discharge curve. It represents the steady flow of the pump, pumping up to the maximum pressure at which the relief valve is set, which is reached at point H. The distance of D from the vertical axis represents the extra time

per operation when a pump of this capacity is employed. The position of the point E gives a similar figure for a pump of double this capacity, and G for a rather smaller one. The times for washing, air-drying and emptying the press have also been added to the chart, and this enables a reasonable judgment to be made as to what correction should be allowed for the pump. On rates of filtration thus obtained the most economical size of press and pump may be calculated, due attention being given to the effect of any variation in the size of press on the discharging period.

Such a diagram is useful in so far as it gives a clearer perception of the various problems which enter into the determination of pump capacity and total output. In some cases, where filtration is free almost up to the last moment, and the pump is not too large, it is possible to force the liquid through at the full capacity of the pump during the whole filtration period, thus avoiding waste flow through the relief valve. This generally entails

of pipe between the press and any of the valves are to be avoided and the pressure relief valve is best brought up near to the feed valve where possible to avoid clogging or settlement in the feed pipe during the final consolidation period.

In addition to the pump connexions, steaming valves have been indicated both on the feed inlet and wash water outlet passages together with an air connexion for blowing air into the cake and a drain connexion on the bottom passage to serve as an outlet for any liquor blown off by this means.

The diaphragm type of pump (figure 36) is a very good method of dealing with thick sludges and is also useful for acid liquors, as it lends itself to coating with lead or vulcanite or other materials on all parts except the rubber diaphragm.

A single-acting pump is often a necessity for small duties, but it is really the worst type, as even with a large air vessel pressures are apt to be fluctuating. For larger duties double-acting, or better still, treble-acting pumps, are desirable.

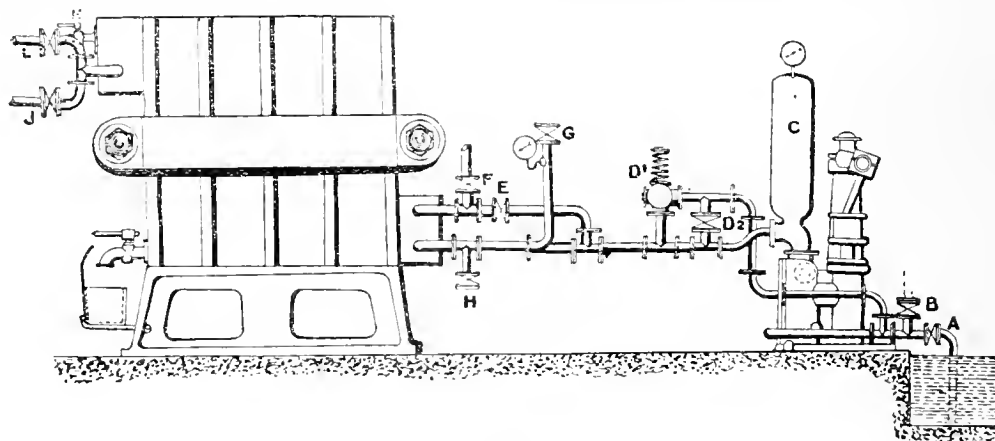


Fig. 35.

Diagram of connections on press head.

- A. Liquor to Pump.
- B. Water to Pump. (Only if connexion G is unusable).
- C. Air Vessel and Pressure Gauge.
- D^a. Automatic Relief Valve.
- D^b. Hand Pressure Regulating Valve.
- E. Feed Valve.

- F. Steam, Air, or Feed Washing Valve.
- G. Wash Valve from Service Main.
- H. Drain for Air Blowing.
- J. Wash Water Outlet.
- K. Air Release Tap (for washing).
- L. Air Blowing Valve (for drying).

somewhat slower filtration than might otherwise be obtained.

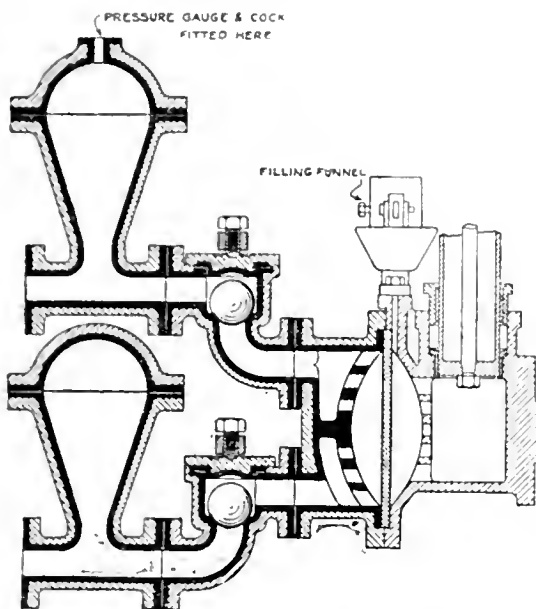
Fig. 35 shows the full connexions of a thorough washing filter press fed by a vertical plunger pump. It will be noticed that the air vessel is much larger than usual in order to minimise pressure fluctuations. In filtering sensitive materials with a single-acting pump this point should not be overlooked. A pressure gauge is desirable, as it enables the operator to see whether the pump is working properly and the pressure is being put on at a suitable rate. An automatic relief valve, set at the maximum pressure, is placed on the pipe between pump and press, and the liquor from this returns into the pump feed connexion. A re-circulating valve, worked by hand, is sometimes provided so that the first layer can be placed comparatively slowly on the cloths. Connexions are also shown for pumping wash water into the press, and in this case it is necessary to flush the pump and connecting pipes before washing. It is preferable, however, to have separate arrangements for the water supply, as the best pressure for washing is not as a rule the pressure at which the liquor should be fed to the press. Generally speaking, more than enough pressure will be got from the water main or even from an overhead tank. In practice dead lengths

Special pumps are also constructed in such a way that the effective stroke is decreased with increasing pressure, thus diminishing the rate of flow and causing the pressure to be put on gradually. The duplex steam-driven type of pump is a good pattern, as it gives a reasonably even feed and is to a large extent self-adjusting to meet increasing pressure. Even this type is generally fitted with a relief valve and large air vessel. The ram should be of the outside packed pattern where packings are likely to require frequent attention.

Undoubtedly the best way of charging the press, where gravity feed is not applicable, is to use an egg and force the slurry in by compressed air. Unfortunately this method is expensive except where a large number of presses have to be supplied or duties are large. Valve troubles are avoided, the flow adjusts itself automatically, and the pressure is perfectly even. On starting to filter the full initial flow is obtained at low pressure with a minimum amount of air. If several batches are necessary to fill the press it is only the latter ones which need air at the maximum pressure.

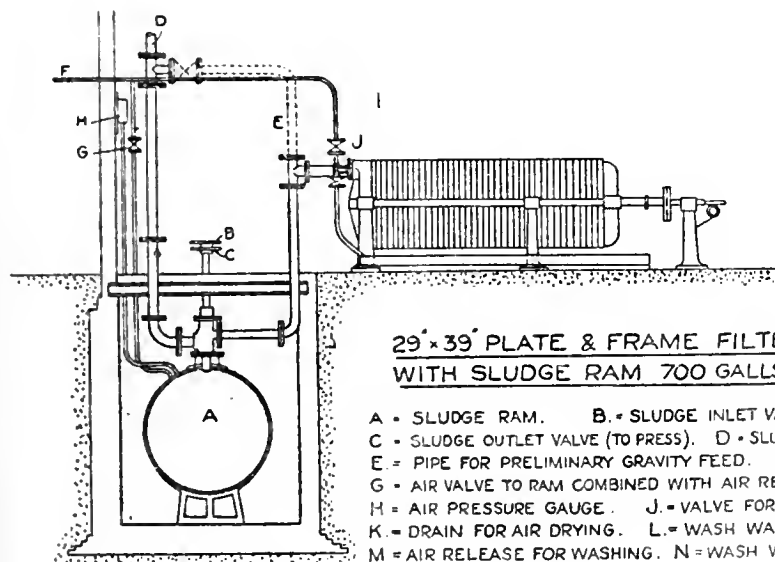
Figure 37 shows a wood press fed by a ram situated in a chamber below the floor. The feed pipe from the supply tank on the upper floor has a connexion to the press for initial filling by gravity,

thus economising air. For heavy sludges the air inlet pipe may be taken to the bottom in order to give a stirring effect, though for very heavy precipitates an agitating gear should be fitted. Rapid filtration in early stages is very valuable in the case of materials having a strong tendency to settle.



Sectional arrangement of vertical diaphragm pump.

Fig. 36.



29' x 39' PLATE & FRAME FILTER PRESS
WITH SLUDGE RAM 700 GALLS CAPACITY.

- A = SLUDGE RAM. B = SLUDGE INLET VALVE (TO RAM).
C = SLUDGE OUTLET VALVE (TO PRESS). D = SLUDGE SUPPLY PIPE.
E = PIPE FOR PRELIMINARY GRAVITY FEED. F = AIR MAIN
G = AIR VALVE TO RAM COMBINED WITH AIR RELEASE
H = AIR PRESSURE GAUGE. J = VALVE FOR AIR DRYING.
K = DRAIN FOR AIR DRYING. L = WASH WATER INLET
M = AIR RELEASE FOR WASHING. N = WASH WATER OUTLET

Fig. 37.

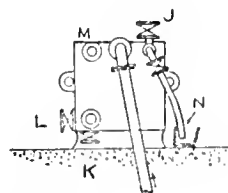
capacity of their press during the earlier stages. This is an important point to consider when the best washing results are desired.

In a case which recently came under the writer's notice feeding with a small pump gave cake which could not be washed below 2% soluble matter with any amount of time or wash water, but when a ram with agitating gear was installed the cake could be washed to under 0.2% with but little trouble.

Washing in the Filter Press.

Washing can be carried out with great advantage in the filter press. The simplest method is to force water in through the feed passage. This is best done in the plate and frame type, and it is preferable that a solid cake should not be formed, but that a space should be left between the layers built up on each cloth. Here a bottom feed is advantageous, as the strong liquor can be drained from the chambers prior to washing, though in the ordinary type it can generally be removed by air pressure. It is possible to wash this way in any plate and frame press or even in recessed presses, although they may not have been specially designed for the purpose. If a solid cake is not formed it is the only practicable way except re-lixivation.

The trouble is that owing to sedimentation the cake is apt to be thicker at the bottom, and in order to keep the two sides from touching space may be wasted at the top. Further, the press capacity is diminished and more labour is necessary in emptying. In some cases, however, solid cakes can be rough washed if they are sufficiently permeable. General reports from users appear to show that more wash water is requisite, but this would depend very much upon the manner of the operation. This method of washing is usually referred to as "simple" washing. Another consists in forcing the water from one side of the cake to the



ment, and in such cases where the grains are not uniform it will be found best to instal a large egg or receiver, in which the whole batch can be thoroughly stirred up and forced rapidly into the press. This method will give a more uniform and permeable cake than would be formed by a pump, more especially as most users are averse to paying for a pump sufficiently big to take care of the full

other, and may be referred to as "through" washing. It is also called "back" washing, as the water is admitted to the back of the cloths.

Fig. 38 shows this method applied to recessed plates. The wash water inlet passage forms a continuous channel in an upper corner of the rims. It has inlet ports to each alternate plate, on which the filtrate cocks must be closed during washing.

The water enters behind the cloths, passes through these and the cakes to the back of the cloths on the intermediate plates, the cocks on which have been left open to permit it to escape. This is sometimes called "tap washing," and its chief merit is its

Then, again, the washings should be taken out at the top of the press, so that there is exactly the same pressure tending to force the water through the cake whether this be measured at the top or at the bottom. The importance of these factors

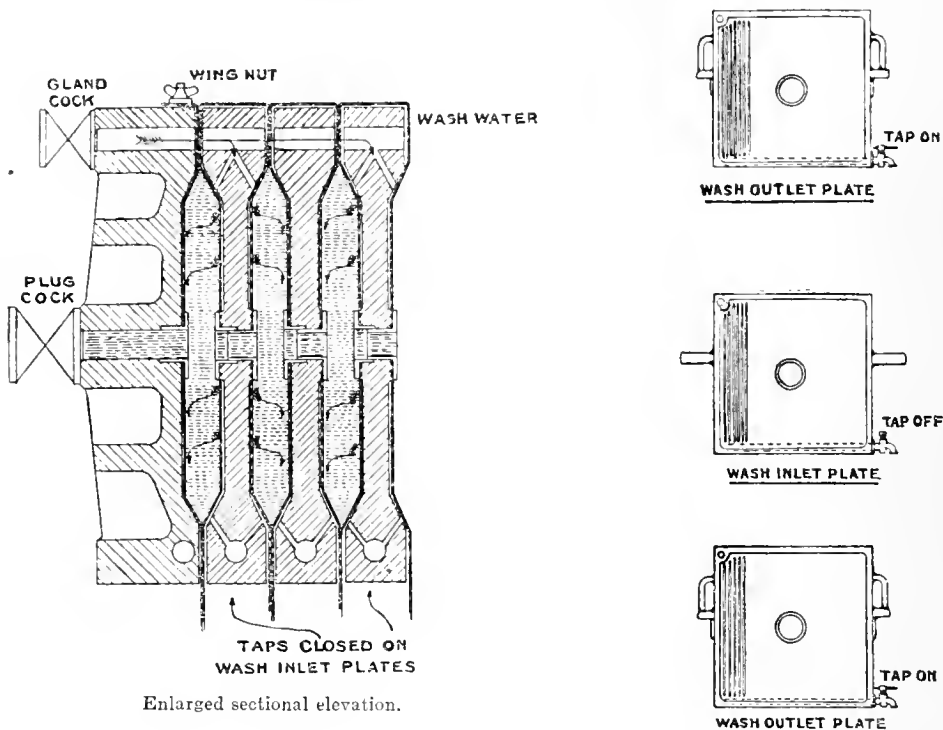


Fig. 38.

Recessed plates. Washing type.

simplicity, as only one special passage is required. One disadvantage of an internal feed is that a soft core will permit shortcircuiting, while a hard one may not get washed properly. For the very best results plate and frame presses should be employed, owing to the even thickness at the cake at the edges and the fact that any soft material in

will be better understood when it is realised that comparatively low pressures are best for washing purposes, as will be seen later. Except where due provision has been made, time will be required under such conditions to force trapped air out of the press, and in addition any unbalanced hydrostatic head (due to unrestricted draining on the

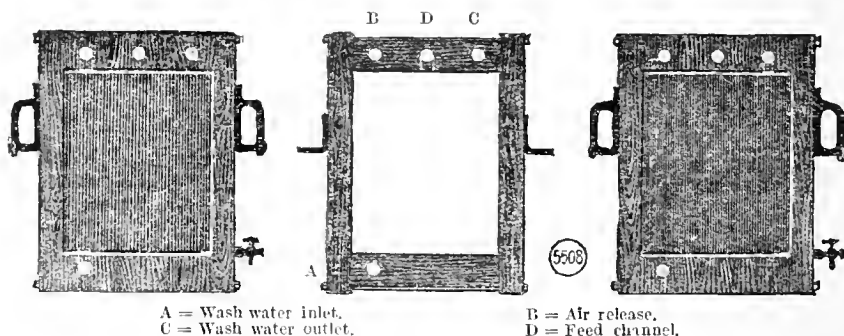


Fig. 39.

the feed passage is isolated. Further, provision should be made for the escape of any air which may have leaked into the space behind the filter cloths at the conclusion of filtration, as this may prevent the water flowing through the upper portions of the cakes. Even ordinary "tap washing" is distinctly improved by the provision of air cocks.

outlet plates) will aid in causing a disproportionate amount of wash water to percolate through the bottom of the cakes.

A filter press in which all these points have had attention may properly be described as a "thorough" washing press (fig. 39). The illustration shows wood plates and frames constructed on

this system. At the conclusion of pressing all the filtrate taps are shut off and wash water is admitted to the enclosed channel at the bottom. This passage connects by ports to every alternate plate and permits the water to rise behind the cloths on these, driving the air before it into the special air channel which connects with the wash inlet plates only. As soon as there is any flow from this

This permits plain cloths without holes to be used, and there is no difficulty in fitting and keeping them in place. The joints on the lugs are often made by cloth sleeves, which are slipped over them. These must be changed with the cloth, and for that reason rubber joint rings are often preferred. These may be let into special grooves around the wash passages, or may fit inside as shown in fig. 41.

PLATES WITH EXTERNAL LUGS

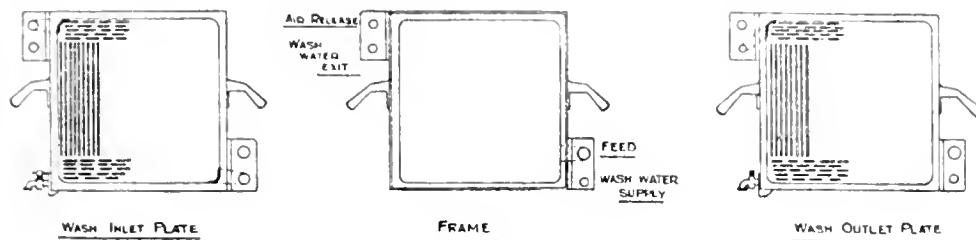


Fig. 40.

it is shut off, and the water must then pass horizontally through the cakes and cloths to the intermediate plates, where it rises in the grooves till it escapes into the enclosed outlet at the top. In this way provision is made for as perfect a displacement as possible of the strong liquors in the cake. An even better arrangement would be to have wash outlet cocks fitted on the tops of the plates, deliver-

Hydraulic lipped rings are shown, but quite good results may be obtained with plain rings, which should be just a push fit inside the channels. Rubber is not always permissible, and a more expensive press is required than if cloth sleeves are used.

In any event the cost of outside lugs is only justified where the filtering medium is subject to frequent washings or renewals. Normally the cloth is kept in place by adherence to the plates once it has been used, so that even where passages are in the rims no difficulty is experienced in keeping the holes in their right positions. If the cloths last a reasonable time and proper arrangements are made for cutting the port holes, the cost of this is small in proportion to the work done. A useful type of plate has two external passages in lugs which form part of the handles (see fig. 42). One of these is a feed passage, the other the wash water inlet passage. This form permits the outside lug plate to be made more cheaply, and in a form very convenient for manipulation.

Now, assuming that a press has been selected with all facilities for washing, as described above, and care has been taken that the filtering surface has been kept free and unchoked, giving the wash water access to every part of the cake, there are

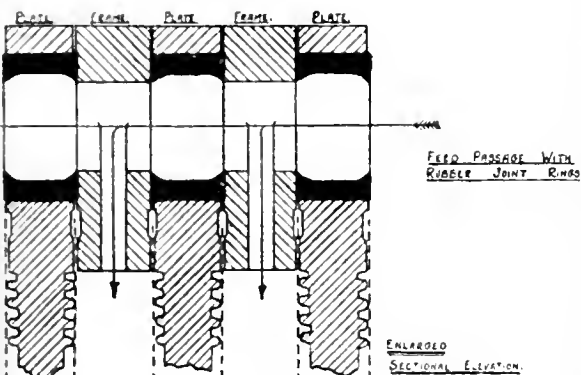


Fig. 41.

ing into a special trough, as this would permit any chamber giving muddy washings to be shut off at will. In general practice, however, enclosed outlets are found more convenient and perfectly satisfactory.

An interesting point about presses of this type is their extreme adaptability. There are a dozen possible variations of the washing connexions alone, since any passage can be used as an inlet, without counting possibilities in regard to steaming, air-drying, etc. The chief methods of washing are, however, ordinary thorough washing as above; washing in reverse direction, either through the enclosed outlets or through the taps and ordinary simple washing as first described. The wash water and air passages can be used as enclosed filtrate outlets. It is obvious that air, steam, or water may be admitted to the wash passages when the press is empty, but closed up ready to restart. By this means the cloths are given a reverse washing, and may be made to last longer before clogging.

In the case of iron plates, these wash passages are made either in the plate rims or can be with advantage in external lugs (fig. 40).

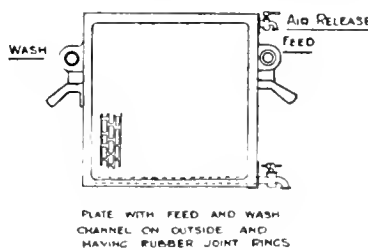


Fig. 42.

still other factors essential to satisfactory results. These are:—

- (1) Care must be taken to secure a well-formed cake.
- (2) The cake must be of as coarse a grain as is possible, without causing settlement in the press, and the various grades of particle must be uniformly distributed.

- (3) The washing pressure must not be too high and should not fluctuate. It should be applied as quickly as possible after the cake has been formed.
- (4) Care must be taken that the press is worked properly and especially that the air-taps are made use of.

Economical washing depends on even displacement, and good practice results when this ideal is approached as nearly as possible. If the cake is not complete, obviously there will be gaps, through which water can pass. Further, if the deposit is soft and not properly compacted such gaps may readily be formed during washing, while if this operation is delayed some cakes tend to sag a little and do not fill the chamber up to the top. The frames may be fitted with an internal rib or fin to counteract the effect of this, but some users consider there is more difficulty in cleaning out the cake. Then if the grain is too fine it will be difficult to get the wash water to penetrate without undue pressure, which may cause channelling and short circuiting. In such cases adsorption effects are also likely to hold back soluble matter in spite of even the most perfect washing. If the grains are reasonably coarse they will be more permeable, and will have less surface to retain strong liquor. A truer and quicker displacement is then likely to result. If they are not of fairly uniform size, the coarser ones will probably have settled to the bottom of the chamber, and the wash water will pass in preference through them, giving mixed or weak washing. If the particles themselves are porous, time will be needed to extract the absorbed soluble matter, quite apart from surface effects.

Finally, high pressures should only be applied with caution, as these may cause channelling and short circuiting. The lowest which will give a reasonable flow should therefore be used. The supply must be steady, and a gravity feed or a connexion to the town water supply is usually preferable to the use of a pump, where either can be employed. While the best results are usually obtained at pressures much less than those in general use for consolidating the cake, it should not be overlooked that the filtrate has only to penetrate half of its thickness, while the wash water must pass through the whole depth of the consolidated mass. In order that washing may not be too prolonged, the best cake thickness is usually somewhat less than for plain filtration. Good supervision is necessary, as one often sees presses provided with all the appurtenances for scientific washing being handled in a most unintelligent fashion. The author has seen thorough washing presses with practically every valve and tap open except the air cocks, while wash water was being admitted behind the cakes and into the feed passage at the same time. Needless to say, in such an instance every ounce of pressure that could be got out of the mains or wash pump would be applied.

Fig. 43 shows some tests carried out by the author on the washing of very weak caustic solutions from spent lime cake. It will have been realised from the photomicrographs that many of the particles in such a cake are extremely small, and consequently there is plenty of surface to hold back traces of alkali. Nevertheless, in these experiments almost theoretical results were obtained, the strength of the washings keeping right up to or quite close to the original strength till a volume equal to that of the liquor in the cake had been displaced. It will be observed there is still a fair strength of washings coming off even after this point has been reached, this being doubtless due to residual liquor behind the cloths and in various passages immediately connecting with the press. Adsorption, and possibly capillary effects in the case of the clusters, doubtless play their part. The

tests were made with a 4-in. cake at 20 and 40 lb. per sq. in. and a 2-in. cake with 20 lb. per sq. in. respectively. Not a great deal of difference is observable in the form of the curves. They all fall practically down to the specific gravity of the pure wash water after an amount equal to the total volume of the cake has been employed.

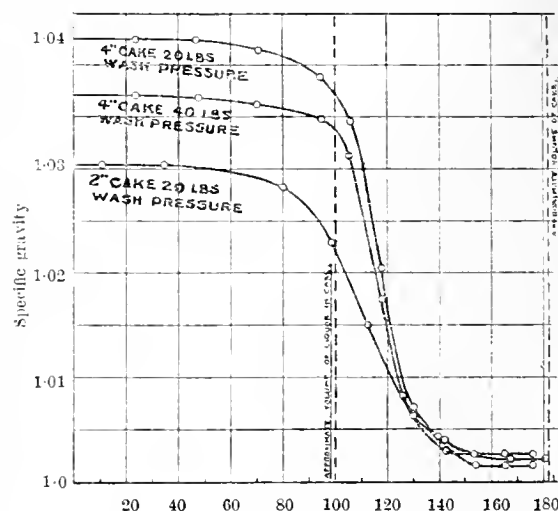


Fig. 43.

Washings calculated as percentage volume of mother liquor in cake.

Washing weak caustic soda solutions from carbonate of lime cake (experimental).

Of course such results can hardly obtain in large-scale presses unless a good deal more care than usual is observed. In dealing with carbonate of lime one firm reports the use of about $1\frac{1}{2}$ volumes of wash water (compared with cake volume) at 15 lb. pressure to wash cakes $1\frac{1}{2}$ in. thick and 24 in. square, the residual free alkali in the cake being 0.2% or slightly under on a fair average of the cake. The time for washing was about ten minutes. In another case sulphate of lime cakes 2 in. thick were washed free from all but a trace of a certain organic salt with $1\frac{1}{2}$ volumes of washings. In dealing with gold slimes cakes about 36 in. square by 1 in. to $1\frac{1}{2}$ in., of practically pure silica, were washed free of cyanide solution with about $2\frac{1}{2}$ volumes of water compared with the volume of liquor in the cake. Sugar scums, filtered by the aid of kieselguhr or lime admixtures, would require about $1\frac{1}{2}$ to $1\frac{1}{4}$ cake volumes.

If the deposit contains slimy or colloidal matter in any quantity, and the particles are fine, much more water in proportion will be required and the washing time will be protracted. Certain classes of dyestuffs cannot be washed for the reasons previously stated when discussing the photomicrograph of benzopurpurine (see p. 276 r).

Fig. 44 shows the relation between strength of washings and time taken in freeing from soluble matter a carbonate of lime cake contaminated with colloidal silica, which tended to wash out on to the cloths and choke them. In this case it will be seen that to economise water washing has been carried out in three stages, one with a strong liquor, followed by a second wash with a weaker solution, and, thirdly, with pure water. The volume of washings in each case was about $1\frac{1}{2}$ to $1\frac{1}{4}$ times the volume of the cake. In treating a difficult wolfram tin slime, from which the coarser particles had been separated, only the fines being taken to the press, a head of only 4 ft. of water was used for

washing. One and a-half cake volumes of washings left 1% of soluble matter, 24 volumes left 0.16%, while 3 volumes left 0.08%, or occasionally

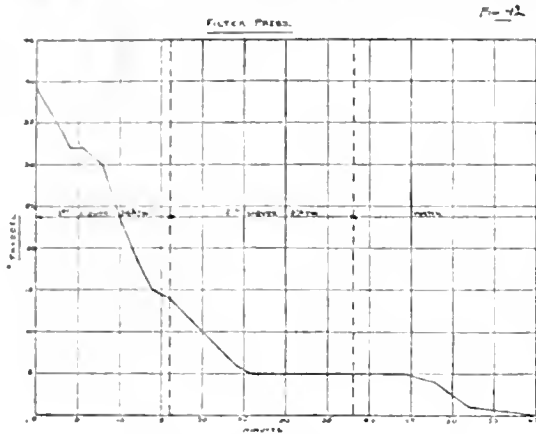


Fig. 44.

Washing carbonate of lime filter cake (contaminated with colloidal silica).

only a trace. With an especially difficult sample consisting of practically pure MnO and FeO , somewhat over twice this amount of wash water was required, the residual soluble matter being 0.3%.

Temperature control; steaming; hot and cold presses; air drying etc.

Viscosity is an important factor, as the rate of flow varies in inverse ratio. Since small increases in temperature may reduce viscosity in much greater proportion, the wise user will not overlook this means of increasing output.

various types of plates which may be used. A very satisfactory form is to cast some simple passages in the plate to take steam at a few pounds pressure. The steam enters and the drain leaves by continuous passages similar to those used for washing. This is quite sufficient to keep the plates hot and counterbalance the loss of heat from the press. Alternatively a coil of pipe may be cast in the plates, but this is an expensive method and does not give any more satisfaction. Where the utmost application of heat is necessary the plates may be hollow throughout. This construction is also very useful where liquors have to be kept cool while they pass through the press, as the greater area of directly cooled surface counteracts the somewhat low temperature head available for heat transference in such cases. It is not desirable, however, to look upon the filter press as a heating battery; the devices just described are only intended to prevent the temperature of the liquid dropping (or rising) unduly.

In order to get drier cakes air may be blown through the feed passage or from one side of the cake to the other, using the wash passages, in which case it is better to take in the air at the top of the press and bring it out at the bottom, so as to allow of thorough draining. In the case of a permeable substance the mixture can be substantially reduced by this treatment in a very short time. In one case spent lime cakes were brought from 38% moisture down to 20% or 22% in about 4 minutes with air at $82^{\circ}C$. Air blowing is equally useful in the case of finer materials, but much more time is required. A pressing of β -naphthol was reduced from 60% moist to between 30% and 40% by 1 hour's treatment. Two hours is a not unusual allowance for dyestuffs, but occasionally it is considered worth while to continue for far longer periods. Where a separate air-supply is not available, the feed pump may be allowed to run for a longer or shorter period,

INTERNALLY HEATED OR COOLED PLATES.

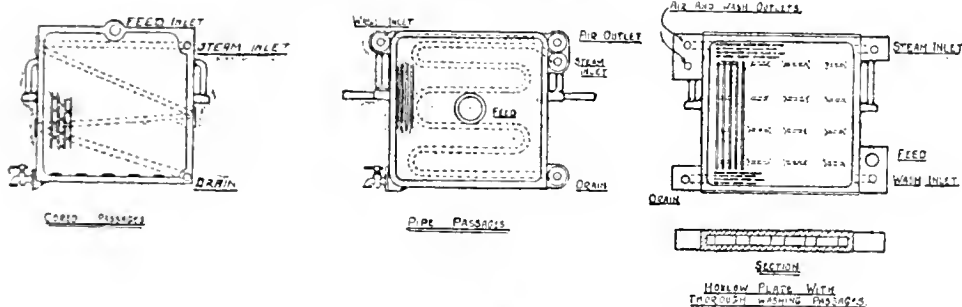


Fig. 45.

If the rate of flow is fairly large, the liquor itself will usually keep the press sufficiently hot. A steam valve is often fixed on the feed inlet, and this may be employed to pre-heat the press. It is also a very useful adjunct for steaming the cake and cloths through at intervals to keep them warm and render them more permeable, as in the case of wool grease recovery. In dealing with cakes of fuller's earth from the refining of lard, oils, etc., it is usual to steam these well through at the conclusion of filtration. This is best done one chamber at a time to concentrate the effect. After such treatment the cakes will usually drop out in the form of a loose meal, practically dry to the touch. They would then contain, say, 14% of lard, but reports are sometimes received of as little as 10% of grease remaining in the cake.

Where filtration is slow, the press may require special heating arrangements, and fig. 45 shows

sucking air and forcing it into the feed passage with satisfactory results in many cases.

Discharging the filter press.

Labour costs are dependent upon the press itself, the general arrangement of the plant, the grade of material, and local conditions.

While small plates mean that a greater number have to be handled, very big ones are difficult to move rapidly and, generally speaking, medium sizes give the best results. Wood plates may be somewhat larger on account of their lightness. If the material drops away cleanly from the cloth, and especially if the plates are of the simple recessed type with central feed, then larger sizes may still be economical in labour, as discharging is then a simple matter. The question of the best number of chambers for high output has been discussed previously. It suffices to say that no great saving

of labour is achieved in the case of long presses, as usually the time lost in attending to the closing gear, letting the press drain, etc., is a fairly insignificant portion of the discharging period. The 20 minutes per operation taken in the mathematical example was a very generous allowance. In most cases not more than 5, or at the most 10, minutes would be necessary, as will be judged from the examples to follow. Recessed plates require distinctly less handling than plates and frames, especially where no washing passages are required. Outside lugs do not make the difference one would expect, except where the cloths are frequently washed. There is also not much to choose among the various closing gears, so long as one is selected which will give efficient pressure. There is distinctly more to be saved by a proper arrangement of the press house, especially if it can be arranged for the operators to proceed directly from closing one press to opening another.

Reasonable room should be allowed all round each press, especially at the side on which the cake is to be brought away. It is helpful to have plenty of space below the press for dumping. Cake trays are distinctly labour economisers, but it is better where practicable to have the presses on an upper floor and discharge through shoots into trucks. Where this is impossible a useful plan is to have the tray or floor made sloping so that the discharged material can easily be raked down into a suitable receptacle. A good arrangement is to have the mixers, auto-claves or dryers which deal with the next operation so grouped with the presses that the discharged cake can be shovelled in direct. The pumps and sludge rams are preferably worked from the same platform as the filter presses, and the mixers which deliver the liquor to them should also be within easy access.

Most of the work depends, however, on the material itself, and the proper building of the cake. If the cake breaks up readily and drops cleanly away from the cloth, labour will be only perhaps one-half of what is necessary if the cake is half formed and sloppy, as then the whole cloth face must be thoroughly scraped. If such a partially built cake is of a clayey nature holding firmly together and binding to the cloth we have the worst conditions of all. Such clayey cakes, if properly built may nevertheless fall away quite readily. Materials containing hard sandy grains require careful attention to the joint surfaces, or these may not come well together. Slimy substances also require more attention in careful cleaning of the cloth faces. If the liquor is such as to make the cloths tender, or if corrosive materials are being handled which might injure the hands of the operators, then again extra care and time are required. Then again, hard wash waters may give a deposit on the plate surfaces when washing out alkali, and labour in cleaning this off may be minimised by a proper attention to the water supply.

Low labour costs also depend on the proper choice of a cloth and the adaptation to filtration of the chemical and physical qualities of the material.

In order to eliminate the varying factors of thickness and weight per cubic foot of the cake it is useful to compare the number of man-hours per operation per 1000 sq. ft. of filter surface.

A 38-in. plate and frame press, with 50 chambers, dealing with tin slimes, took two men $2\frac{1}{2}$ hours to discharge and get ready for restarting, giving a factor of 5.5. In the same establishment a wood plate and frame press, 40 in. \times 45 in. externally, having 50 chambers, treating tungstic oxide, was dealt with by the same gang in $1\frac{1}{2}$ hours, the factor being 4.2. The operation of the closing gear took under 10 minutes. In both cases the presses were of the thorough washing enclosed outlet type, and the cast-iron press did not have outside lugs. In

another works a similar press, but 1 metre square, having outside lugs with rubber joint rings, took about 3 hours to discharge.

In yet another works a 36 in. square press, making thirty 14-in. cakes, with spur wheel and pinion closing, and feed and single wash passage in rim, took 2 men one hour to empty, giving a factor of about 4.2 man-hours. The cakes were dyes of a fairly soft, slimy nature. A press next to it working on the same product was 32 in. square with 40 chambers. It had toggle closing gear and outside ears for feed and wash-ports, and employed practically the same proportion of labour. The times for manipulating the closing gear took 3 minutes and 5 minutes in all for each operation respectively. The whole installation consisted of 6 presses, 3 of each kind, and employed two emptiers, one man shovelling cake from under the press into the mixing vessels, and one operating valves. The work proceeded on the three-shift system, and an additional man was employed during one shift to look after the cloths and keep the plate grooves free. As there were about 30 operations in 24 hours, the factor for the total labour worked out at about 7.15 on the average. In all the above instances unfavourable conditions might increase the time taken by 25% to 50%, while in the case of some dyestuffs the factor may be as large as 11 or 12.

As against this a 38-in. recess plate filter press with 50 chambers would certainly be dealt with in from 30 to 40 minutes all told when working on sewage, giving a factor of 1.48. These figures assume that the sludge is not in the most favourable condition for discharging, nor the labour of the very best.

It is possible to obtain much better results, as, for instance, at the Dalmarnock Works, Glasgow. Through the courtesy of Thomas Melvin, Esq., it may be said that the installation consists of 19 presses, 12 of which have 40 chambers 40 in. square externally, the remainder having 34 chambers. Two labourers discharge from 36 to 37 pressings in an 8-hour shift, averaging one every 13 minutes throughout the day. This gives a factor of about 0.6. A timed operation on one of the shorter units showed that two men took well under 10 minutes, including replacing a cloth, giving a factor of 0.49. The total labour in this instance included in addition a working foreman who operated the valves, one man on the lime mixer and one looking after shoots and wagons. The total labour factor is thus 1.47, and in this instance supervision is included.

In another works, using the very largest type of press, the factor for discharging works out at about 0.91 and the total labour factor 1.36 without including foremen.

It will be seen therefore that the labour required is very dependent on the class of operator and material, much more so than on the size and details of the press itself. This is emphasized by the fact that among the records in the author's possession is one relating to a press with 25 in. square recessed plates, having a number of ports formed in the rim, in which the discharging factor amounted to less than 0.9 when dealing with an intermediate product.

Through the courtesy of Joseph Garfield, Esq., M.Inst.C.E., it is possible to give the following description of the sewer grease recovery plant at Esholt, Bradford. The installation contains 128 presses, making 47 cakes 36 in. square and 12 in. to 2 in. thick. It works continuously day and night, and is operated by compressed air, having an effective pressure of about 80 lb. per sq. in. The sewage is treated with sulphuric acid, causing the solid matter to separate and deposit in settling tanks as sludge. This sludge contains some 80—85% water, and of the remainder some 30—40% is grease. Each filter press is worked for some 71 hours before a complete cake is formed,

sludge being forced in for a couple of hours and then steamed well through for from $\frac{1}{2}$ to $1\frac{1}{2}$ hours according to the amount of cake that has been formed. During the final ten hours the cake is not steamed, as nothing extra is gained. When finished it contains 15–18% of grease and 28–30% of water. Each press holds about 33 cwt. on the average, and this works out at about 78 lb. per cu. ft. The rate of filtration is about 0.06 gall. per sq. ft. per hour. The presses are fitted with pneumatic closing gear and have steam connections to the feed passage at each end of the press. Cotton twill cloths are used, as jute will not stand the steaming, and the life of the cloth is about 6–10 weeks.

About 40–50 presses are discharged per day, and it takes two men an average of about 40 mins. to open, empty and re-close a press, about three cloths being replaced. This gives a factor of 1.79. The total labour is four steamers for each shift, four emptiers, day shift only, and one man on trucks, day shift only. The total factor is, therefore, about 4. This excludes one night and one day supervisor.

In conclusion it may be said that the best success of a filter press installation depends, not only on the choice of plant, but more especially on those in charge of the operations. It is nearly always possible to reduce high labour and cloth charges by proper methods in the lay-out of the plant, and especially in the formation of the precipitate or suspension, and those who take these facts duly to heart will get the most satisfactory results.

The author has to tender his best thanks to the many firms and individuals who have enabled him to collect various data and have given permission to use them for the present purpose. He has in most cases to respect the condition that these should remain anonymous, but he may especially mention Arthur C. Auden, Esq., M.Inst.C.E.; also H. Griffiths, Esq., A.R.C.S., B.Sc., who gave valuable assistance in criticising the proofs. He would also like to thank his wife, who prepared the various microscopic slides etc. for the camera, and carried out some useful researches in regard to filter cloths.

Edinburgh Section.

THE DEPOLARISATION BY OXYGEN OF A VOLTAIC CELL.

BY A. M. WILLIAMS, M.A., D.Sc.

The experiments here described were undertaken at the suggestion of Prof. Henry Briggs, Heriot Watt College, Edinburgh, with a view to obtain an indicator automatically recording the oxygen percentage in the air of mine rescue apparatus and oxygen chambers. The underlying idea was that the polarisation in a voltaic cell due to a film of hydrogen on the positive pole might be removed by a stream of oxygen, and it was sought to establish a relation between the amount of depolarisation and the percentage of oxygen in the current of gas passing over the positive plate.

After some preliminary experiments it was found that a cell in which the gas current passed over a silver gauze separated from a zinc plate by a pad soaked in calcium chloride solution gave a depolarisation relative to air of 0.20 volt with 98% oxygen. The curves obtained on running down through a voltmeter of 167 ohms resistance are shown in fig. 1. It will be noted that in the case of 98% and 65% oxygen there is a large initial drop, the E.M.F. passing through a minimum and then rising to a fairly constant final value.

On taking a second series of observations with a voltmeter of resistance 830 ohms, the final value of

the E.M.F. was much more rapidly attained in the case of the higher percentages of oxygen, there being no minimum such as was noted with the smaller external resistance. It was further found that the depolarisation was proportionately greater with the smaller percentages of oxygen. This is shown in fig. 2. With the 30% oxygen, alternation of air and oxygen caused appreciable movement of the voltmeter needle. There was no refill of the cell during the observations in fig. 2, and hence the cell was apparently capable of giving concordant values for, say, six two-hour runs with rests in

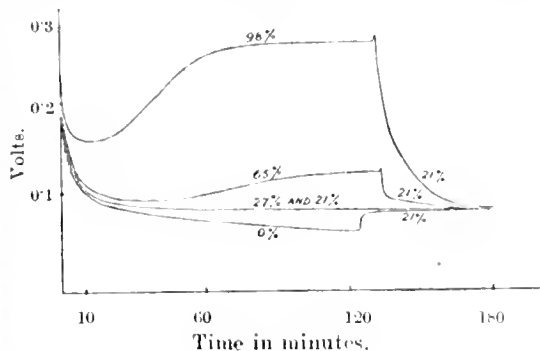


Fig. 1.

between. The rate of flow of gas in these experiments was always about 5–6 litres per minute.

It was noted that pressure on the gauze altered the E.M.F. as registered on the voltmeter—accidental pressure on the cell completely altered the final E.M.F. for several runs. So when a trial cell was specially constructed care was taken that the gauze was tightly stretched and secure from accidental pressure. The introduction of any obstacle in the orifice increased the pressure of the gas in the cell, and since this was equivalent to an increase in the concentration of the oxygen there was an increase in the E.M.F. It was therefore necessary

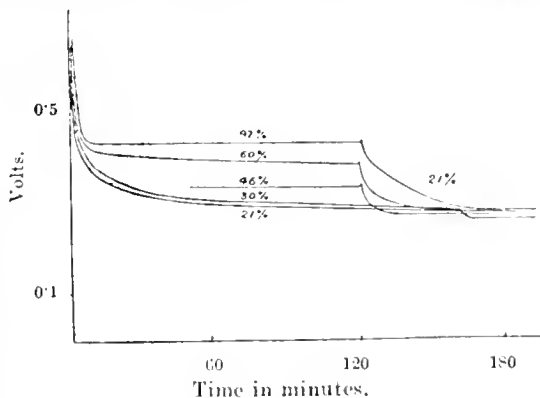


Fig. 2.

for the orifice to be free from accidental blocking of any kind (and incidentally necessary to correct for any large variation in the barometric pressure).

The effect of diluting the current of gas with carbon dioxide was tested. As a result of several series of observations it was concluded that carbon dioxide acted mainly like the nitrogen in the air—it served simply as a diluent of the oxygen and did not of itself seriously affect the E.M.F. In consequence subsequent observations were confined to oxygen-nitrogen mixtures, save on one occasion when the results with the carbon dioxide merely confirmed the earlier observations.

The above results appeared sufficiently satisfactory to justify the construction of a specimen working cell. It consisted of an ebonite frame across which a piece of silver gauze was stretched tightly. To reduce internal resistance the zinc plate was bent so as to leave a space of only 3 mm. for the pad. The cell fitted into a wooden box through which a current of gas could be blown. The E.M.F. of this cell was 0.830 volt at 20° C., and the internal resistance was 32 ohms. On refilling, the E.M.F. was found to be 0.824 volt and the internal resistance 28 ohms, *i.e.* the condition of the cell was appreciably reproducible.

Observations were taken as before in order to find the value of the E.M.F. with varying percentages of oxygen when the cell was running down through an external resistance of 830 ohms. The resulting curves were of the same type as those shown in fig. 2 and the final values are given in Table I., where $V - V_0$ is the depolarisation. The values under V have been corrected both for internal resistance in the cell and for error in the voltmeter.

TABLE I.

Order of expt.	% oxygen.	V .	$V - V_0$.
2	99	0.75	0.27
4	55	0.645	0.165
5	30	0.53	0.05
1	21	0.48	0.00
3	21	0.48	—
7	21	0.47	—
6	56-21	0.515	—

One series of observations was taken where the percentage of oxygen passing was varied at intervals until 21% was reached. There was a constant 3% of carbon dioxide in the gas in this case, which is represented by the dotted line in fig. 3. The lag is very obvious. In the earlier experiments when air replaced oxygen it will be seen from fig. 2, for example, that the time before the air value was reached varied from 60 mins. with 97% oxygen to 12 mins. with 30% oxygen, and in this case the lag is certainly 20-30 mins.

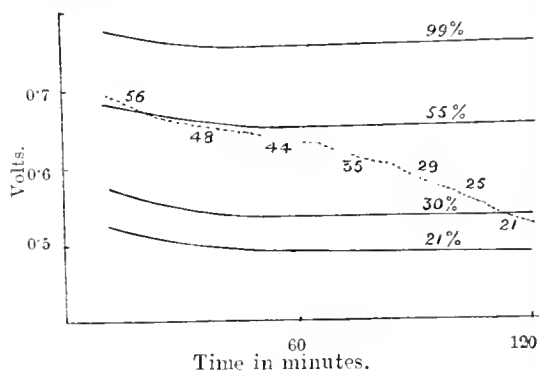


Fig. 3.

Some experiments were tried with zinc-silver cells containing pads soaked in solutions containing various proportions of zinc chloride and calcium chloride. It was found that the greater the concentration of the zinc chloride the more nearly reversible the cell. Thus with a solution containing 25% of zinc chloride and 25% of calcium chloride the initial E.M.F. was 0.563 volt, falling to 0.550 volt in one minute and after two hours to 0.532 volt. The effect of a current of oxygen on such a cell was to increase the E.M.F. by 0.037 volt—comparable with the effect with an earlier carbon-zinc cell.

Other experiments were directed to finding how the depolarisation varied with different metals,

using the solution employed with the typical silver gauze cell. The internal resistances varied somewhat as the cells were roughly constructed. The following table gives some of the results.

TABLE II.

Gauze.	Registered depolarisation.	Actual depolarisation.
Silver	0.22	0.275
Copper	0.11	0.14
Nickel	0.18	0.33
.. .. .	0.145	0.21

The nickel cell resistance was greatest owing to the difficulty in wetting the gauze. When the gauze was more efficiently wetted the depolarisation fell, pointing to the fact that thorough wetting tended to render less easy the access of the oxygen to the electrode.

The experiments so far described were all performed at room temperatures. It was now sought to find the influence of temperature on the standard type and a set of observations was taken at 40° C. The change in the initial E.M.F. with temperature was small, being from 0.83 volt at 15° C. to 0.82 volt at 40° C. when in air. To eliminate troubles due to drying of the pad it was found necessary to pass the air and oxygen currents over a surface of water heated to a temperature a little below that of the cell. The depolarisation registered was much the same as at 15° C., but there was this difference—the cell ran down much quicker at the higher temperature and refills were more frequent. Equilibrium when air replaced oxygen was attained in about 20 mins., which was much more rapid than with the corresponding observations at room temperatures.

A set of observations was also taken at 40° C. with two small cells in series. These were constructed according to specification and were about 2.5 cm. x 3.8 cm. each. The initial E.M.F. was over 1.70 volt and the registered E.M.F. fell from more than 1.00 volt to 0.46 volt in 1 min., and to 0.285 volt after 2 hrs. in a stream of air. With an oxygen current the E.M.F. fell to a steady value of 0.70 volt in 40 mins., and on replacing oxygen by air after a further 40 mins. the value fell from 0.695 to 0.350 in 1 min. and attained the equilibrium value of 0.285 volt in 20 mins. The registered depolarisation was thus 0.41 volt, while the actual depolarisation was 0.50 volt.

Summary and conclusions.

1. The depolarisation of a voltaic cell by a stream of oxygen passing over the positive plate may amount to 0.3 volt, which is much greater than can be accounted for by a simple gas-concentration effect.
2. The depolarisation is greater the less wet the gauze constituting the positive plate.
3. With a low resistance the polarisation takes place more quickly than the depolarisation, though this eventually overtakes and reduces the polarisation.
4. The less run down the cell the more sensitive it is to depolarisation by the oxygen.
5. The more nearly the cell approaches reversibility, the more closely does the depolarisation by the oxygen approach a gas-concentration effect.
6. Polarisation and depolarisation take place more rapidly at higher temperatures.

In conclusion the author desires to thank Professor James Walker, F.R.S., for his kindness in providing facilities for the prosecution of this research in the Chemical Laboratory, University of Edinburgh.

Communications.

THE ACCELERATION OF VULCANISATION. PART II.

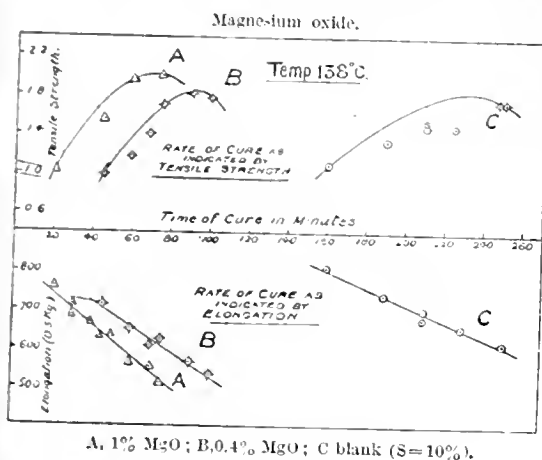
A Discontinuity in the Effect of Vulcanisation.

BY D. F. TWISS AND C. W. H. HOWSON.

In the previous paper on the above subject (J., 1920, 125 τ) the results were given of comparative experiments with aldehyde-ammonia, *m*- and *p*-phenylenediamine, thiocarbanilide, and hexamethylenetetramine.

The present paper contains the results of further tests. The method of procedure was the same as that adopted earlier, the most important feature being the use of a heated bath of oil for vulcanisation of the samples, whereby the periods of vulcanisation are rendered more definite than with steam heating.

Fig. 1 represents the progress of vulcanisation of the standard mixing (pale cr pe rubber 90, sulphur 10) with and without the addition of 1% and 0.4%.



A, 1% MgO; B, 0.4% MgO; C blank (S=10%).

Fig. 1.

of a high-grade, light calcined magnesia. Judging the rate of vulcanisation by the period required for the attainment of maximum tensile strength, the acceleration factor (*loc. cit.* p. 129 τ) for the magnesium oxide in question is approximately 3.3 and 2.5 respectively for 1% and 0.4%; the presence of the finely dispersed particles of undissolved magnesium oxide somewhat affects the extensibility of the rubber, and the acceleration factor assessed by the periods required for the reduction of the extensibility at 0.5 kg. per sq. mm. to 650% possesses a slightly exalted value.

In fig. 2 is represented the effectiveness of 0.5% of potassium hydroxide introduced as a solution in three times its weight of glycerol (see Twiss, J., 1917, 1185). The acceleration factor judged by the tensile strength method or by the period for the production of extensibility of 650%, has a value between 3 and 4, which compares very favourably with many popular organic accelerators at the same concentration and is approximately equal to the factor for the magnesia in the preceding experiment at twice the concentration.

The insistence of supporters of the use of thiocarbanilide that this substance needs the simultaneous presence of zinc oxide for the development of its catalytic effect, was suggestive of the possibility that the active substance might really be carbodiphenylimide, $C(NC_6H_5)_2$, produced from the thiocarbanilide, $CS(NH.C_6H_5)_2$, by the desulphurising effect of the zinc oxide, and which is known to be remarkably active chemically. An ex-

periment made with a sample of carbodiphenylimide prepared by the action of mercuric oxide on thiocarbanilide in benzene solution revealed no marked catalytic activity, but on account of the easy polymerisation of carbodiphenylimide this evidence could not be regarded as final, and it was considered necessary to produce the carbodiphenylimide *in situ* by the interaction of the thiocarbanilide and mercuric oxide in the rubber mixing. Parallel vulcanisation experiments were therefore made using (a) the standard mixing of rubber 90, sulphur 10; (b) the standard mixing with the addition of 1% of thiocarbanilide and 3% of yellow mercuric oxide; and (c) the same mixture as in (b) but without the thiocarbanilide. The results given in fig. 3 show that mixture (b) possesses no marked advantage in rate of vulcanisation relative to (c), and that any advantage in rate of vulcanisation imparted to a mixing by the introduction of thiocarbanilide cannot be ascribed to the formation of carbodiphenylimide. It is noteworthy, however, that

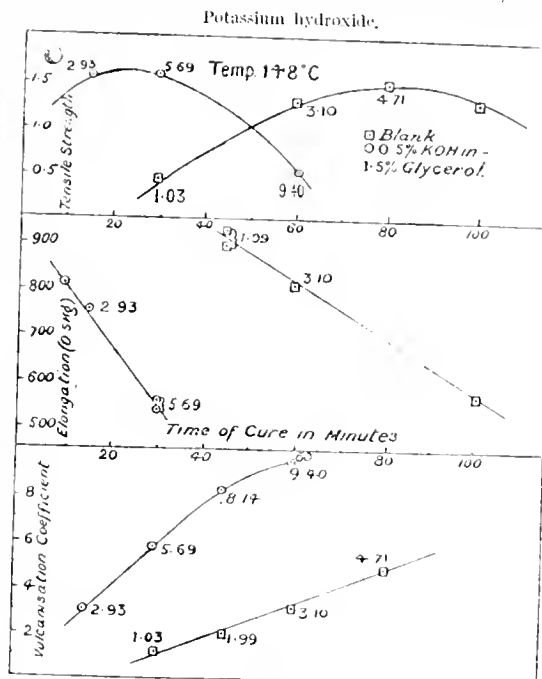


Fig. 2.

the curves for mixing (c) indicate marked catalytic power on the part of the mercuric oxide, which, indeed, might have been expected from the basic character of this substance.

Reference to fig. 12 in the previous paper (*loc. cit.*, p. 131 τ) reveals the remarkable fact that, although the mixing containing the organic catalyst together with zinc oxide, vulcanises much more rapidly than the blank mixing, the slope of the extensibility or elongation curve in the lower part of the diagrams, instead of being steeper, tends to be more gradual. Closer examination of the behaviour of mixtures of this composition reveals the fact that the extensibility curve in question undergoes a remarkable change of direction at an early stage of the vulcanisation process. A similar phenomenon is observable with a rubber-sulphur mixing containing as accelerator a mixture of triphenylguanidine and zinc oxide. The results are reproduced in figs. 4 and 5. In both cases the extensibility at first decreases very rapidly with increasing vulcanisation, but finally assumes a much more gradual rate of change.

The presence of zinc oxide appears to be favourable, if not conditional, to the occurrence of the

inflexion in the extensibility curve within the usual range of vulcanisation. From the results given in the previous paper it is evident that no indication of such inflexion exists in the extensibility curves

the bend appears to be absent the extensibility curve undergoes a change of direction at a stage of vulcanisation beyond the range of convenient testing.

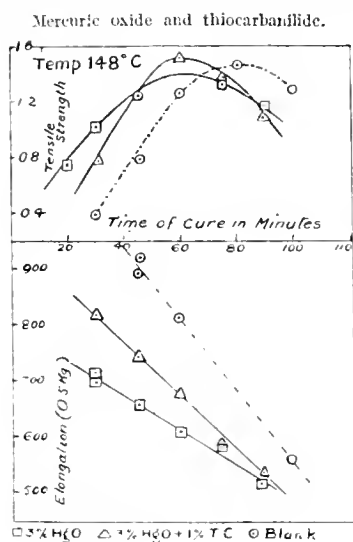


Fig. 3.

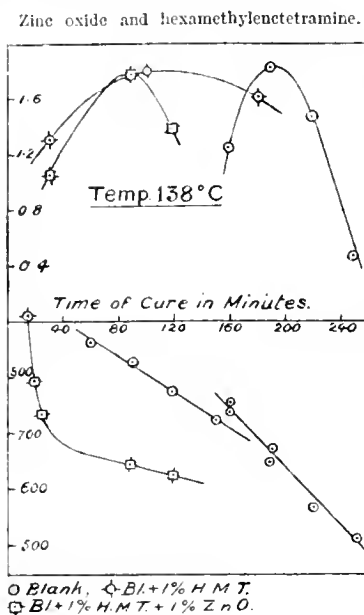


Fig. 4.

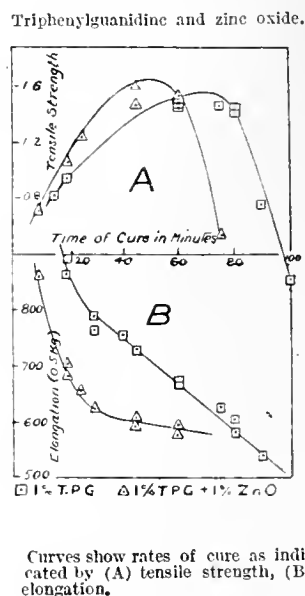


Fig. 5.

for the mixings containing aldehyde-ammonia; when the aldehyde-ammonia is used in conjunction with zinc oxide, however, an inflected extensibility

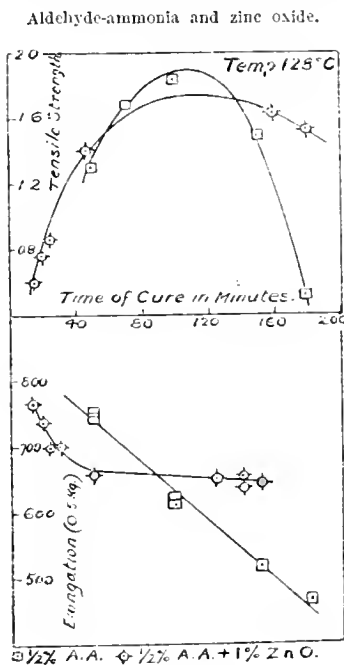
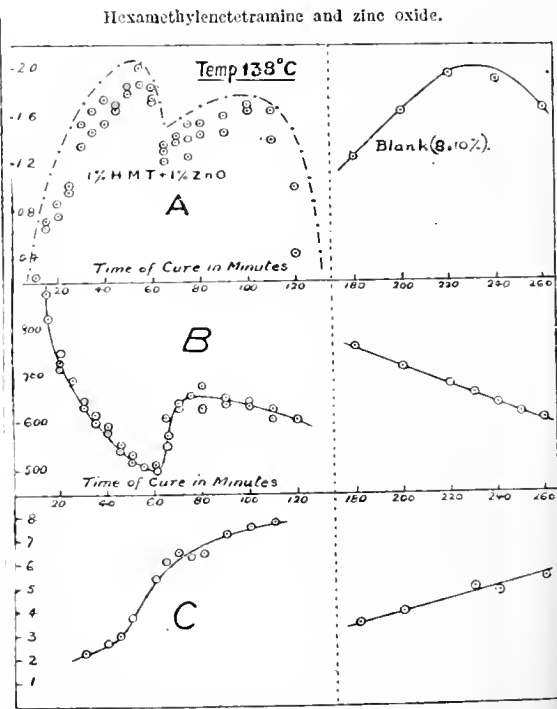


Fig. 6.

curve is obtained, as is demonstrated by the results in fig. 6. No such inflexion appears to occur in the progressive vulcanisation of the standard rubber-sulphur mixing with the addition of thiocarbamide and zinc oxide. It is possible, however, that when



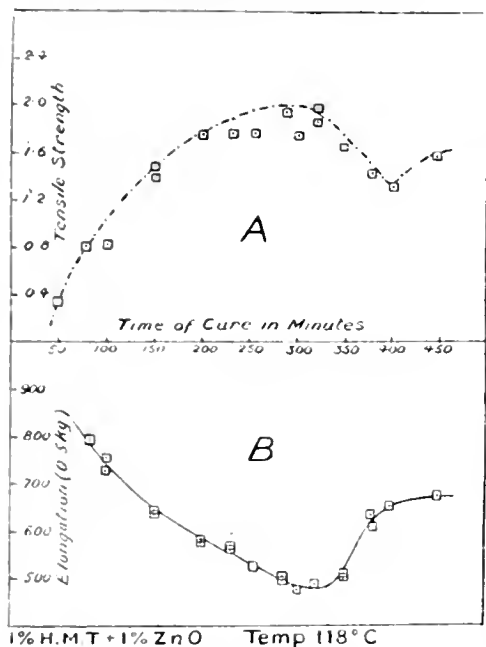
Curves show rates of cure as indicated by (A) tensile strength, (B) elongation, (C) vulcanisation coefficient.

Fig. 7.

By concentrating the number of tests around the point of inflexion of the extensibility curve for the hexamethylenetetramine-zinc oxide mixing it is found that, in at least this case, the curve in reality undergoes a double inflexion within a short in-

terval of time (figs. 7 and 8).^{*} This surprising result has been confirmed by repeated re-investigation with different mixings of the same composition vulcanised at the same and different temperatures. The results also indicate some concomitant discontinuity in the tensile strength and combined sulphur curves, but the actual outline of the tensile strength curves in figs. 7 and 8 is given with a certain amount of reserve on account of the somewhat greater variability in the values obtainable. It is noteworthy that the inflexion in the extensibility curve occurs at a vulcanisation coefficient of 4–5, indicating that the first peak in the tensile strength curve represents the ordinary "optimum cure," and that the subsequent peak is the abnormal one; it may also be observed that the maximum extensibility attained just after the inflexion of the curve, has approximately the value of 650 commonly observed at the tensile optimum of a rubber-sulphur mixing vulcanised under ordinary

Hexamethylenetetramine and zinc oxide.



Curves show rates of cure as indicated by (A) tensile strength, (B) elongation.

Fig. 8.

conditions. The explanation of this unexpected irregularity in the alteration in the physical characteristics of the rubber is not at all obvious; it appears probable that the two sections of the extensibility curve indicate two distinct processes causing alteration in the physical characteristics of the rubber, the point of inflexion representing the stage at which the one preponderating earlier is overtaken by the other, which then becomes predominant.

From the curve in fig. 7 it will be seen that for one mixing it may be possible within a small range of cure to apply three distinct periods of vulcanisation and yet to obtain identical extensibility in the resulting rubber in spite of the three different vulcanisation coefficients. Such a result, which is independent of the further complications introduced into the relation between the physical properties and the vulcanisation coefficient by subsequent "ageing" (see Reports of the Progress of Applied

Chemistry, 1919, 4, 338), serves to emphasize more strongly than ever the lack of simplicity in the relationship between the physical alteration effected in rubber during vulcanisation and the chemical change by which this alteration is induced.

Grateful acknowledgment must again be made of the permission of Mr. J. V. Worthington, Technical Superintendent and Director of the Dunlop Rubber Co., for the publication of the foregoing results.

NOTE ON A PAPUAN NATURAL PETROLEUM. PART II.

BY PROF. J. READ, M.A., PH.D., AND MISS A. C. P. ANDREWS, B.Sc.

In a previous communication from this laboratory some of the characteristics of a hitherto undescribed natural petroleum, from Papua, were recorded (J., 1919, 319 t). The results demonstrated that a "170° run" of the Papuan oil contained about 11% of aromatic hydrocarbons, from which a fraction having a combined content of benzene and toluene amounting to about 10% could readily be obtained. The Papuan oil thus contains appreciable amounts of aromatic hydrocarbons, although it is less rich in these than Borneo petroleum (*cf.* Jones and Wootton, Chem. Soc. Trans., 1907, 91, 1146). The observations now recorded deal largely with the higher boiling fractions, and were made upon material obtained from the second sample of Papuan petroleum (No. 5 bore, Government Oil-field, Upoia, Papua) mentioned in the foregoing paper.

Crude oil.

The crude oil had sp. gr. 0.7965 at 22° C., and $n_D^{20} = 1.4489$.

"180° Run."

When the crude oil was distilled up to 180° C. from an ordinary flask the fraction passing over ("180° run") amounted to 44.65%, by weight, of the original material. It had sp. gr. 0.755 at 22.25°, $n_D^{20} = 1.425$. When distilled (*loc. cit.*) through a column, the "180° run" yielded the fractions numbered 1 to 4 in Table A below.

Residuals boiling above 180°.

The reddish-brown fluorescent residue left in the distilling flask after the preceding distillation amounted to 55.35%, by weight, of the crude oil, and had sp. gr. 0.831 at 25° C.; $n_D^{20} = 1.4615$. The total amount utilised in the work described below was about 1800 grams.

Upon heating this residue in an ordinary distilling flask, distillation began at 90° C., and a small fraction had collected at 180°. The first fraction was cut at 220° and second and third fractions were collected up to 260° and 300° respectively (see fractions 5, 6, and 7, in Table A). In each fraction a cloudiness was observed, followed by the separation of a small heavy layer of liquid. This layer became semi-solid in the last fraction. In all three cases the fraction became homogeneous when shaken and allowed to stand. The first of these fractions, which was very similar in appearance and odour to good kerosene, was colourless, the second faintly yellow, and the third pale yellow. Slight fuming was noticeable as the temperature approached 300° C., but there was no rank odour indicative of "cracking." At 235° a white semi-solid deposit was formed on the sides of the condenser-tube. The temperature remained constant for some time at 251° and at 269° C. The specific gravities and refractive indices (see Table A) show a gradual increase with rising boiling point.

By interrupting the distillation at 210° and allowing the residue to cool, a dark-brown gelatinous mass was obtained. The residue left after distilling to 300° set to a semi-solid cake when cold; the

^{*} The effect, which is observable over only a small range of concentration, is probably to be attributed to the physical nature of the zinc oxide as an exceedingly fine powder rather than to its chemical nature; 1% of carbon black (gas black or lampblack) introduces a similar irregularity.

amount of this residue corresponded to 16.6%, by weight, of the original petroleum.

Residuals boiling above 300°.

The bulk of the residuals boiling above 300° was distilled under reduced pressure, but in one instance the distillation was carried to 350° under atmospheric pressure. Towards the end of this process the distillate solidified in the condenser-tube. Fractions collected between the limits 300°—340° and 340°—350° amounted to 6.2% and 2.2%, respectively, calculated on the weight of the original crude oil, and the residue represented 8.2% of the crude oil.

The main residues boiling above 300° were distilled from a Claisen flask under a pressure of 18 mm. in portions of about 60 g., the temperature of distillation being carried to 255° by means of a metal bath. The four portions noted in Table A (Nos. 8, 9, 10, and 11) were thus collected. From about 225° the distillate partially solidified to a brownish-yellow waxy mass as soon as it reached the receiver, and at a higher temperature partial solidification occurred in the condenser-tube. Each fraction had a darker yellow colour than the one preceding. The last two fractions (Nos. 10 and 11) exhibited a crystalline formation interspersed throughout a viscous liquid. The first of these fractions, when warmed, became completely liquid at about 20°, but in the other case (No. 11) the material remained partly solid at 35°, and the specific gravity was therefore observed at 40°. The dark-brown solid mass remaining in the Claisen flask corresponded to 5.9% of the original oil.

Residuals boiling above 255° at 18 mm.

The solid dark-brown residue just referred to possessed a greasy feel and was almost odourless. It was not appreciably decolorised by treatment with animal charcoal. Attempts to obtain pure crystalline material from it by slow deposition from solvents and by volatilisation were unsuccessful. A portion of the residue was again distilled under reduced pressure, and in this way further fractions containing crystalline material were obtained (Nos. 12, 13, and 14). Fraction 12, when liquefied, exhibited a pronounced brown fluorescence, whilst with fraction 13 the fluorescence was green. The final residue, which failed to distil below 310° at 20 mm., was quite hard; it was black, somewhat sticky, and practically odourless. The amount was 0.9% of the crude oil.

Summary of fractions obtained.

The appended table indicates the character and yield of the various fractions into which the crude oil was resolved:—

TABLE A.
Fractional distillation of Papuan petroleum.

Fraction no.	B.p., °C.	Pressure.	Weight percent- age of crude oil.	Sp. gr.	Refractive index, n_D^{20}
1	60°–90°	Atm.	5.0	0.715/24°	1.4088
2	90°–120°	"	16.6	0.744/24.5°	1.4161
3	120°–150°	"	14.0	0.7615/25°	1.4278
4	150°–180°†	"	8.0	0.7905/24°	1.4405
5	up to 220°	"	13.6	0.796/20°	1.4440
6	220°–260°	"	13.9	0.820/20°	1.4568
7	260°–300°	"	10.2	0.842/20°	1.4684
8	up to 190°	18 mm.	3.0	0.56/25°	1.4757
9	190°–200°	"	1.4	0.855/25°	1.4760
10	200°–225°	"	3.3	0.861/25°	1.4791*
11	225°–255°	"	3.1	0.860/40°	1.4931*
12	up to 275°	20 mm.	3.0	—	1.5189
13	275°–305°	"	1.6	—	—
14	305°–310°	"	0.3	—	—
15	above 310° (residue)	"	0.9	—	—

† Not redistilled.

* After removing crystals.

None of these fractions up to No. 8 exhibited any measurable optical rotation when examined in a 2 dm. tube at 20° C. in sodium light. Fraction 9, however, under these conditions gave a dextro-rotation of 0.05°; whilst fraction 10 gave $\alpha_D = +0.10^\circ$ in a 2 dm. tube. Fraction 11 was too dark to read accurately, even in a 1 dm. tube, but it appeared to possess a small dextro-rotation. The above values are of the same numerical order as those recorded by Kakuzin for Russian and American petroleum, both of these groups being also as a rule dextro-rotatory (J. Russ. Phys. Chem. Soc., 1904, 36, 554). Jones and Wootton (*loc. cit.*) also observed similar values for Borneo petroleum, but in this case the rotation was to the left.

Isolation of normal tricosane.

The crystalline material present in fraction 10 (b.p. 200°–225° at 18 mm.) was separated from the accompanying viscous liquid by filtration with suction and dried by pressure on a porous plate. The almost colourless crystals so obtained were readily soluble in light petroleum or in ether, but were only sparingly soluble in cold alcohol. When purified by crystallisation from hot alcohol the product consisted of colourless leaflets melting at 48° (corr.). The melting point was unaltered by recrystallisation. The original thick oily filtrate slowly deposited further quantities of similar crystalline material upon standing in the cold.

The substance, in benzene solution, showed no pronounced reaction with bromine and upon combustion it yielded C=85.0% and H=14.9%. It was therefore identified as normal tricosane, $C_{23}H_{48}$ (m.p. 48°; b.p. 234° at 15 mm.; C=85.1%, H=14.9%), a hydrocarbon which hitherto has been identified in brown coal paraffin and in Pennsylvania petroleum.

Fraction 11 (b.p. 225°–255° at 18 mm.) yielded a further quantity of the same hydrocarbon, but the total weight obtained from the whole of the distillates amounted only to a few grams.

Upon concentrating the alcoholic mother liquors from the purified tricosane a crystalline deposit melting at 40°–42° was formed. It seems probable that this contained heneicosane, $C_{21}H_{44}$ (m.p. 40.4°; b.p. 215° at 15 mm.).

Isolation of normal pentacosane and normal heptacosane.

By treating fraction 12 (b.p. 255°–275° at 20 mm.) in the manner just outlined, and recrystallising the solid product three times from hot alcohol, colourless leaflets were obtained of m.p. 54.3°–54.8° (corr.). The substance was similar to tricosane in appearance and properties, and upon combustion it gave C=85.0% and H=14.8%. It was accordingly diagnosed as normal pentacosane, $C_{25}H_{52}$ (C=85.1%, H=14.9%). This hydrocarbon does not appear to be well known; Mabery, who isolated it from Pennsylvania petroleum, recorded the melting-point as 53°–54° (Amer. Chem. J., 1902, 28, 193); and Marie, who prepared it from cerotic acid, gave the melting point as 54° (Ann. Chim. et Phys., 1896 [7], 7, 214).

From fraction 13 (b.p. 275°–305° at 20 mm.) was isolated in a precisely analogous manner a colourless crystalline substance possessing similar properties and melting at 60.5°–60.8° (corr.). Upon combustion this yielded C=85.0%, and H=14.8%; it was thus recognised as normal heptacosane, $C_{27}H_{56}$ (m.p. 59.5°, or 60°; b.p. 270° at 15 mm.; C=85.2%, H=14.8%). This hydrocarbon has been recorded as a constituent of beeswax, tobacco, and soot (Meyer and Jacobsen, Lehrbuch d. org. Chem., 1907, I, i, 164).

The weight of each of these hydrocarbons isolated amounted only to a few grams. The final residues probably contain still higher members of the paraffin series. The highest paraffin hydrocarbon reported by Mabery (*loc. cit.*) in Pennsylvania petroleum is $C_{28}H_{58}$; but the next three members

have been found in various eucalyptus and other natural essential oils (*cf.* Smith, J. Proc. Roy. Soc. N.S. Wales, 1913, 47, 95).

None of the three hydrocarbons described above exhibited optical activity when dissolved to a 7% solution in light petroleum and examined in a 2 dm. tube in sodium light.

The weak optical activity of Papuan petroleum suggests an organic origin for this material. It is thus particularly interesting that each of the three (or four) high paraffin hydrocarbons yet isolated from it possesses an odd number of carbon atoms in the molecule, so that they stand in close chemical relationship to such typical products of organic activity as bebenic, cerotic, melissic, and other high fatty acids with an even number of carbon atoms in the molecule. In the case of Pennsylvania petroleum, however, Mabery (*loc. cit.*) isolated an almost unbroken series of paraffin hydrocarbons from $C_{11}H_{22}$ to $C_{25}H_{50}$; a more detailed examination of the paraffins occurring in Papuan petroleum would therefore be of considerable interest when larger amounts of the material become available for distillation.

Main characteristics of Papuan petroleum.

Among the leading features of this petroleum are absence of pronounced colour or odour in the original oil and in the various fractions; absence of sulphur and nitrogen; and low original specific gravity. Connected with the last feature are the unusually high yield of low-boiling distillates and the small proportion of high-boiling residuals afforded by the oil, the "benzine" fraction (to 150°) amounting to 36%, and the "kerosene" fraction (150°–300°) to 46%, by weight, of the crude oil.

Corresponding data for other natural petroleum are listed in the following table (Meyer and Jacobson, *loc. cit.*, 187; Jones and Wootton, *loc. cit.*):—

TABLE B.

Fractional distillation of various natural petroleum.

Locality.	Sp. gr. of crude oil.	"Benzine," to 150° (°).	"Kerosene," 150–300° (°).	Residues, above 300° (°).
Pennsylvania	0.79–0.82	10–20	55–75	10–20
Caucasus	0.85–0.90	5	25–30	60–65
Gallia	0.82–0.90	5–30	35–40	30–55
Ohio	0.80–0.85	10–20	30–40	35–50
Borneo	0.83–0.87	19	51	27
Papua	0.796	36	46	17

In the case of Borneo petroleum it is stated that no distillate passes over below 100°, whereas the Papuan product yields 5% of distillate up to 90°, but there is a decided similarity between these two petroleum. The total amount of aromatic hydrocarbons present has not been determined, but the crude oil contains about 4% of such hydrocarbons distilling below 170° C. (*i.e.*, benzene, toluene, xylenes, etc.). Chemically, Papuan petroleum consists largely of paraffin hydrocarbons, not only with aromatic hydrocarbons, but also with other cyclic hydrocarbons, as is indicated by the high physical constants exhibited by the residues from nitration processes (*cf.* table C.).

Similar considerations hold for the fractions of higher boiling point. For example, the fraction distilling at 220°–260° has sp. gr. 0.820, whilst the value for the corresponding paraffin hydrocarbon is about 0.775. Again, tricosane, with sp. gr. 0.778, occurs in a fraction having sp. gr.

over 0.86. In these cases, as in the others quoted, there is no evidence of the presence of open-chain hydrocarbons other than paraffins, and the high

TABLE C.

Physical constants of original fractions and of residual hydrocarbons after nitration.

Fraction.	Specific gravity.		Refractive index, n_D^{20}	
	Original fraction.	Residual hydrocarbons	Original fraction.	Residual hydrocarbons
60°–90°	0.715 / 24°	0.710 19.5°	1.4088	1.3994
90°–120°	0.744 / 21.5°	0.747 17°	1.4161	1.4090
120°–150°	0.7615 / 25°	0.768 19.5°	1.4278	1.4160

specific gravity and refractive index must therefore be ascribed to the presence of aromatic hydrocarbons, possibly in association with other types of cyclic hydrocarbons. Although much work remains to be done upon these constituents of the oil, the main character and potentialities of this interesting natural product may be gauged from the data now rendered available.

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THE CONSISTENCY OF GREASES.

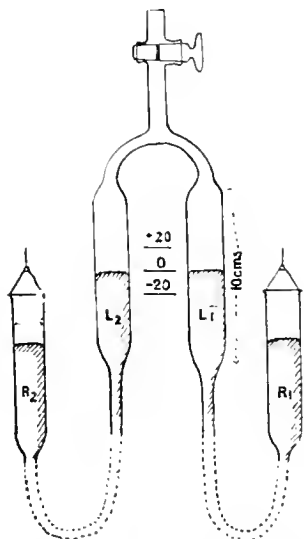
BY F. S. CLULOW, B.SC., A.R.C.S., A.I.C., AND C. W. TAYLOR, A.R.C.S., A.I.C.

In the course of experiments on the viscosity of lubricating greases it was found that the results obtained with present-day apparatus were of little practical value for comparing the behaviour of greases under actual working conditions. For example, a stiff grease used for lubricating a piece of mechanism such as a gear box has its consistency considerably reduced after working, although the temperature remains practically constant. This led to the realisation that the viscosity or consistency of a grease at a given temperature was not a definite physical property, as in the case of an oil, but seemed to depend on certain other conditions. Results obtained from experiments with a Küssing penetrometer appeared to support this view. The grease under examination was melted and poured into a large crystallising dish, where it was allowed to cool. When cooled and tested with the penetrometer it was found that the consistency at the centre was greater than that near the edges (except very near the edge, where the close proximity of the side of the vessel affected the result). The only explanation of this seemed to be that the rate of cooling affected the result, as the centre portion would have cooled more slowly than the edges.

Experiments were carried out with the Küssing viscometer, but the results obtained were unsatisfactory. In order to obtain anything like concordant results the same tube has to be used, as the friction of the piston varies with the different tubes, and this entails waiting a day between each experiment. The chief disadvantage of the apparatus, however, is that it affords no means of examining the viscosity under different conditions, except that of temperature, and subsequent work showed that it indicated a higher viscosity than the grease possessed under actual working conditions.

In order thoroughly to investigate the problem an attempt was made to measure any change of viscosity due to mechanical treatment or the rate at which the grease had been previously allowed to cool after melting. The apparatus used for the purpose consisted of two elongated bulbs about 3 cm. diam. and 10 cm. long, connected by a

tube 3 cm. long and 3 mm. internal diameter, enclosed in a constant temperature bath. The time required for the lower surface of the grease to rise between two marks in the lower bulb, at a given temperature and under a known head of mercury from a reservoir attached to the lower bulb, was observed. The apparatus gave a series of useful results, but had one or two defects, the chief being a tendency for the mercury to push through the centre of the grease occasionally, thus forcing a thin core of grease above the surface at the top. This, of course, diminished the volume of grease forced through the capillary. In order to overcome this difficulty a new apparatus was devised in which both surfaces of the grease were in contact with mercury. Also, arrangement was made to give a constant head of mercury throughout the experiment.



The apparatus consists of an inverted U-tube, 4 mm. int. diam., having a cylindrical bulb on each limb 10 cm. long and 14 mm. diam., the narrow tube connecting the two being fitted with a branch tube and tap for filling purposes. The free ends of the limbs are connected by means of pressure tubing to movable mercury reservoirs, 14 mm. diameter, which reservoirs are connected by a gut string passing over small pulleys, so that when one reservoir moves up the other moves down an equal amount. This amount of movement is measured by means of a millimetre scale behind them. By altering the length of the gut, the head of mercury can be varied at will. A jacketing apparatus is provided to keep the U-tube and bulbs at constant temperature.

The method of procedure is as follows:—The temperature of the water jacket is raised to a few degrees above the melting point of the grease. By raising both reservoirs, with the tap open, the tube is completely filled with mercury. The melted grease is poured in through the tap, the reservoirs being slowly lowered until the mercury in both bulbs is at the zero mark. The tap is turned off and the grease allowed to cool to the temperature at which the experiment is to be conducted. The right-hand reservoir is then brought to the zero mark and the gut adjusted so that the mercury surface in the left-hand reservoir is 140 mm. below this level. This is the head of mercury used throughout the succeeding experiments with this apparatus. As the mercury level in the right limb moves up, forcing the grease through the tube, the right reservoir is slowly raised, keeping the mercury surfaces level. By means of the gut string an equal downward movement is communicated to the left reservoir,

thus maintaining a constant pressure. The time noted is that required for the mercury surface to move 20 mm. above and below the zero in either limb.

When a grease is freshly solidified, the pressure of 140 mm. is occasionally insufficient to cause movement, in which case it may be set in motion by constricting the pressure tubing of the right limb, after which the normal pressure is sufficient.

Standardisation of apparatus.

As one apparatus will differ slightly from another in dimensions, it is necessary to standardise each apparatus. Castor oil was found to be suitable for this purpose if tested at about 0° C. The pure oil employed had sp. gr. 0.96345 at 21° C., and absolute viscosity 8.4586 at 21° C. The head of mercury, as stated before, was 140 mm., and the experiment was conducted at temperatures between -2° C. and 4° C. A direct determination at 0° C. was found to agree with that obtained from the time-temperature curve.

Appended are the results:—

Temp. ° C.	Time of flow, secs.	Temp. ° C.	Time of flow, secs.
-1.2	16.2 16.0	0	13.5
-1	16.1	+1.5	12.0
-0.5	15.5	+0.5	13.0
	14.0 14.2		
-0.2	14.4	+1.7	11.7
	13.8 13.7	+4.5	9.1

It is proposed that each apparatus should be standardised by using such a head of mercury as will produce a time value of 13.5 seconds for castor oil at 0° C. By this means the results obtained with different pieces of this apparatus will be rendered comparable.

The first grease to be examined was a sample of American anti-foaming grease, and it was tested in the first form of apparatus. Having filled the tube, the temperature was adjusted at 32° C., and after allowing half an hour for the grease to reach this temperature, the experiment was commenced. It was found that each successive time the grease was forced through the tube the rate of flow increased, although the temperature was kept constant throughout. After working the grease up and down many times, a constant time was at last obtained which could not be decreased by further working. Another sample of the same grease was similarly tested and was found to give the same minimum value.

The following are the time values obtained at 32° C.

Time of flow in seconds:—720, 345, 304, 259, 185, 155, 140, 135, 125, 127, 115, 113, 98, 92, 85, 84, 83, 81, 76, 60, 56, 48, 37, 35, 33, 35, 32, 31, 26, 28, 28, 24, 23, 21, 20, 20, 19, 18, 19, 17, 16, 17, 15, 15, 15, 15, 15, 15.

It was found that the viscosity only increased very slightly when the worked grease was allowed to remain for 90 hours at about the same temperature and then again tested at 32° C., rapidly dropping to the same minimum value on working, as the following figures show:—

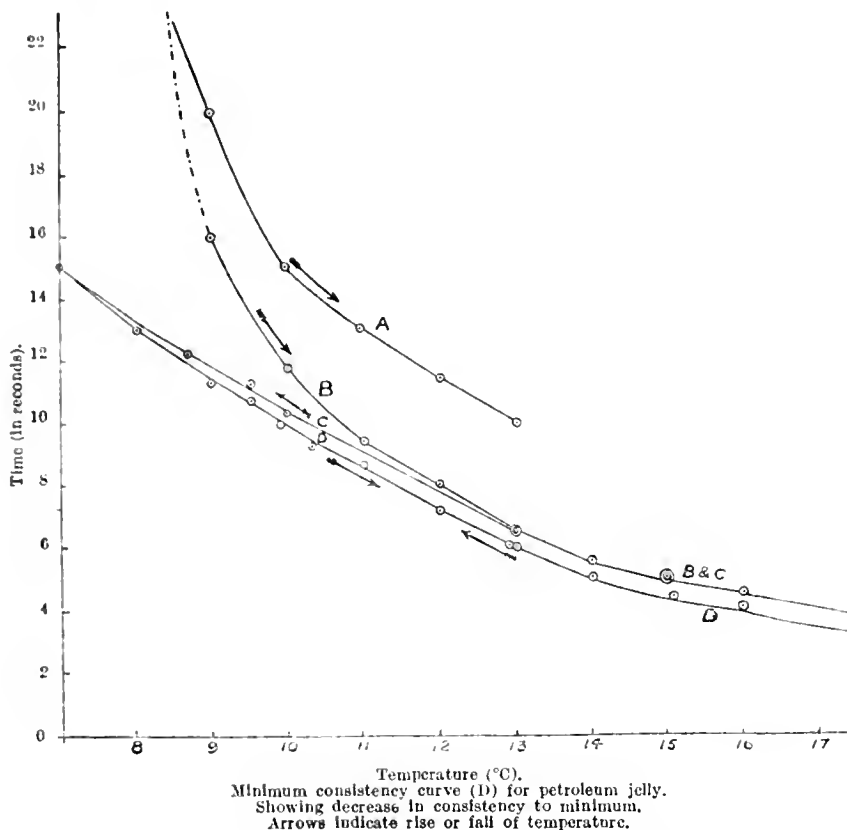
Time of flow in seconds:—45, 20, 17, 16, 16, 16, 15, 15, 15, 15, 15.

The worked sample of grease was then re-melted in the tube by heating the water jacket. It was allowed to cool to the original temperature of 32° C. when it was found to have regained its high initial viscosity, which gradually dropped to the same minimum value after working.

The method of experiment was then varied. The temperature was gradually increased or decreased a degree or two at a time, and the time of flow at

each temperature taken, allowance being made for the grease to attain that temperature. After repeating this several times, it was found that a constant value was again obtained for each tem-

the grease only regained its original high viscosity on re-melting and cooling, led to the theory that the grease was originally crystalline, the crystalline structure being broken up on working.



perature, which represented the minimum viscosity at that temperature. On plotting the temperature against corresponding time values, with the temperature rising and falling alternately to its highest and lowest limits, a series of oscillating curves were obtained, which gradually became closer and finally coincident. This final curve represented the minimum viscosity of the grease. The following results obtained for "vaseline" (sp. gr. 0.8762 at 21° C.) illustrate this:—

Rising temperature.

Temp., ° C.	Time of flow, seconds.		
	Curve A.	Curve B.	Curve D.
7	—	—	15
8	—	—	13
9	20	16	11.3
9.9	—	11.8	—
10	15	10	—
11	13	9.5	8.6
12	11.5	8	7.2
12.9	—	6	—
13	10	6.5	—
14	—	5.5	5
15	—	5	—
16	—	4.5	4
18	—	3.5	—
18.5	—	—	3.3

Falling temperature.

Temp., ° C.	Time of flow, in seconds.	
	Curve C.	Curve D.
18	3.5	3.1
15.1	—	4.3
15	5	—
13	6.5	6
10.3	9.3	—
10	10.3	—
9.5	11	10.8
8.7	12.2	—
7	15	—

To examine this theory, photo-micrographs of the anti-foaming grease were taken between crossed Nicols under differing conditions. The results obtained proved this assumption to be correct.

A film of greaso was also melted between two cover slips which were placed on mercury to get almost instantaneous cooling. Only a slight trace of crystalline structure was observed.

After further working of the same sample the crystalline structure had entirely disappeared. This represents the condition of the grease at its minimum viscosity, and explains why the value is always the same for the same grease at the same temperature.

Slides prepared for "vaseline" exhibited a similar crystalline structure.

In order to demonstrate that the slowly cooled grease actually has a higher initial viscosity than

The foregoing results indicate the lowering of viscosity by mere mechanical treatment, which points to an alteration in the structure of the grease. This, in combination with the fact that

a more rapidly cooled one, and also has its viscosity reduced more slowly on working, the following experiments were carried out.

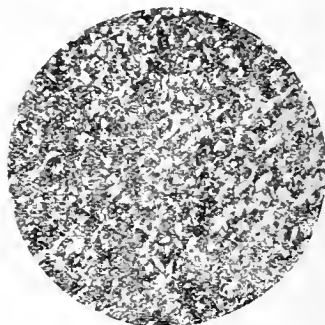
(1.) The viscometer was filled with melted "vaseline," the water jacket allowed to cool as

slowly as possible, and a series of viscosity tests performed at 14° C. The results are shown in Curve 1.

(2.) The viscometer was filled with melted "vaseline," and the water jacket cooled as rapidly



1. Grease melted on the slide and allowed to cool exceedingly slowly (about 1 day). $\times 126$.



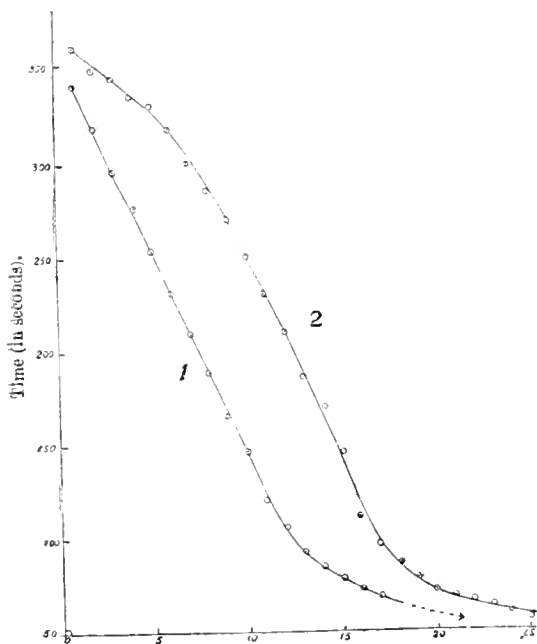
3. Grease cooled quickly by placing slide on a stone slab. $\times 126$.



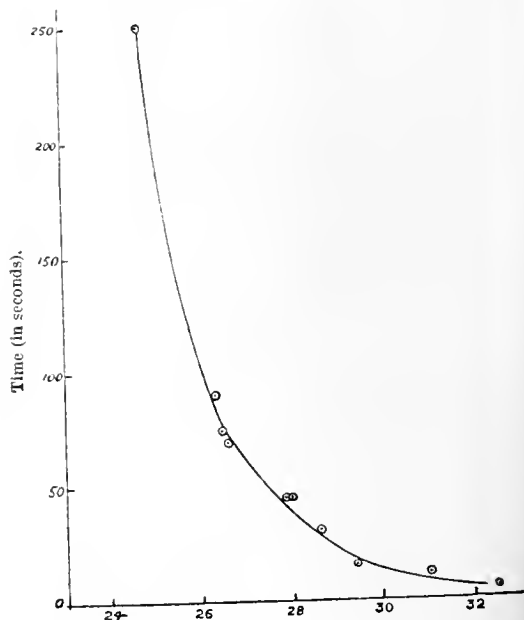
2. Melted grease allowed to cool in 1 hour. $\times 126$.



4. Slowly cooled grease after being mechanically worked for some time. $\times 126$.



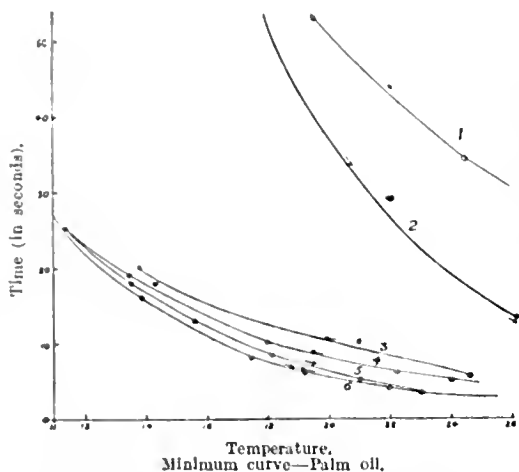
Curves showing reduction in consistency of (1) slowly and (2) quickly cooled vaseline. Temp. 14° C.



Wool fat. Sp. gr. $21^{\circ}/4^{\circ}$ 0.9397.

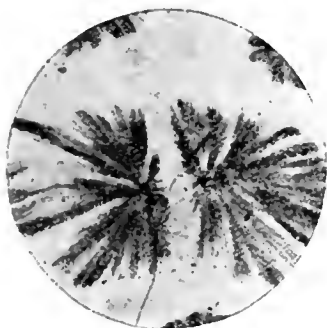
as possible, and a series of viscosity tests performed at 14° C. The results are shown in Curve 2.

The curves show that the initial viscosity of 1 was higher than that of 2, and in the case of 2 the viscosity was more rapidly diminished on working, as was expected. If it had been practicable to obtain a greater difference in the rates of cooling of the two samples, the curves would no doubt have shown still greater divergence.



Slides were prepared of various greases tested in the viscometer. Their behaviour in each case was such as would be expected from their appearance under the microscope.

A sample of wool fat photographed between crossed Nicols showed no crystalline structure, being completely isotropic. It was therefore expected that the viscosity would not alter on working. This was found to be the case. No matter for how long the grease was worked, the viscosity for each temperature was always the same.

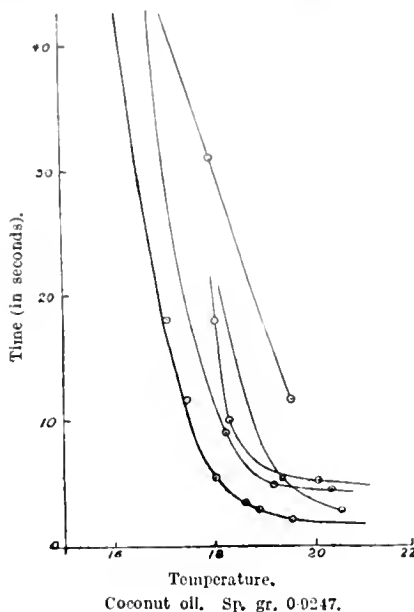
Coconut oil $\times 25$.

The crystals of palm oil are of a feathery type, which appear as if they would be easily broken up on working. It was, therefore, expected that the viscosity would drop rapidly at first and then rapidly reach the minimum. This was actually the case as shown by the curve.

Coconut oil, as seen from the photograph, crystallises very distinctly, the temperature of crystallisation being about 18.2° C. The grease was very difficult to work below this temperature in the viscometer.

It was found easier to start at the highest temperature and work down to the lowest, owing to

sudden crystallisation at the lower temperatures, so the curves are all drawn from tests with a falling temperature.

Palm oil $\times 25$.

The foregoing results show that for the measurement of viscosity to be of practical value it is necessary to state under what conditions the viscosity was determined, and these should resemble as nearly as possible the conditions under which the grease is to be utilised. It is obviously useless to quote the viscosity at a certain figure when, after a few minutes' working in a piece of machinery, it diminishes to a fraction of that amount. In this case the minimum viscosity, as determined by the apparatus described, is the only one which is of any practical value, as it is independent of all factors which tend to produce varying results with other empirical forms of apparatus.

The results obtained with the improved form of viscometer may be expressed as time values or as the ratio of the time value for the grease under examination, to that for castor oil at 0° C. If this latter method be adopted, a measure of the absolute viscosity of the grease may be obtained by knowing the absolute viscosity of castor oil at 0° C., since the ratio of the time for castor oil at 0° C. to the time for the grease at t° C. is equal to that of the absolute viscosity of castor oil at 0° C. to the absolute viscosity of the grease at t° C.

THE DISTILLATION OF CAMPHOR IN INDIA.

BY JOHN LIONEL SIMONSEN AND TARAK PRASAD GHOSE.

The object of this paper is to record the results of experiments which have been in progress at Dehra Dun for some years past on the camphor content of the various portions of the *Cinnamomum camphora* grown at Dehra Dun and elsewhere within the Indian Empire.

As a general rule camphor wood is the main source from which the camphor and camphor oil are distilled, but in view of the length of time required for the growth of the trees, these experiments have been mainly concerned with determining whether the leaves from coppiced trees could not be utilised. For purposes of comparison twigs have also been distilled and the whole of two trees.

The distillations have been performed on a "semi-large scale," from 30 to 40 lb. of leaves being distilled at one time in a specially designed still (see Puran Singh, Chem. Eng., 1915, 45, 185), having double surface condensers which enable a portion of the camphor to be condensed directly and the oil to be collected separately.

The results of the experiments are tabulated in Tables 1, 2, and 3, the yields being calculated on the dry leaves, twigs, or wood.

TABLE I.
Distillation of leaves.

Year.	Month.	Camphor.	Oil.	Total.
<i>I. From coppiced trees grown at Dehra Dun.</i>				
1917	April	0.59	4.24	4.83 (young leaves).
1917	April	0.25	1.63	1.88 (old leaves).
1917	July	—	—	3.06 (young leaves).*
1917	July	—	—	3.53 (old leaves).*
1917	October	—	—	1.5 (young leaves).*
1917	October	—	—	3.2 (old leaves).*
1918	June	0.26	4.01	4.27
1918	August	0.34	3.79	4.13
1918	October	0.38	3.66	4.04
1919	June	0.67	3.88	4.56
1919	August	0.56	3.85	4.41
1919	October	0.84	2.5	3.34
<i>II. Leaves from trees in Burma.</i>				
1916	October	1.03	0.48	1.51
1919	May	0.30	1.36	1.66
<i>III. Leaves from trees on Madupatty Estate, Madras.</i>				
1914	December	1.99	0.63	2.62
<i>IV. Leaves from Cochin.</i>				
1918	March	2.01	0.32	2.33

* Owing to the small quantity of leaves available these distillations were conducted in the laboratory and are therefore not strictly comparable with the other distillations.

TABLE II.
Yield of oil from twigs (grown at Dehra Dun).

Year.	January.	June.	August.	October.
	%	%	%	%
1917	—	—	—	0.34
1918	0.46	0.21	0.24	0.20
1919	—	0.19	0.3	0.61

The yield of camphor and of camphor oil is thus satisfactory and compares not unfavourably with the yield obtained elsewhere (cf. Parry, "Essential Oils," p. 151).

The leaf oil, from which 38% of camphor had been separated, was carefully investigated and was found to contain *d*- α -pinene, dipentene, cineol, terpineol, caryophyllene, and a trace of cadinene. Safrol, which is present in camphor wood oil, was

absent. The presence of caryophyllene in camphor oils has not, so far as the authors are aware, been previously observed. In the oil examined it was the principal sesquiterpene present; bisabolene could not be detected and cadinene only by means of its colour reactions. Sufficient of the sesquiterpene fraction was not available for so detailed an investigation as was made by Semmler and Rosenberg (Ber., 1913, 46, 768).

TABLE III.
Distillation of trees.

Part of tree.	Camphor.	Oil.	Total.
<i>I. Tree grown at Dehra Dun. Age 17 years.</i>			
Leaves and short twigs..	0.42	0.74	1.16
Twigs	0.31	0.26	0.57
Small branches	0.25	0.15	0.40
Large branches	0.51	0.43	0.94
Stem	0.97	0.86	1.83
<i>II. Tree grown at Dehra Dun. Age 22 years.</i>			
Leaves	1.32	0.69	2.01
Twigs	nil	0.12	0.12
Branches green bark	nil	0.13	0.13
Minor branches (thick bark)	0.14	0.29	0.43
Thick branches	0.50	0.25	0.75
Stem	0.69	0.29	0.98
<i>III. Tree grown in the Royal Botanic Gardens, Calcutta. Age unknown.</i>			
Leaves	0.54	1.5	2.04
Small branches	nil	0.07	0.07
Large branches	nil	0.17	0.17
Stem	nil	0.95	0.95
Roots	nil	2.03	2.03

The oil from the twigs resembled that from the leaves, except for the absence of cineol. It contained much less camphor in solution, but this is a matter of little importance, since in any case the yield of oil from the twigs is too small to warrant their commercial distillation.

The wood oil from Indian camphor trees is similar, though apparently simpler in constitution to that from other places. It contains safrol in considerable quantity, but cineol is absent. The sesquiterpene fraction contained caryophyllene and cadinene. A considerable proportion of a high boiling fraction was isolated which was possibly identical with Semmler and Rosenberg's camphoracene. Sufficient was not, however, obtained for investigation.

It thus appears probable that the distillation of camphor and of camphor oil from the leaves of coppiced trees of the *Cinnamomum camphora* could be profitably undertaken, but before a definite opinion can be expressed it must be determined how frequently the leaves can be plucked and the annual acreage yield. Experiments in this direction are in progress.

Experimental.†

Camphor leaf oil.—The oil available for these experiments was pale yellow, and on cooling in a freezing mixture did not deposit any appreciable quantity of camphor. It had sp. gr. 30°/30° 0.9165, $n_D^{30}=1.47$, $[\alpha]_D^{30}=+32.74$. After washing with dilute alkali to remove any acids and phenols,* the oil was carefully fractionated at the ordinary pressure (705 mm.), using a four-pear Young still-head, when the following fractions were obtained: 150°—190° (sp. gr. 0.8795, $n_D=1.463$, $[\alpha]_D=+25.55$) 41.7%; 190°—212° 39%; residue boiling above 212° 19.3%. The second fraction, which was semi-solid,

† All specific gravities were taken at 30°/30° C. and refractive indices and rotatory powers at 30° C.

* The acids and phenols present did not comprise more than 0.1% of the total oil and were not further investigated.

owing to the separation of camphor, was thoroughly cooled in a freezing mixture and the camphor (22.2 g.) removed by filtration, the filtrate being repeatedly re-fractionated and the camphor removed each time by cooling.†

The three fractions, after the removal of the greater part of the camphor, were systematically fractionated three times, when the following separation resulted, camphor being removed from fractions 7–12 by freezing.

No.	B.p.	Sp. gr.	n_D	$[\alpha]_D^{20}$	Composition, C, % H, %	Approx. yields, %
1	155°–160°	0.8564	1.461	+19.53	86.2 11.8	6.3
2	160°–165°	0.8621	1.4625	+19.52	84.0 11.5	7.9
3	165°–170°	0.8709	1.4627	+20.82	83.5 11.5	6.3
4	170°–175°	0.8797	1.463	+23.41	81.7 11.1	5.7
5	175°–180°	0.8905	1.463	+27.13	81.2 11.3	3.1
6	180°–185°	0.8998	1.464	+29.63	78.3 9.8	1.9
7	185°–190°	—	—	—	—	1.3
8	190°–195°	—	—	—	—	1.7
9	195°–220°	—	—	—	—	3.4
10	220°–225°	—	—	—	—	1.3
11	225°–230°	—	—	—	—	1.2
12	230°–235°	—	—	—	—	0.5
13	235°–240°	—	—	—	—	0.5
14	above 240°	—	—	—	—	9.2
15	camphor	—	—	—	—	38.9
Loss (by difference)						10.8

As fractions 7–12 still contained camphor not removable by cooling they were treated with phenylhydrazine. For example, fraction 7 (b.p. 185°–190°, 22 g.) was mixed with alcohol (20 c.c.), phenylhydrazine (5 g.), and acetic acid (3 g.), the mixture heated on the water bath for two hours and then steam-distilled. The oil which passed over in the steam was extracted with ether, and the ether dried and evaporated. The residual oil (16 g.) distilled at 175°–180°. The residue not volatile in steam (9 g.) distilled at 250°–260° (100 mm.), and consisted of camphor phenylhydrazone.

By treating all the fractions 7–12 in this manner camphor phenylhydrazone (97 g.) was obtained, which corresponds to an additional camphor content of 3.6%. After this treatment practically the whole of the oil volatile in steam distilled below 225° at 705 mm.

Fractions 1 and 2, which were examined separately, consisted essentially of *d*- α -pinene, as confirmed by the preparation of pinene nitrosochloride, which after precipitation from chloroform solution by methyl alcohol decomposed at 109° and yielded on treatment with piperidine the nitrol-piperide, which crystallised in tablets m.p. 118°–119°. Tests for β -pinene and camphene gave negative results.

Fraction 3 was apparently free from dipentene, since it gave no dipentene tetrabromide on treatment with bromine. It yielded a small quantity of pinene nitrosochloride, whilst on treatment with phosphoric acid it gave a good precipitate of the cineol compound. Quantitative estimation indicated the presence of 0.7% of cineol (calculated on the total oil).

Fractions 4, 5, and 6 consisted mainly of dipentene and cineol; limonene, phellandrene, and terpinene could not be detected. The presence of dipentene in each fraction was proved by the isolation of the tetrabromide m.p. 125°. Cineol was estimated in fraction 4 by the phosphoric acid method,

† The method usually recommended for the determination of camphor in camphor oils (Allen, "Organic Analysis," Vol. IV, 325) invariably gave very low results owing to a considerable quantity of camphor being carried over both in the high and low boiling fractions and remaining dissolved in the oil. It was found convenient to remove such camphor by treatment with phenylhydrazine (*cf. infra*). It is hoped to test the accuracy of this as an analytical method.

which gave 1.4% (calculated on the total oil). The high rotation observed in all these fractions was due to camphor.

Fractions 7–12 were freed from camphor by means of phenylhydrazine and fractionated. The main portion distilled at 165°–167° (200 mm.), and consisted almost entirely of terpineol. A careful examination of these fractions and also of fraction 13 showed that safrol was absent.

Fraction 14 (the sesquiterpene fraction), when distilled under diminished pressure (70 mm.), was separated into two main fractions, (i) 150°–170°, (ii) 170°–185° (7%), the residue remaining in the flask being 2.2%. These two fractions were repeatedly re-fractionated, when three fractions were obtained, viz.:—(1) b.p. 151°–155° (39 mm.); sp. gr. 0.8969; n_D = 1.496, $[\alpha]_D$ = –3.0. (2) b.p. 158°–162° (40 mm.); sp. gr. 0.9014; n_D = 1.4986, $[\alpha]_D$ = –0.1. (3) b.p. 170°–210° (40 mm.).

Fraction (3) was not obtained in sufficient quantity for investigation. It was deep blue in colour, and evidently consisted mainly of the so-called "blue oil."

Fractions (1) and (2) possessed constants agreeing closely with those of caryophyllene (b.p. 148°–149° (34 mm.) sp. gr. 0.89789, n_D = 1.4965, $[\alpha]_D$ = –10.94), and the presence of this sesquiterpene was proved by the preparation of the nitrosate and nitrosochloride. The nitrosate, separated in needles from a mixture of chloroform and light petroleum, decomposed at 163° (Deussen, Ann., 1909, 369, 42, gives 162° as the melting point of α -caryophyllene nitrosate); the nitrosochloride decomposed at 158°–160°, which is the point usually found for a mixture of the α - and β -nitrosochlorides. An attempt to separate them by Deussen's method (Ann., 1907, 356, 2) was not successful, but by crystallisation from benzene, the almost pure α -nitrosochloride was obtained in fine needles decomposing at 173°. Mansukhani and Sudborough (J. Ind. Inst. Sci., 1918, 2, 43) were likewise unable to separate the two nitrosochlorides by Deussen's method. The presence of caryophyllene was further confirmed by the preparation of caryophyllene alcohol, m.p. 95°–96°. When a little of the sesquiterpene was dissolved in acetic anhydride and treated with a drop of concentrated sulphuric acid, the characteristic colour reactions of cadinene were observed, but it was not found possible to isolate any solid derivatives of this hydrocarbon.

Camphor twig oil.—The oil was deep brown in colour, and had sp. gr. 0.8777, n_D = 1.473. It was too deeply coloured for its rotatory power to be determined. After washing with alkali to remove any traces of free acids or phenols it was distilled three times, using a four-pear Young still-head, and the following fractions collected:—

No.	B.p.	Sp. gr.	n_D	$[\alpha]_D$	Yield.	Constituents.
1	155°–165°	0.8544	1.464	+22.45	20%	<i>d</i> - α -pinene
2	165°–170°	0.86	1.4662	+30.14	5.8	<i>d</i> - α -pinene (β -pinene absent).
3	170°–175°	0.8665	1.467	+35.41	5.8	dipentene, camphor.
4	175°–185°	0.8795	1.469	+38.82	5.8	dipentene, camphor.
5	185°–195°	—	1.4701	—	1.0	camphor.
6	195°–225°	—	—	—	14.2	terpineol, camphor.
7	225°–245°	—	—	—	6.5	terpineol, camphor, sesquiterpenes.
8	above 245°	—	—	—	16.3	caryophyllene cadinene.
9	camphor	—	—	—	14.2	

β -Pinene, phellandrene, terpinene, cineol, and safrol were absent from these fractions. Fraction 6

contained approximately 6% of camphor as estimated by the phenylhydrazine method, which brings the total camphor content of the oil to about 20%. The sesquiterpene fraction consisted essentially of caryophyllene; cadinene was detected by its colour reactions. The sesquiterpene distilled mainly at 160°–165° (50 mm.), and had sp. gr. 0.8987, $n_D = 1.4970$, $[\alpha]_D = -1.57^\circ$.

Camphor wood oil.—The oil used was pale yellow in colour, and had sp. gr. 0.9313, $n_D = 1.4767$, $[\alpha] = +34.44^\circ$. After washing with dilute alkali to remove the acids and phenols, the oil was repeatedly distilled, using a twelve-pear Young still-head, when the following fractions were obtained:—

No.	B.P.	Sp. gr.	n_D	$[\alpha]_D$	Yield.	Constituents.
1	155°–165°	0.8484	1.4645	+32.62	12.2	<i>d</i> - α -pinene.
2	165°–170°	0.8503	1.4668	+39.46	4.0	<i>d</i> - α -pinene
3	170°–175°	0.8561	1.4685	—	2.5	dipentene.
4	175°–185°	0.8664	1.4690	+50.31	3.7	dipentene & camphor.
5	185°–195°	—	1.4695	—	2.2	camphor.
6	195°–225°	—	—	—	24.8	camphor, terpineol, safrol.
7	225°–240°	—	—	—	7.4	safrol, sesquiterpenes.
8	above 240°	—	—	—	17.7	caryophyllene, cadinene, camphoracene.
9	camphor	—	—	—	27.0	

The following substances were absent:— β -pinene, phellandrene, terpinene, cineol. Safrol was readily detected in fractions 6 and 7 after removal of the camphor by means of phenylhydrazine. The fraction boiling at 225°–232° at 705 mm. (yield 3.5%) was oxidised with potassium permanganate in the usual manner, when piperonylic acid was obtained which, after crystallisation from hot water, melted at 227°–228° and gave a crystalline silver salt (Ag=39.4%; calc. Ag=39.8%). The sesquiterpene fraction differed from those obtained from the leaf and twig oils in that it contained a high boiling terpeno possibly identical with camphoracene. It was not present in sufficient quantity for examination. "Blue oil" was practically absent.

In conclusion the authors wish to express their thanks to Mr. Puran Singh, formerly Assistant Forest Chemist, for making the earlier experiments,

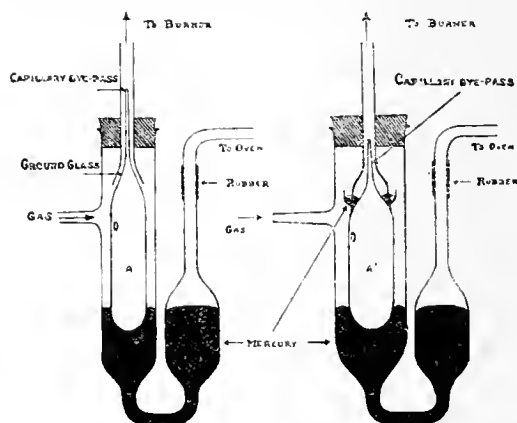
and to Messrs. Troup, Marsden, and Howard, who have collected and supplied the material used for distillation.

Forest Research Institute and College,
Dehra Dun, India.

AN AIR-OVEN THERMO-REGULATOR.

BY H. M. ATKINSON.

The float, A, loaded slightly with mercury or small shot, carries at the top a small capillary tube, which acts as the by-pass. Gas coming through the side tube passes freely to the burner, till the pressure on the mercury reservoir resulting from the expansion of air in the tube inside the oven raises the float upwards and cuts off the main supply by either of the two methods illustrated.



The apparatus is small and compact; it need not be more than 2–2½ in. long, and avoids the soiling action of the gas on the mercury, which is often a cause of trouble in the ordinary form of regulator.

When the float is raised and cuts off the main supply, a little gas can still reach the burner passing through the hole in the side of the float and the capillary. The capillary itself can be closed or widened to any desired extent. The bulb inside the oven is best made of one or two turns of glass tubing, about $\frac{1}{16}$ in. bore, to avoid too rapid action and to equalise the temperatures inside.

Manchester Section.

Meeting held at The College of Technology on
Friday, April 23, 1920.

MR. JOHN ALLAN IN THE CHAIR.

DISTILLATION PLANT, WITH SPECIAL REFERENCE TO THE RECTIFICATION OF ALCOHOL, ACETALDEHYDE, AND OTHER VOLATILE PRODUCTS AND CONTINUOUS ETHER MANUFACTURING PLANT.

BY STEPHANE MASFARAUD.

The object of distillation may be defined as being the separation of a volatile liquid from a non-volatile substance, and, more generally, the more or less complete separation of liquids possessing different rates of volatilisation.

In order, however, to obtain products in the highest state of purity, it is necessary to effect several consecutive distillations, a process called "fractional distillation" in the laboratory or "rectification" in industry.

Distillation of binary mixtures of immiscible liquids.

It is well known that if a mixture of water and turpentine is boiled in a still, the turpentine, b.p. 160°C ., passes over into the distillate in greater quantity than the water at a temperature below 100°C .; also that water vapour carries over mercury at a temperature below 100°C . At atmospheric pressure a mixture of ethylene dichloride with water has a fixed b.p. (75.6°C .), which is lower than that of water or of pure ethylene dichloride (82.4°).

Since the constituents of such mixtures are without action on each other, it follows that the vapour pressure, P , of the mixture is equal to the sum of the partial pressures, F and F' , of the liquid constituents at the boiling temperature, T , of the mixture. F and F' separately therefore must be less than P . Thus in the cases mentioned the boiling temperature, T , is lower than that of either of the two liquids boiling in the pure state under pressure P .

It is possible to forecast the composition of a distilled liquid. If A and B represent the respective weights of the two liquids volatilised simultaneously, and D and D' the densities of their vapours,

$$\frac{A}{B} = \frac{D}{D'} \times \frac{F}{F'}$$

and since D and D' are proportional to the respective molecular weights, M and M' ,

$$\frac{A}{B} = \frac{M F}{M' F'} \quad \text{in which } F + F' = P.$$

This statement sums up the distillation of a binary mixture of immiscible liquids.

The formula $P = F + F'$ is verified by the following experiments carried out by Gernez so long ago as 1878:—

1. Carbon bisulphide and water (CS_2 has b.p. 46.04°). $T = 43.68^{\circ}$; $P = 765.35$ mm.; $F + F' = 766.61$ mm.; $(F + F') - P = 1.29$ mm.

2. Carbon tetrachloride and water (CCl_4 has b.p. 78.1°). $T = 66.17^{\circ}$; $P = 745.45$ mm.; $F + F' = 747.36$ mm.; $(F + F') - P = 1.91$ mm.

3. Benzene and water (C_6H_6 has b.p. 80°). $T = 69.42^{\circ}$; $P = 764.44$ mm.; $F + F' = 765.92$ mm.; $(F + F') - P = 1.48$ mm.

The result of many experiments leads us to conclude that:—

- (1) The formula $\frac{A}{B} = \frac{M F}{M' F'}$

is exact, the differences observed being so slight that the law may be regarded as general.

(2) Variations of temperature do not modify that law.

(3) If, for one of the substances, M and F are small in proportion to M' , the distilled weight, B , will be less than the distilled weight, A .

Of all the substances that are dealt with in distillation processes water has the lowest value for M/F . This at once accounts for the fact that steam distillation rapidly extracts essential oils from flowers or leaves, even at a low temperature, and for the fact that water carries over toluene at 27.5° , aniline at 23° , nitrobenzene at 22.5° under 23–27 mm. pressure.

In general, then, it may be said that in all cases where, at a given temperature, the resultants of $M \times F$ and $M' \times F'$ are distinctly different the separation of the two substances by distillation is easy. If $M \times F$ and $M' \times F'$ are nearly equal, the two substances are difficult to separate.

If M is greater than M' , F must necessarily be smaller than F' .

To modify the proportions of the two substances in the distillate the ratio $M F / M' F'$ must be increased or diminished so as to make it differ from 1. If it is increased by creating conditions such that F/F' becomes larger or smaller, the equality $M F / M' F'$ must cease to exist and the two substances be more easily separable.

The question whether the ratio F/F' should be increased or diminished to effect the separation of the substances must depend on the nature of the mixed substances. Let us suppose two substances, A and B , mixed, and boiling at a temperature, T , for which $M F = M' F'$. If by increasing the pressure T is raised and in consequence A is distilled in larger proportion, it follows that if the boiling point is reduced by diminishing the pressure, B will be distilled in the major quantity.

In each particular case, therefore, the products $M \times F$ and $M' \times F'$ must be ascertained for the given mixture at the boiling point under the given pressure, so that it may be ascertained whether the boiling point must be raised or lowered in order to separate the mixed substances as rapidly as possible.

The influence of pressure is made manifest in alcohol rectifiers and especially in the distillation of wines, where it has been observed that the distillate from wines distilled under pressure has a stronger flavour. In this special case, however, a certain amount of esterification takes place, a condition favoured by the increased distillation temperature. The spirit obtained by vacuum distillation, for that reason, contains only a small amount of ester. A few esters, being insoluble, are volatilised only when the pressure, i.e., the ratio F/F' , is propitious. Generally they form a true emulsion, and with the oils properly so-called they are to be found in the vinasses and residuary waters.

Conclusions.—Given a mixture containing two immiscible liquids, of which the molecular weights are known, it is possible to determine by calculation:—

- (1) The boiling point under a given pressure;
- (2) The composition of the evolved vapours;
- (3) The composition of the residue after partial distillation; and
- (4) The best method of separating the substances, either by raising or by lowering the boiling point by varying the pressure.

We have considered, so far, only the case of a mixture of two pure liquids. The presence of dissolved salts may modify the results.

Distillation of a binary mixture of miscible liquids.

The simple laws established for mixtures of insoluble liquids cannot be applied when the liquids

are mutually soluble. In this case the laws are complex, and for a long time distillation of such mixtures was purely empirical.

Regnault divided mixtures of miscible liquids into two classes. The first class includes substances which dissolve, more or less, but of which such quantities are used that, after reciprocal saturation, two separate liquids remain. The second class consists of binary mixtures of substances which dissolve in all proportions and always resolve into a single liquid.

The vapour pressure of a mixture of water and ether, which belongs to the first class, is nearly equal to that of pure ether. The following table gives the results obtained by Regnault for this mixture:

Vapour pressure in mm.				
Temperature.	Mixture.	Water.	Ether.	
15-56° ..	362-95 ..	13-16 ..	361-4 ..	
24-21° ..	510-08 ..	25-30 ..	500-0 ..	
33-08° ..	710-62 ..	27-58 ..	711-6 ..	

With mixtures of the second class, such as ethyl alcohol and water, ethyl alcohol and ether, carbon bisulphide and water, ethyl alcohol and ether, carbon bisulphide and carbon tetrachloride, the ratio of the vapour pressure of the mixture to the sum of the vapour pressures of the two substances is nearly constant whatever may be the temperature.

Distillation of mixtures of water and ethyl alcohol.

The first important study of the principles of such distillation was made by Duclaux, who distilled a definite volume of an alcoholic liquid of known composition and collected the distillate in equal fractions, which he afterwards analysed.

term dPa may be neglected and we have $dPU = Pda + dPa$.

$$\text{Whence: } U = a + P \frac{da}{dP}$$

Tracing the curve of exhaustion and determining the angular coefficient of the tangent to that curve, we obtain U .

This method was employed by Duclaux and Sorel in studying the distillation of alcoholic liquids.

Duclaux has established the relation:—

$$\frac{da}{de} = M \frac{a}{a+e} \quad \dots \dots \dots (i)$$

in which a represents the percentage (by vol.) of alcohol in the original liquid, e that of water, da the percentage of alcohol in the vapours, and de that of water in the vapours.

The relation (i) is represented by a hyperbola of which the equation is:—

$$\frac{y}{100-y} = Mx \quad \left(y = da; \quad x = \frac{a}{a+e} \right)$$

The following values have been obtained for M with mixtures of water with certain alcohols:—

		a=not more than		M.	
Alcohol.					
Methyl	30%	..	10-9	
Ethyl	25%	..	15-4	
Propyl	10%	..	20-9	
Butyl	4%	..	41-5	
Amyl	1-6%	..	49-6	
Capryl	0-25%	..	61	

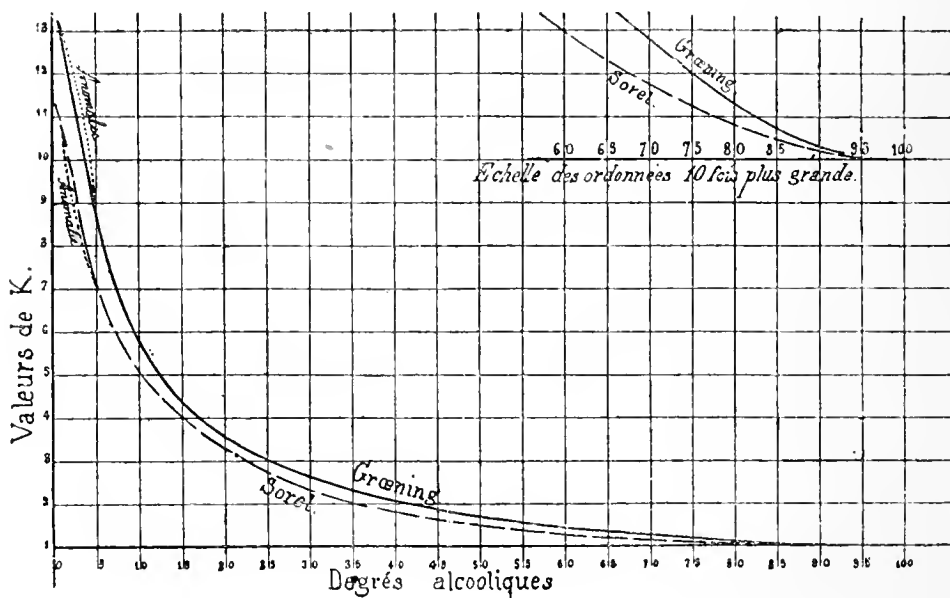


Fig. 1.

If P is the weight remaining at a particular time, a the percentage of the particular substance under examination in the mixture, and U percentage of the substance in the fractions distilled, then the weight of the substance in the original mixture must be equal to the total quantities of that substance that are found in the residue and in the vapours evolved and condensed, that is:—

$$Pa = (P - dP)(a - da) + dPU,$$

or

$$dPU = Pda + dPa - dPda.$$

But as, by hypothesis dP is infinitely small, the

If, therefore, a coefficient, k , is determined so that:—

$$k = \frac{da}{a} = \frac{\text{richness of vapour}}{\text{richness of liquid}}$$

or $da = ka$, it shows clearly the tendency of the phenomenon.

The following are some values of k as given by Sorel:—

$a=1, k=9.9; a=5, k=7.15; a=20, k=3.3; a=40, k=1.8; a=60, k=1.3; a=80, k=1.08; a=95, k=1.003; a=97, k=1.001.$

From 95% to 100%, k is very nearly equal to 1, which means that if one of the substances under distillation is present only in extremely small

GRAPHIQUE DE LA TABLE DE GROENING

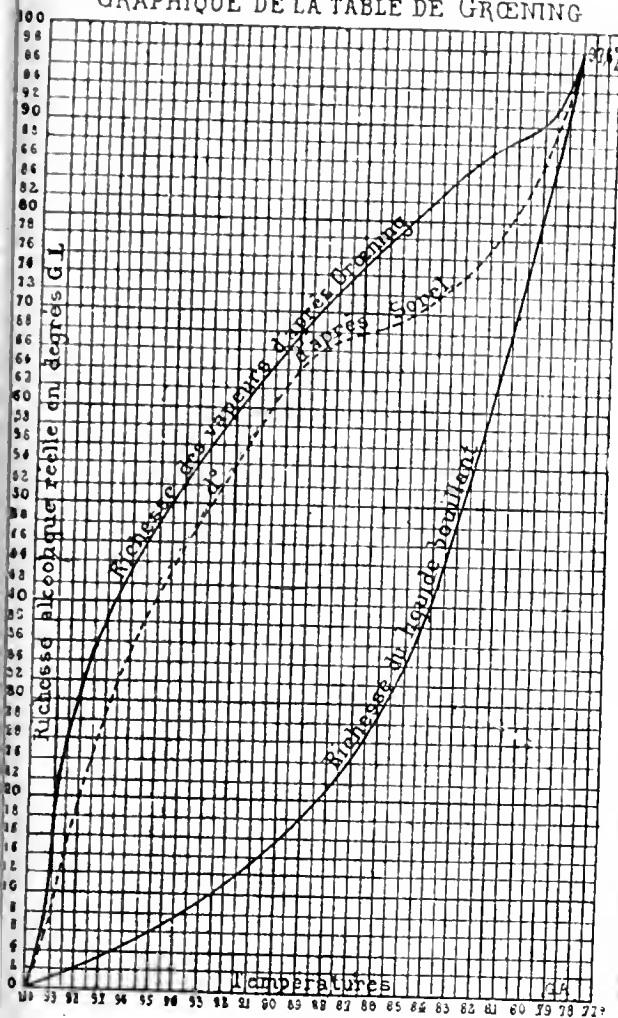


Fig. 2.

quantities it is strongly retained by the substance present in abundance.

To solve problems relating to the distillation of mixtures of miscible liquids it is necessary in a series of experiments to determine the vapour

Sorel's table giving the coefficient, k , may be taken as theoretically exact, but it must not be forgotten that it is essentially theoretical, and therefore very different results may be obtained in practice.

This was first pointed out by E. Barbet (Bull. Assoc. Chim. Sucr., January, 1902) as follows:—"The coefficients of Groening and Sorel are true only for surface heating. They vary with the intensity of heating, and with the bulk of liquid used. Thus they are only true in the special circumstances under which these authors worked and between rather narrow limits."

The slower and more gentle the ebullition the more the coefficient of concentration of alcohol is raised. "The people of Charente have known for a long time that they can obtain *eau-de-vie* of higher strength by gentle heating than by violent ebullition. Gentle ebullition permits of the total exhaustion of wine, containing 10% of alcohol by vol., if only one-third of its volume be distilled, while in the laboratory M. Duclaux found that to obtain exhaustion it was necessary to distil half the volume even for wine of 5% strength. The difference was due to the different methods of applying heat to the distillation vessel."

Sorel studied the special case presented by alcohol rectification plant dealing with alcoholic liquids in industry. In these liquids, two substances predominate, water and alcohol; the impurity is present only in small quantities. If S is the weight of this third substance contained in 1 kg. of mixed vapours, and s the weight in 1 kg. of the original liquid,

$$S = ks + k's^2 + k''s^3 + k'''s^4 + \dots$$

but since s is very small, terms containing powers of s may be neglected, and the equation becomes $S = ks$.

Sorel, from the experimental data he obtained, calculated the values of k for the different alcoholic concentrations, for eight different substances, constantly maintaining s below 0.02. The appended table gives the results thus obtained.

Sorel called the coefficient, k , the "coefficient of solubility." Barbet substitutes for Sorel's "coefficient of solubility" one of much greater interest and utility, which he has termed the "coefficient of purification," as it actually shows the behaviour of alcohol during the process of purification. Barbet argues that in distillation processes the matter of special interest is the ratio of impurity of the vaporised alcohol to the impurity of the alcohol in the liquid under distillation. This ratio is not given by the coefficient k . Thus, with alcohol at 25% by volume, containing 0.01% of amyl alcohol, in relation to ethyl alcohol alone, $k=5.5$, i.e., the vapour eliminated carries with it per 100 c.c. of condensate $25 \times 5.5 \div 100 = 1.375$ c.c. of amyl alcohol. But the vapour is richer in alcohol than the liquid under

Values of k for various substances added.

% by vol.	Amyl alcohol (fermentation).	Ethyl formate.	Methyl acetate.	Ethyl acetate.	Ethyl isobutyrate.	Ethyl isovalerate.	Amyl acetate.	Amyl isovalerate.
10	—	—	—	29	—	—	—	—
20	5.5	—	—	18	—	—	—	—
25	5.5	—	—	15.2	—	—	—	—
30	3.0	—	—	12.6	—	—	—	—
40	1.62	—	—	8.6	—	—	—	—
50	1.20	—	10.5	5.8	—	—	—	—
60	0.80	10.4	7.9	4.3	—	—	2.8	—
70	0.54	9.5	6.4	3.6	4.2	2.3	1.7	1.30
80	0.34	7.2	5.4	2.0	2.3	1.7	1.1	0.82
90	0.30	5.8	4.6	2.4	1.4	1.3	0.8	0.50
95	0.25	5.1	4.1	2.1	1.1	0.9	0.6	0.35
Bolling point	132°	51.3°	56°	77.1°	110.1°	134.3°	147.6°	196°

pressures of several mixtures of two of these liquids, and then to introduce the third substance and again determine the vapour pressures, etc.

distillation, being, according to Sorel, at a concentration of 67.95% by volume. The impurity of the vapours, that is, the proportion of amyl alcohol

in relation to ethyl alcohol, will therefore be $1.375 \times 100 \div 67.95 = 2.02$.

The "coefficient of purification," k' , of Barbet, is thus 2.02. This coefficient gives more exact and accurate information as to the progress of purification of the product under consideration.

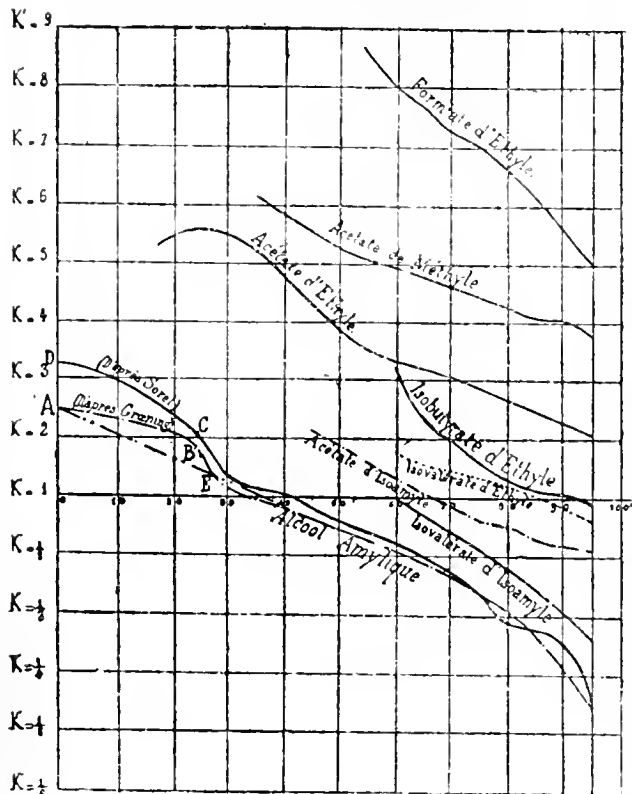


Fig. 3.

Barbet has constructed a graph which gives the values of k' for the various impurities studied, and represents the relative proportions of impurity in the liquid under distillation and in the evolved vapour. If, for example, towards 65% by volume the coefficient of purification of amyl alcohol is $k' = 0.50$, the proportion of amyl alcohol in the resi-

Sorel has only examined cases where the impurity is less than 2% and his coefficients should not be applied to higher percentages. In practice also the alcoholic liquid does not contain only one impurity, but a number (acids, aldehydes, ethers, alcohols, amines, etc.), which influence each other and to a certain extent also the coefficient of purification.

All the impurities under consideration are represented by descending curves, since in proportion as the alcohol concentration increases, the temperature decreases and consequently the vapour pressure of the impurity and its tendency to entrainment decreases also. On the other hand, all the substances considered here are very soluble in alcohol and only slightly or not at all soluble in water; since their molecular weights are higher than that of water, and in pure water they form a mixture of two immiscible liquids and are rapidly carried over by vapour, k' is very great for the lower alcohol concentrations.

Cases of very complex mixtures. Natural hydrocarbons.

When the mixtures are very complex the products are only roughly fractionated by distillation. This is called the law of predominance. Some separation is certainly effected, but it is necessarily very imperfect, for if the most volatile products predominate in the vapours, they are accompanied by very heavy products.

For example, crude petroleum was distilled, and the fraction passing over on distillation from 60° to 156° C. collected separately. This fraction had sp. gr. 0.779. By a fresh fractionation this product furnished 50% of various fractions boiling up to 150°; 10% from 150° to 162°, sp. gr. 0.792; 10% b.p. 162°–200°, sp. gr. 0.806; 10% b.p. 200°–210°, sp. gr. 0.822; and 20% of residue. The fractionation in such a case as this can never be more than relative, i.e., the final mixture will be a little less complex than the initial one.

Distillation apparatus.

For discontinuous distillation the simplest still consists essentially of the boiler or cucurbit, heated directly by a furnace, or indirectly by steam, and the still-head directing the vapours through a pipe into a coil immersed in cold water.

Continuous distillation.

The columns are generally vertical, and receive the fermented mash or wine to be distilled in the upper part, being heated at the base. The liquid, in descending, meets many obstacles which subject

Values of k' (Barbet's coefficient).

Alcohol % by vol. in liquid under distillation.	Amyl alcohol.	Ethyl formate.	Methyl acetate.	Ethyl acetate.	Ethyl isobutyrate.	Ethyl isovalerate.	Amyl acetate.	Amyl isovalerate.
1	3.30	—	—	—	—	—	—	—
10	—	—	—	5.07	—	—	—	—
25	2.02	—	—	5.43	—	—	—	—
30	1.30	—	—	5.43	—	—	—	—
40	1.05	—	5.83	4.77	—	—	—	—
50	0.80	—	5.26	3.86	—	—	—	—
60	0.615	8.0	4.92	3.3	3.23	1.76	1.866	1.0
70	0.44	7.26	4.61	3.07	1.96	1.45	0.94	0.7
80	0.36	6.6	4.25	2.77	1.30	0.74	0.74	0.463
90	0.26	5.68	4.01	2.37	1.07	0.882	0.588	0.343
95	0.22	5.08	3.78	2.09	0.897	0.797	0.548	0.299

due is twice that in the condensate. At 25% by vol. $k' = 2.0$, and the vapour is twice as impure as the liquid. So long as $k' > 1$ the vapours contain more impurity than the liquid under distillation. When $k' = 1$, the two degrees of concentration are identical. When $k' < 1$, the impurity concentrates in the liquid.

it to contact with vapour, and escapes at the lowest part as a residue free from alcohol. Continuous distilling systems may be divided into four classes: (1) Systems with heating surfaces. (2) Bubbling systems. (3) Contact systems, including (a) horizontal columns with mechanical movement; (b) percolating columns. (4) Full columns.

Rectification.

Rectification may be discontinuous or continuous.

The *discontinuous rectifier* is only in reality a high-strength still, and contains the four essential parts of this apparatus; the boiler, the rectification column, the condenser, and the tubular cooler.

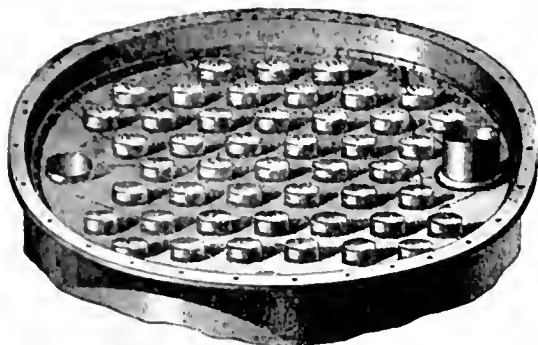


Plate with perforated caps for cylindrical columns.

Fig. 4.

In discontinuous rectifiers losses of alcohol occur which vary with the apparatus, the quality of the phlegms, the duration of the operation, etc.

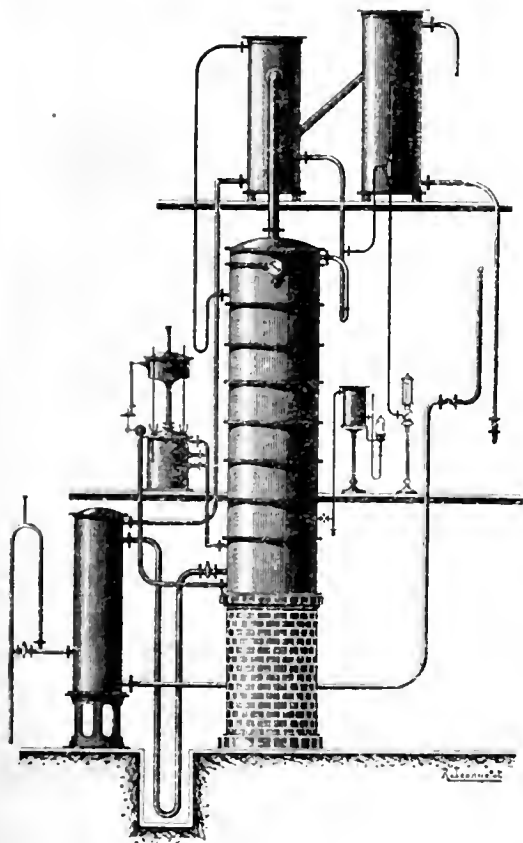


Fig. 5.

Continuous rectification has the advantage of constant and invariable working, thereby ensuring regular production of uniform products with the minimum of fuel. Instead of fractionating the

liquor into ten parts more or less pure, a single rectification by the continuous process gives three

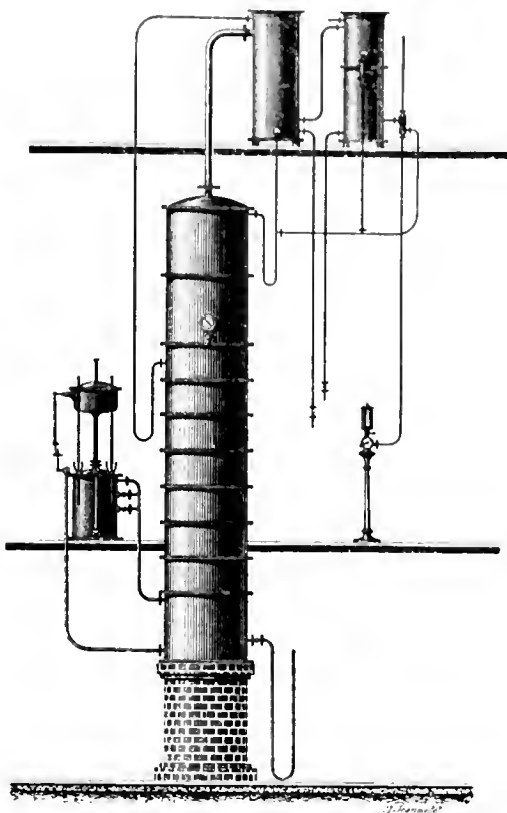


Fig. 6.

parts, the first including all the head products, the second pure alcohol, and the third all the tail products.

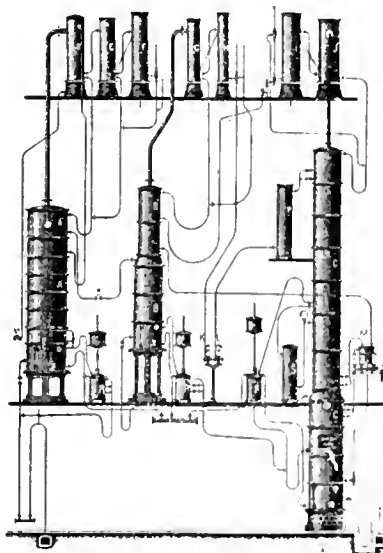


Fig. 7.

About forty years ago M. Barbet made the first continuous rectifier which was of practical use in

industry. His improvement consisted in providing the distillation column with an invariable regulation of the flow, and by adding to it the efficient system of purification known as "pasteurisation," or "superepuration." The continuous rectifier

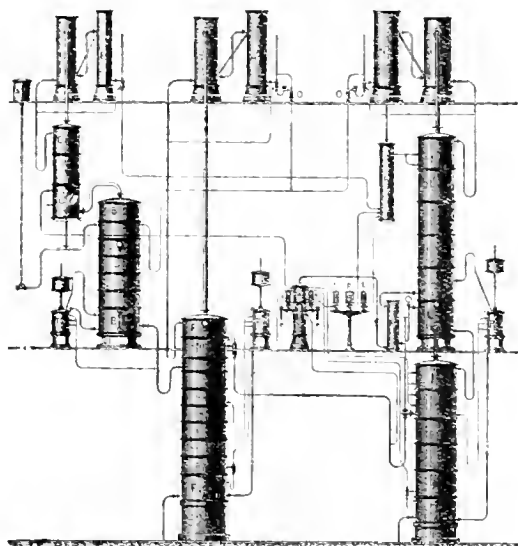


Fig. 8.

yields at the "eprouvettes" all the alcohol charged into it.

Some idea of the different types of distillation and rectification apparatus generally used may be gathered from the following account of some of Barbet's apparatus:

(1) *Distilling columns.*

(a) Fig. 5. Columns for low-strength alcohol should be used in all cases where the phlegm produced is at once rectified in the same factory. When a continuous rectifier is used, the phlegm should be pasteurised, so as to expel all fermentation gases, more especially the sulphurous gases, and also to extract a small proportion of highly-concentrated aldehydes. (b) Fig. 6. Column for high-strength alcohol. This column produces in one operation alcohol of 92–94%, which is suitable for denaturation. It should also be used where the phlegm is sent away to be rectified. Two kinds of phlegm can be produced simultaneously, one ready to "denature," the other pasteurised for subsequent rectification.

(2) *Continuous alcohol rectifiers.*—These Barbet types combine in one apparatus the continuous distilling column and the rectifier for phlegms. This

combination economises pumps and tanks for phlegms. It is also provided with means for a first purification of the phlegms, so that the alcohol is submitted to three successive refinings. It therefore produces spirit of exceptional purity. Besides these there are several simpler types of the Barbet apparatus. (See fig. 7.)

(3) *Continuous rectification of wood spirit.*—

The rectification of wood spirit is based on the same principles as those given for the rectification of ethyl alcohol obtained by fermentation, and can be carried out in some of the types already described, parts of the apparatus being suitably varied. (See fig. 8.)

Instead of three, or even four, successive discontinuous rectifications, with corresponding losses, which often reach and even exceed 8% it is possible to have only one continuous rectification with no appreciable loss. A powerful type of continuous rectifier gives methyl alcohol of 99.5–99.7% containing less than 0.1% of acetone.

Rectification of petroleum and benzol.

Amongst the most important difficulties encountered in distilling crude petroleum are: (1) The high temperature required for the distillation of a heavy petroleum, which makes distillation by steam impossible unless it is carried out in a vacuum. (2) The influence of "cracking," so that the removal of low-boiling constituents can never be completed, since some light products are constantly being formed during the distillation of the heavy ones. (3) A deposit of coke is formed by the "cracking" which adheres to the sides of the still. This coking renders it necessary to stop for cleaning, and, therefore, there can be no question of a

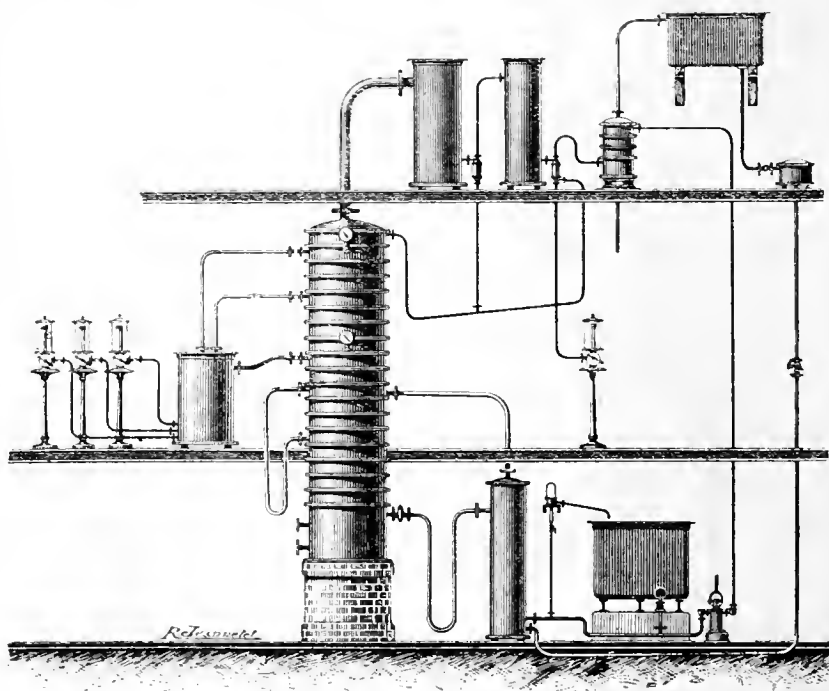


Fig. 9.

continuous operation where direct fire-heating is required in the distillation of high-boiling paraffins such as those containing vaseline or wax.

Where direct fire heat is necessary, distillation is carried out in large stills, and the distillates are

fractionated successively according to the temperature of their formation. These fractions are, however, far from being homogeneous.

Continuous rectification of crude petroleum spirit or of light benzols.

This problem is a very simple one because it is possible to rectify by means of steam and under atmospheric pressure, provided the vaporiser of the still is adapted to high pressure. Continuous rectification will easily fractionate crude petroleum

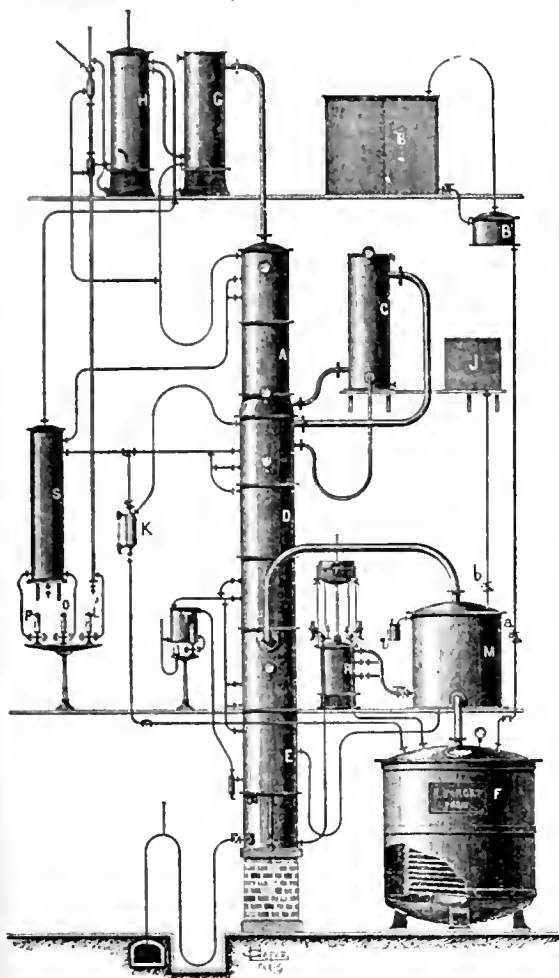


Fig. 10.

spirit in one operation into seven portions: gasoline of sp. gr. 0.65; spirit for soldering lamps, sp. gr. 0.68; various products of sp. gr. 0.70, 0.715, 0.73, 0.74; white spirit, sp. gr. 0.75—0.76; and some burning oil, which is not distilled but is discharged from the lower part of the apparatus (fig. 9).

Sulphuric ether apparatus.

This apparatus is in use in the powder factories of Great Britain, France, Italy, Russia, etc., and in the principal factories making artificial silks. Sulphovinic acid is placed in a lead-lined still, and alcohol vapours are caused to bubble through the liquid. The vapours leaving the still are washed with caustic soda in a scrubber, and thence pass to a rectification column. Ether of sp. gr. 0.721 at 15° C. can easily be obtained. Several large apparatus of this type built by the author for the British Government are each capable of delivering 30 tons of ether per 24 hours. (See fig. 10.)

Yorkshire Section.

Meeting held at Queen's Hotel, Leeds, on May 3, 1920.

MR W. M'D. MACKEY IN THE CHAIR.

THE COMMERCIAL EXAMINATION OF SULPHONATED OILS.

BY GEORGE FENWICK PICKERING.

The whole of the published methods for the analysis of sulphonated oils can only be regarded as giving approximate results. For example, it appears generally to be taken for granted that sulphonated fatty acids are completely decomposed on boiling with aqueous hydrochloric acid, whereas this is far from being the case.

The hydrochloric acid always causes polymerisation, which unfits the resultant fat for examination. The acetyl value of the fatty acids from sulphonated castor oil may be as low as 85; in no case have I found a figure as high as the 126 given by Lewkowitsch. When the fat from one of these samples is saponified with alcoholic potash and again separated with acid the acetyl value will be between 140 and 146. This saponification and separation of the fatty acids is essential if the separated fat is to give figures in any way comparable with those of the oil sulphonated.

Among other difficulties, the estimation of water is complicated by the charring of samples made from oils of high iodine value, the formation of lactones, and in oils containing ammonia, amide formation. The volumetric process for estimating fat, although still largely used, gives results from 3 to 20% too high. The results obtained by decomposing with hydrochloric acid and shaking out with ether may be too low through formation of poly-acids, and may be too high through the taking up of hydroxyl groups, and the fat extracted with ether contains 0.75—2.75% of combined sulphuric acid.

The refractive index and fat content are proportional. A graph plotted from these is very useful.

Sulphuric acid is present in sulphonated oils as sodium sulphonates of fatty acids, as free sulphonic acids, as free sulphuric acid, as sodium or ammonium sulphate formed during neutralisation, and finally as free sodium sulphate dissolved by the oil during the washing. In the determination of total acid by the fusion method the results may be 0.5—1% too low unless care be taken and time allowed for the initial carbonisation.

The only direct method for determining combined acid is the volumetric method of Hart (J., 1917, 1139), but when the fat from this process is extracted with ether and fused with sodium carbonate 0.7—1.0% more sulphuric acid is produced. The only accurate method of estimating combined acid is by deducting the percentages of the other forms of acid from that of total acid obtained by fusion.

The process for determining free fatty acids and sulphonated oil, which consists in adding glycerol and excess of ammonia, extracting with ether, and removing the water-soluble oils and soaps by washing the ether with water, is slow, and gives a "neutral" oil with 9—13% of free fatty acids; further, the fatty acids obtained later are never free from neutral oil.

The methods described below have given reliable results in routine work:—

A few c.c. of the sample is boiled with three times its volume of hydrochloric acid until the fatty layer is quite clear. The lower layer is drawn off, diluted with water, and tested with barium chloride solution. A precipitate of barium sulphate indicates

the presence of sulphonated oil. If the oil layer obtained in the above test is free from charred particles the methods given under "oils of low iodine value" are used. When charring takes place the methods for oils of high iodine value must be used.

Water.—Distillation with xylol is the most accurate method, but drying in the oven may be used with oils of low iodine value. About 2 g. of the sample in a 1½ in. Petri dish is dried in the water oven, with frequent stirring. If ammonia is present its amount must be deducted from the loss of weight on drying.

Total fat—low iodine value oils.—About 5 g. is boiled with dilute hydrochloric acid (1:1) until the fatty layer is quite clear, allowed to cool, and extracted twice with ether, washing the ether with water until free from acid. The ether extracts are distilled, the fat dried in the water oven, cooled, and weighed.

High iodine value oils.—5 g. is dissolved in 50 c.c. of pyridine, 25 c.c. of strong hydrochloric acid added, and the mixture heated on a water bath for at least 1½ hrs., with frequent shaking; when cool the liquid is extracted twice with ether, the ether washed, distilled, and dried.

Total sulphuric acid.—This is applicable to all samples, but care is necessary if low results are to be avoided. The result cannot be reported as combined plus free acid, since it also includes all the sulphuric acid combined with soda as free sulphate.

The bottom of a 1½ in. platinum dish is covered with powdered sodium carbonate, about 2 g. of the sample is added in small drops, over the surface of the carbonate, and the oil is covered with a further

Since practically all the common salt used is converted into sodium sulphate during the washing the salts left in the oil may be reported as Na₂SO₄.

The brine extracts after titration in the preceding operations are heated to boiling, the sulphate precipitated in the usual way and calculated to sulphuric acid. The amount minus any free acid present is calculated to and reported as free sodium sulphate.

Alkalis.—Ammonia may be detected by boiling 2–3 c.c. of the sample and testing the evolved steam. To estimate the alkalis, 10 g. of the sample is dissolved in water and titrated with N/2 acid, using methyl orange as indicator. In the absence of ammonia the alkali is calculated to sodium oxide. In presence of ammonia a further 10 g. of the sample is distilled with excess of caustic soda into 25 c.c. of N/2 acid, and the result calculated to ammonia. The difference between the two results is recorded as sodium oxide.

Unsulphonated oil.—About 5 g. of the sample is dissolved in 50 c.c. of cold water, the alkali just neutralised with N/2 acid, using methyl orange as indicator, and extracted with 100 c.c. of ether. The ether is washed several times with water until all the water-soluble oil has been removed. The water layer and washings are put aside. The ether extract is mixed with 25 c.c. of water and 25 c.c. of, roughly, N/1 aqueous potash and well shaken, the soap solution separated, and the ether layer washed with water until free from soap, uniting the washings with the soap solution.

The ether, now containing only unsulphonated neutral oil, is distilled, and the residue dried and

Samples.	Sp. gr.	Refr. index.	Acidity.	Total oil.	Water.	Alkali as Na ₂ O.	Ash.	Free Na ₂ SO ₄ .	Combined H ₂ SO ₄ .	Iodine value.	NH ₃ .
85% castor ..	1-0229	1-4561	18-68 to 40-54	—	17-59	0-95	—	—	4-24	—	—
70% castor ..	1-0214	1-3875	6-7	26-44	19-20	0-94	7-20	1-87	6-20	53-7	1-35
60% castor ..	1-1045	1-4502	38-42	75-42	65-06	3-68	7-93	5-05	2-84	58-1	—
50% castor ..	1-0321	1-3850	6-38	26-08	36-95	0-54	—	—	4-55	—	1-27
40% castor ..	1-0550	1-4405	—	57-42	65-32	—	—	—	—	—	—
30% castor ..	1-0125	1-4061	1-51	39-44	39-94	0-19	1-12	—	1-27	34	0-15
Monopol oil ..	1-1039	1-4191	39-48	52-32	54-01	3-12	7-64	—	7-03	—	3-15
Turkon oil ..	1-0175	1-3910	1-06	34-55	52-47	0-98	1-12	—	3-75	—	2-05
85% from 95% oleine	1-1029	1-4300	18-68	42-44	59-98	3-10	7-05	—	3-84	—	—
70% from 95% oleine	1-0140	1-3762	13-30	26-01	68-78	0-71	3-48	—	—	—	—
60% from 95% oleine	1-0371	1-3910	16-22	32-50	62-01	3-40	—	—	—	—	—
50% from 95% oleine	1-0190	1-4371	21-15	62-27	24-84	0-97	5-63	—	3-45	47-1	—
50% from maize oil ..	—	1-4500	31-01	68-79	28-06	—	—	—	—	—	—
50% from seal oil ..	1-0562	1-4380	17-98	55-18	—	1-65	9-80	6-02	2-57	—	—
70% from wool grease	—	—	—	82-50	13-58	—	—	—	—	—	3-03
oleine ..	—	1-4494	—	65-02	25-32	3-67	—	—	—	—	—
50% from herring	—	—	—	26-75	—	—	—	—	—	—	—
oil ..	1-1008	1-4220	32-08	51-62	39-32	0-9	7-15	3-46	2-37	—	—
Dark sole leather oil (chromoline type)	1-0190	—	28-78	52-70	40-27	1-61	7-90	—	—	33-3	—
oleine ..	—	—	—	—	—	—	—	—	—	46-4	—
50% from wool grease	—	1-4656	—	—	—	—	—	—	—	—	—
oleine ..	—	—	—	—	—	—	—	—	—	—	—
50% from herring	0-9997	1-4548	21-86	—	49-03	—	4-49	—	—	—	—
oil ..	1-0200	1-4080	8-81	40-87	47-84	0-64	4-32	—	4-27	—	—
Dark sole leather oil	1-0209	1-4136	16-22	42-38	—	0-86	4-51	—	—	—	—
(chromoline type)	0-9660	1-4696	25-03	—	11-7	—	—	—	—	—	—
	0-9813	—	35-96	—	21-65	—	—	—	—	—	—

layer of carbonate ¼ in. thick. The dish is heated, first over a luminous Argand flame until the oil is completely carbonised, then over a Teclu burner until the contents of the dish are white. The contents of the dish are washed into a large beaker, covered with a clock glass, and excess of hydrochloric acid poured down the beaker spout. The liquid is heated to boiling and precipitated with barium chloride in the usual way.

Free sulphuric acid.—About 10 g. of the sample is dissolved in 50 c.c. of ether and shaken out three times with saturated brine of known sulphuric acid content, using 50 c.c. each time. The united brine extracts are titrated with N/4 alkali, using methyl orange as indicator.

Alkali salts.—The only salts used for washing sulphonated oils are sodium sulphate and chloride.

weighed. The water-soluble matter in the flask may be salted out and the solution shaken out with a mixture of 5 pts. of ether and 1 of alcohol by vol., but it is not easy to remove the salt. The soap solution referred to above is decomposed with acid, shaken out with ether to dissolve the fatty acids, the ether washed with water, distilled, and the residue dried and weighed. These fatty acids will be found to be fairly clean. The process described above, although tedious, requires less than half the time of the inaccurate German process. The fat from all good samples of sulphonated oil should not contain more than 15% of unsulphonated oil.

The results of the examination of commercial sulphonated oils are given in the table above.

My thanks are due to Messrs. A. Hess and Bro., Ltd., in whose laboratory the above work was done.

Communications.

THE USE OF THE REFRACTOMETER IN ASCERTAINING THE PURITY OF CERTAIN REFINED EDIBLE OILS.

BY F. H. THIM, B.Sc.

By suitable refining processes, crude vegetable oils giving an acid reaction can be converted into "neutral oils" suitable for edible purposes: the greater the efficiency of the refining processes, the more nearly do these oils become colourless, tasteless, and odourless. These latter properties constitute the "quality" of the oil in question, and can be tested by suitable organoleptic methods. Though, however, a sample of oil might pass the quality tests, it is also desirable to know its purity, i.e., freedom from admixture.

Since edible oils such as palm kernel, coconut, and arachis, are now prepared in enormous quantities, mixtures of any two or of the three oils are likely to be met with. Admixture may occur inadvertently during expression of the raw material or the refining of the crude oil, but most likely during storage and transport. The presence of small quantities of any two of them in a consignment of the third oil may not impair the oil for its specific purpose, yet for control purposes a simple method of detection combining rapidity and fair accuracy would be advantageous.

With respect to mixtures of the above-mentioned oils, distillation and turbidity tests have their limitations, and are generally too complex for routine work. It is therefore necessary to choose other determinations. The determination of melting point is essential for the examination of an edible fat, and if conducted under standard conditions gives reliable and consistent results. It can give good indications of the amounts of constituents in dual mixtures such as palm kernel and arachis, coconut and arachis, or palm kernel and coconut (see tables 2, 3, and 4).

For the examination of mixtures of all three oils another "constant" is necessary in addition to the melting point. A rapid optical determination commends itself for this purpose, either refractive index or dispersive power. The determination of these by the Pulfrich refractometer yields results of high accuracy in a short time. The refractive index is of greater analytical value than the dispersive power, since it has a greater range from one oil to another. Though the latter "constant" is independent of free fatty acidity and temperature considerations, and the former is intimately dependent upon both, yet this advantage of dispersive power over refractive index is nullified in dealing with refined oils of free fatty acidity 0.04% (as lauric acid), provided the temperature is stabilised, say, at 40° C.

TABLE 1.

Constants of refined edible oils.

Oil.	Standard n _D ⁴⁰	Variation.	m.p., °C.	Free fatty acids (as lauric). %
Arachis ..	1.46235	± 0.00020	-8.0	0.05
Palm kernel ..	1.45097	± 0.00020	28.35	0.04
Coconut ..	1.44897	± 0.00010	24.85	0.03

Standard results.—Since the melting point and refractive index are to be employed in the examination of mixtures, it is necessary to have certain standards obtained for oils of known purity. The standard sample should not be prepared from crude oils already supplied without certainty as to freedom from admixture. It should be prepared from

the original raw material by extraction or expression, and the crude oil so obtained treated by laboratory methods corresponding closely to works' methods. The results in table 1 were obtained from numerous standard specimens.

Coconut oil showed the least variation in refractive index. The refractive indices of refined oils from four different copras are here quoted:—Ceylon, 1.44887; Straits, 1.44902; Singapore, 1.44887; Malabar, 1.44897.

Mixtures of refined oils.—Dual mixtures of pure palm kernel, coconut, and arachis oils were made and the melting point and refractive index determined for each. From graphs obtained by plotting melting point with percentage composition, and refractive index with percentage composition, tables 2, 3, and 4 were compiled.

TABLE 2.

Mixtures of palm kernel and arachis oils.

Palm kernel.	Arachis.	n _D ⁴⁰	m.p., °C.
100	0	1.45097	28.35
90	10	1.45211	27.95
80	20	1.45325	27.30
70	30	1.45439	26.40
60	40	1.45553	25.50
50	50	1.45666	24.35
40	60	1.45780	23.00
30	70	1.45894	20.80
20	80	1.46008	17.25
10	90	1.46122	11.60
0	100	1.46235	-8.00

It will be noted that the refractive index is directly proportional to percentage composition, and that the presence of 10% of arachis oil in palm kernel oil only lowers the melting point by 0.4° C.

TABLE 3.

Mixtures of coconut and arachis oils.

Coconut.	Arachis.	n _D ⁴⁰	m.p., °C.
100	0	1.44897	24.85
90	10	1.45031	24.20
80	20	1.45165	23.45
70	30	1.45299	22.65
60	40	1.45433	21.70
50	50	1.45566	20.30
40	60	1.45700	17.90
30	70	1.45834	14.00
20	80	1.45968	8.00
10	90	1.46102	0.50
0	100	1.46235	-8.00

TABLE 4.

Mixtures of palm kernel and coconut oils.

Palm kernel.	Coconut.	n _D ⁴⁰	m.p., °C.
100	0	1.45097	28.35
90	10	1.45077	27.80
80	20	1.45057	27.30
70	30	1.45037	26.90
60	40	1.45017	26.50
50	50	1.44997	26.20
40	60	1.44977	25.95
30	70	1.44957	25.65
20	80	1.44937	25.40
10	90	1.44917	25.10
0	100	1.44897	24.85

For the three dual mixtures the refractive index shows a linear relationship with percentage composition. The relationship between melting point and percentage composition is not linear in any of the three cases. If, however, refractive index is taken as ordinate and melting point as abscissa a closed graph is obtained (fig. 1). This may be divided into various sections, and any required section used for the estimation of the constituents of either dual or triple mixtures (fig. 2). For

Graph for use with dual or triple mixtures of refined palm kernel, coconut, and arachis oils.

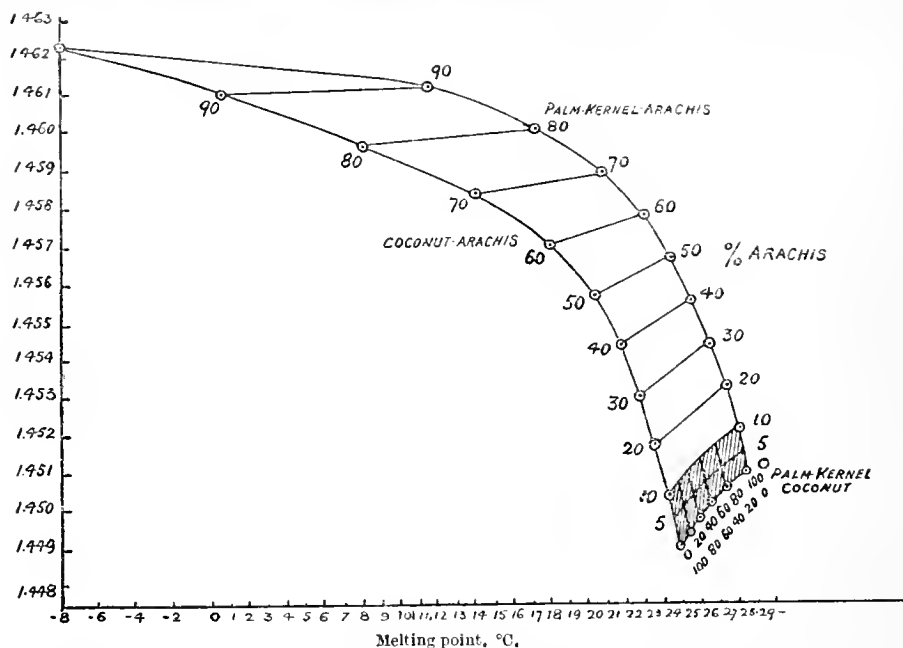


Fig. 1.

example, a given sample is found to have m.p. 27.0°C , $n_D^{40}=1.45150$. These values locate the point A, which at once indicates 8% of arachis oil present. The remaining 92% is then divided proportionately to the coconut-palm kernel percentage, viz., 18:82. Thus the composition of the oil sample is:—palm kernel 75%, coconut 17%, arachis 8%.

Open capillary tubes 0.7 mm. diam. were dipped 1 cm. into the sample, withdrawn and the fat allowed to solidify, keeping the tubes 1 hr. at 0°C . At the expiration of this period they were placed with the lower end 2 cm. under water level, the water being at 15°C , or lower for m.p.s. below 15°C . Electrical heating and stirring then commenced, and the melting point was recorded when the column of melted fat commenced to rise owing to hydrostatic pressure.

Summary.

1. Dual mixtures of coconut, palm kernel, and arachis oils can be accurately determined by the refractive index figure, and also by melting-point determinations.

2. Triple mixtures of the above oils can also be determined using refractive index and melting point figures. Hence the purity of these edible oils can be rapidly ascertained.

THE "SLOPE" OR "TYPE" OF THE RUBBER STRESS-STRAIN CURVE

BY DR. O. DE VRIES.

In a recent paper* Schidrowitz, Goldsbrough, and Hatschek have discussed the nature of the stress-strain curves of vulcanised rubber-sulphur mixtures, and especially the mathematical solution of these curves as belonging to the conchoid family. The figure for "slope" or "type" plays a prominent rôle in these calculations.

Doubtless the slope of the stress-strain curve is an important figure, representing as it does the increase in load necessary to produce a certain elongation. The fact that the stress-strain curve at high elongations (at least for rubber-sulphur mixtures, with which this paper deals exclusively) ends in a straight line, so that the slope of this part of the curve is a constant, gives a special importance to this figure, which represents the resistance to stretching, or the distensibility, at high elongations. The higher the figure for slope of this part of the stress-strain curve, the more easily the

* J., 1919, 3477.

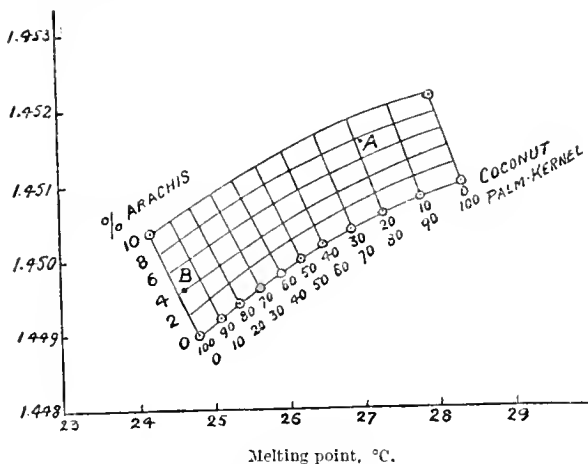


Fig. 2.

Another sample gives m.p. 24.6°C , $n_D^{40}=1.44957$. This locates point B, and would indicate the sample to be coconut oil containing about 4% arachis oil.

In conclusion it must be remarked that the figures quoted throughout refer to the products of a specific process of refining, and allowance must be made at any time for natural variations in the raw material having influence upon the "constants" of the refined edible product. The refractive indices were determined by a Hilger-Pulfrich refractometer, and the melting points by a modified method. The latter determinations were conducted as follows:—

vulcanised product already stretched to ten or more times its original length, yields to a further increase of load, and the weaker it is.

Very characteristically Schidrowitz and his co-workers have called this property "type." It shows markedly the differences between higher and lower grades of rubber, and is a better index of the properties of the lower grades than the tensile strength, the figure for which, in such cases, is often uncertain because of the presence of particles of dirt etc. The different grades of plantation-rubber, in our testing work, gave the following figures for "slope" or "type," determined by the method of Schidrowitz:

	Average.	Normal figures.	Limits.
First quality crêpe ..	35.8	34—38	33—39
Smoked sheet ..	36.7	35—39	33—40
Crêpe from lump ..	37.3	35—38½	33½—40
" " tree-scrap ..	38.8	37—41	35½—44½
" " bark-rubber ..	42.9	40—43	38—47½
" " earth-rubber ..	37.6	36—39	34—40
" " washings ..	39.1	37—41	35½—46

The highest figure was 53*, found in a sample of very inferior crêpe from washings; the lowest figure, 32, is sometimes met with in crêpe from matured rubber.

Especially when figures of 40 or higher are found for slope, the sample may be expected to be inferior, and the higher figures generally indicate a rubber which on keeping is liable to become tacky. Whether in the first grades a difference in slope between, say, 35 and 38 has any practical importance, remains to be seen. Though, theoretically, a rubber with a slope of only 34 to 35 is stronger and therefore preferable, it is not yet clear whether the difference is sufficiently great to affect the manufacture.

The determination of "slope" is easy and necessitates no additional testing, as the stress-strain curve obtained in the determination of tensile strength may serve to read the slope also; and as slope represents a separate property, independent of tensile strength or rate of cure, it certainly deserves more attention than has hitherto been given to it. The more independent properties one takes into consideration, the better a substance with properties so complicated as rubber can be judged.

Moreover, it has been shown on a former occasion† that a close relationship exists between the "slope" and the permanent set, when both are determined for mixtures of 92½ rubber and 7½ sulphur, vulcanised to our standard state of cure (length of 990% at a load of 1.30 kg.; coefficient of vulcanisation appr. 5). The closer nature of this relationship has not yet been worked out, but there appears to be no doubt that this relationship is founded on the intrinsic properties of the vulcanisate, and that there is one factor—be it structure, composition, or some other—which is the cause that a certain rubber stretches easily at high elongations, and that at the same time, after releasing, it shows large deformation.

It is not yet clear which intrinsic property of the rubber is responsible for the slope, and which factors in preparation have an influence on it. From our investigations the following facts are brought out:

(1) The slope becomes greater (the rubber less resistant to stretching) by prolonged and heavy tapping.

(2) The slope decreases by maturation† (decomposition on keeping the still wet coagulum, giving a quick-curing rubber); it also decreases by the use of sulphite and bisulphite in the latex, which

prevent surface-oxidation and discoloration of the coagulum.

(3) The slope increases by coagulation with alcohol*, by the action of lower organisms causing spots on crêpe, and by traces of copper salts, causing tackiness; also by strong heating of the fresh, still wet coagulum.

It would seem that the slope is smaller (the rubber better), the purer the rubber is, and greater, the more decomposition-products are present. The exact nature of these changes is, however, far from clear; addition of foreign substances (such as gypsum or tale) to the rubber-sulphur mixture does not alter the slope, which also remains the same for mixtures with different contents of sulphur.†

The following remarks on the mathematical interpretations of Schidrowitz, Hatschek, and Goldsbrough have no bearing on the above considerations, but tend to give a much deeper meaning to the slope of the upper part of the stress-strain curve.

Before discussing these mathematical interpretations, it may be recalled that Schidrowitz and his co-workers do not use the real slope of the upper part of the stress-strain curve, but an arbitrary figure, being 0.4 (length at 1.04 kg.—length at 0.60 kg.). We have, at first, used the slope of the upper part of the stress-strain curve; when drawn on the scale of the Schopper machine (abscissæ 1 mm.=0.01 kg. per sq. mm.; ordinates 1 mm.=5 units elongation, taking original length at 100) the slope of this part of the curve, in %, is about 30. As will be easily seen, in working on the above scale, the same figure is obtained by taking length at 1.40 kg.—length at 1.20 kg., or in general (as this part of the curve is a straight line) the difference in length for an increase in load of 0.20 kg. per sq. mm. Since the appearance of a paper by Schidrowitz and Goldsbrough‡ we have adopted their method of expression to secure as far as possible uniformity in testing results. We found that the figures for slope, determined in both manners for rubber vulcanised to our standard state of cure, run parallel; the following is the relation between the two:—

TABLE I.

Slope after Schidrowitz ..	34	36	38	40	42
Slope of upper part of curve, %	27.5	29.9	32.2	34.6	37.0

Schidrowitz and his co-workers try to identify the rubber stress-strain curves as conchoidal curves, for which, at a special state of cure, $a=b$. Now it is clear at first sight that this cannot be correct, at least, not without restrictions. The rubber stress-strain curve starts from the origin in the form of an S. This is obvious in all reproductions of curves obtained on Schopper machines (and also in Schidrowitz's figs. 1 and 3, pp. 347 and 349 T, *loc. cit.*). On the Schopper machine, which is especially built for testing at high elongations, the beginning of the curve is often distorted by small irregularities (small shocks in the first stretching of the ring etc.), and is not easy to study in detail. On other machines, such as the Schwartz machine, the S-form of the lower end of the curve stands out much more clearly. This S-form is found for all states of cure; conchoidal curves, however, show it only when $a < b$; for $a=b$ the axis of ordinates (elongations) is a tangent to the conchoidal curve.

This point, showing that Schidrowitz's mathematical solution cannot be theoretically correct for the whole stress-strain curve, may, however, be regarded as of minor importance if the upper part of the curves, where "slope" is determined, and which is the most important in Schidrowitz's considerations, shows a sufficient agreement with the conchoidal curves.

* See O. de Vries, Arch. voor de Rubber Cultuur, 1918, 2, 237, 97 and 557; 1920, 4, 217.

† O. de Vries and H. J. Hellendoorn, J., 1919, 38 T; India Rubber J., 1919, 57, 1165; Arch. voor de Rubber Cultuur, 1918, 2, 783, 791; India Rubber J., 1919, 57, 505.

* J., 1919, 92 T.

† O. de Vries and H. J. Hellendoorn, J., 1917, 1260.

One of the chief points which Schidrowitz and his co-workers bring forward is that the upper ends of the stress-strain curves for different states of cure run parallel, and that the "slope," determined by their method, is a constant, not changing with increasing times of cure.

This is certainly not correct for curves obtained by our method of testing. We have on several occasions* reproduced sets of stress-strain curves which show clearly that the slope of the upper straight part is greater for less cured samples, and becomes smaller and smaller the further the sample is cured. This was regularly found to be the case in our testing of thousands of samples; Table II. gives some figures representing this decrease in slope with increasing time of cure.

The same difference may be noted in the sets of curves published by B. J. Eaton and his co-workers for mixtures of 90 parts of rubber and 10 of sulphur; though no figures are given, and the slope of the curve, in whichever form, is not determined in their testing-work, the reproductions often† show clearly enough that the end of the curve becomes less steep the more the cure is advanced. Even the set of curves reproduced by Schidrowitz‡ would seem to show a similar difference: the curves marked V. and VI. are distinctly steeper than those for further advanced cures.

Whilst therefore the upper, straight ends of the curves do not run parallel for increasing times of cure, we have further to consider whether the slope, determined by Schidrowitz's method, that is, $0.4 \times$ (length at 1.04 kg. ÷ length at 0.60 kg.), remains constant. Schidrowitz and co-workers find this to be the case in their method of testing, and give a number of examples in their Table II. (*loc. cit.*, 350 r). We do not find constancy in our testing; the slope, determined by Schidrowitz's method, increases with increasing times of cure. We have often controlled this fact for the curves obtained in testing large samples (of which slabs of two rings are used as a control in every vulcanisation), and invariably found this increase in slope for increasing times of cure.

The figures in Table II. (following) may be cited as an example. The state of cure is indicated in the first column by the position of the stress-strain curve in our usual manner, i.e., by its length at 1.30 kg. The second column gives the difference in length for 0.20 kg. increase in load, or the slope of the upper part of the stress-strain curve in %, when drawn on the scale of the Schopper machine (*cf. supra*). The third column contains the slope determined by Schidrowitz's method.

TABLE II.

Length at 1.30 kg.	Slope of upper end.	Slope (Schidrowitz).
1070	33.6	34.7
1050	32.2	35.1
1030	30.8	35.4
1010	30.1	35.7
990	29.9	36.0
970	29.7	36.35
950	29.5	36.7

The changes in both properties are clearly illustrated by this table; for the study of the exact nature of the stress-strain curves it is of importance that the slope of the upper part of the curve becomes smaller (the curve flatter) on increasing the time of cure, whilst the slope determined by Schidrowitz's method shows a gradual increase. The two methods therefore do not give results which run parallel for increasing states of cure.

It is not yet apparent what differences in method of curing or testing are the cause that figures in our case show this divergency, whilst in Schidrowitz's testing they remain constant; the mixture is nearly the same (in Schidrowitz's testing

8 sulphur on 100 rubber, in our case $7\frac{1}{2}$ on 92 $\frac{1}{2}$, or 8.1 on 100); the temperature of vulcanisation, the room-temperature during testing, and probably minor details in method of curing and testing differ.

Whilst therefore the mathematical formulæ for the stress-strain curves, evolved by Schidrowitz and his co-workers, are not strictly applicable to our testing results, there is a strong indication that these authors are nevertheless on the right track with their speculations on the deeper nature of the stress-strain curves. They conclude that for each sample there is a correct or optimum state of cure, represented by a conchoidal curve for which $a=b$, and giving an ideal balance of properties, the toughness (tenacity) equalling the limit of extension. This optimum state of cure is calculated to lie lower on the paper, the higher the figure for "slope" or "type." Now we have shown* that the maximum of tensile strength (found when vulcanising for increasing times of cure) is a property which indeed follows this law: it is found lower on the paper, the higher the figure for slope. This proves, in our view, that Schidrowitz and his co-workers, in their mathematical speculations, have arrived at the truth, though their formulæ probably present the case in too simple a form. In a former paper we have given some figures for the position of the maximum of tensile strength for samples with different slopes. As the maximum of tensile strength, in our mixture, is rather flat, it is difficult to determine the exact position of the curve, which gives a maximal tensile strength. Combining our former figures (*loc. cit.*) with later, unpublished results, we estimate the length at 1.30 kg. of the curves giving the maximum tensile strength at 992 and 957 for slopes of 36 and 40 respectively.

Schidrowitz and his co-workers give for the correct cure the following extensions at a load of 1.04 kg. per sq. mm.: 884 and 850 (*loc. cit.*, table I.) This means a length of 984 and 950 times the original; using our figures from table I. (above), and assuming (as is approximately true) that the curve has already reached its straight part at a load of 1.04 kg., we calculate the lengths at 1.30 kg. for Schidrowitz's correct cure as 1023 and 995 for slopes of 36 and 40 respectively. These figures are higher than ours, but the difference in length (distance between the curves) is very similar (38 against 35 units).

Strict comparisons are impossible, as the methods of curing and testing differed; it cannot be said whether Schidrowitz's correct cure and the maximum of tensile strength coincide or not. A relationship between the two is, however, very probable.

Summary.

The slope of the stress-strain curve, that is the increase in load necessary to obtain a certain increase in length, or, in other words, the resistance to stretching, is a property well worth attention in rubber testing. Determined after the formula of Schidrowitz and Goldsbrough it gives, besides tensile strength and rate of cure, an independent property, which is especially typical in judging lower grades, and which, by its direct relationship to permanent set, seems to have a deeper meaning.

The mathematical solution of the stress-strain curves as conchoidal curves, evolved by Schidrowitz, Goldsbrough, and Hatschek, does not strictly hold good for our method of testing; the conclusions of the above authors as to a "correct" cure are, therefore, not generally applicable. Still, the parallelism between this supposed "correct cure" and the actual maximum of tensile strength tends to show that a relationship exists, though the mathematical formulation may be more complicated than that supposed by Schidrowitz and his co-workers.

Buitenzorg, Java.

* O. de Vries and H. J. Hellendoorn, *loc. cit.*

* J., 1919, 91r; India Rubber J., 1916, 52, 717; 1919, 57, 1163; Archief voor de Rubber Cultuur, 1917, 1, 217; 1918, 2, 771.

† See *o.g.*, J., 1916, 715, 1046; and Agric. Bull. F.M.S., No. 27.

‡ J., 1919, 347r, Fig. 1.

Communications.

A NOTE ON THE DETERMINATION OF DIASTATIC ACTIVITY.

BY EDWARD IVAN ROSENBLUM, M.Sc.

IN the course of an investigation into the methods of evaluation of malt substances, which was undertaken by a section of the Analytical Investigations Committee of the Australian Chemical Institute, a number of measurements were made which are of interest as bearing on the question of impurities in commercial starches, and their effect on the amylolytic activity of diastase. The fact that commercial starches usually react acid to phenolphthalein, and alkaline to methyl orange, is attributed by previous investigators to the presence of phosphates, and since these cannot be completely removed by washing with acid as in making soluble starch, and as they do not appear in the ash unless alkali carbonates have been added before ignition, they have been thought to be organic phosphorus compounds.

In their influence on the reaction, or hydron concentration of the starch solutions, however, they may be considered as mixtures of ammonium phosphates, and in most cases behave as if containing both $(\text{NH}_4)_2\text{HPO}_4$ and $\text{NH}_4\text{H}_2\text{PO}_4$. That these impurities, although small in amount, have a very marked influence on the determination of diastatic power is shown by Ford (J., 1901, 414), who found figures varying from 6 to 31 as the diastatic power (Lintner's scale) of starches bought from different dealers, and so liable to be used in the evaluation of malt.

The conversion of potato starch into soluble starch by washing, first with water, then with 0.5% sodium hydroxide, with water again, digesting for seven days with hydrochloric acid of sp. gr. 1.037, and finally washing free from chlorides with distilled water, gives a product which, although considerably improved, still shows, when boiled with water, a slight acidity to phenolphthalein which it appears necessary to attribute to the presence of phosphates. This is shown by the following figures for starches used in the subsequent experiments of this research:—

	Potato starch	Soluble starch
Sugars and dextrin	Nil.	Nil.
Ash	0.33%	0.10%
1 g. requires—		
c.c. N/100 HCl to methyl red	2.2 c.c.	Neutral.
c.c. N/100 NaOH to phenolphthalein	1.5 c.c.	1.0 c.c.

It has been shown by Ford (*loc. cit.*), and more recently by Small (J. Amer. Chem. Soc., 1919, 41, 113), that it is possible to prepare a more highly purified soluble starch, in the one case by repeated precipitation with alcohol, and in the other by digestion with alcoholic hydrochloric acid instead of the aqueous acid.

These methods are not very suitable for the manufacture of commercial soluble starch, or for defining a standard "soluble starch" to be used in conjunction with a standard method, and it would be preferable, if possible, to add a definite quantity of some amphoteric electrolyte which would ensure a constant acidity in the digesting solution. Asparagine has been found to have considerable effect in this direction, and lactic acid has also been suggested, both, of course, operating by increasing the hydron concentration; but the addition of ammonium phosphates would be more practical if proved to be effective. With this end in view the following experiments have been conducted.

The influence of hydrogen ion concentration.

Sherman, Thomas, and Baldwin (J. Amer. Chem. Soc., 1919, 41, 231) have investigated the influence of hydron concentration on the activity of malt diastase. They conclude that the maximum activity in a 30-minute test at 40° C. is obtained when the hydrogen ion concentration is $10^{-4.4}$ to $10^{-4.3}$, or in Sørensen's notation $p_H = 4.4$ to 4.5, a condition which they obtained by the addition of 0.06 mol. of NaH_2PO_4 per litre. A few of their results are as follows:—

Final conc., mols. per litre,					Activity of malt amylase.
H_2PO_4	NaH_2PO_4	Na_2HPO_4	Na_2CO_3	p_H	
0.0167	0.06			2.34	0
0.0057	0.06			2.70	99
0.0007	0.06			4.06	1000
	0.067			4.47	1101
	0.065	0.0007		4.80	1100
	0.033	0.033		6.65	969
		0.02		8.25	210
		0.05	0.0004	8.80	64
		0.05	0.0025	9.33	0

Their measurements of p_H were carried out directly by electro-potential methods, and it is of interest to compare the p_H values of common indicators, as given in Lewis' "Physical Chemistry":—

	p_H .
Methyl orange	4.5—5.5
red	5.7—6.4
Litmus	6.5—7.5
Phenolphthalein	8.2—9.0
Thymolphthalein	10.5—11.5

It should be possible, therefore, to define the p_H value of the solution, approximately at least, by means of indicators.

The first experiments in this investigation were made with a malt extract (A) of the following characteristics: Total solids, 78.0%; sugars calc., as maltose, 65.6%; ash, 2.1%. This was itself amphoteric in reaction and when 5 g. dissolved in 50 c.c. of water was titrated the following results were obtained. They are shown together with the figures for a second sample (B) employed at a later stage in the work.

Reaction of malt extracts.

Reaction to indicator.	Acid or alkali added.	Sample "A."	Sample "B."
With methyl orange to red shade	N/10 HCl	4.06 c.c.	3.75 c.c.
With methyl orange to orange shade ..		3.04 c.c.	—
Neutral to methyl red ..	N/10 NaOH	1.31 c.c.	2.0 c.c.
Neutral to litmus		4.40 c.c.	—
With litmus to a blue shade ..		7.33 c.c.	—
Neutral to phenolphthalein ..		13.30 c.c.	13.0 c.c.

The end-points with phenolphthalein and methyl red were sharp, those with methyl orange and litmus indefinite. The reaction of malt extract, as of starch, is considered to be due to acid phosphates of which the ash is largely constituted and which are believed to exist as ammonium phosphates in the extract itself.

A series of solutions were now made up in which the malt extract as a 0.5% solution was brought to the end-points of the different indicators with N/100 acid or alkali.

Determination of diastatic power.

Twenty c.c. of each solution, containing 0.1 g. of malt extract, was added to a starch solution containing 1 g. of dry starch in 80 c.c. which had been prepared by boiling and then cooling to 46° C., and the flask containing it was immersed in a thermostat at that temperature for exactly 30 mins. At the end of that time the diastatic action was stopped by the addition of a few drops of 20% sodium hydroxide solution, the volume adjusted to exactly 100 c.c., and the solution titrated with Fehling's solution.

This titration is known as T_D and a further titration of a 1% solution of the malt extract alone gives T_M , a measure of the sugar originally contained in the extract.

The formula $9531(1/T_D - 1/10T_M)$ gives the diastatic power, being 100 times the amount of starch converted by one part of malt extract under the above conditions, and this quantity is the diastatic power quoted in the succeeding tables.

The above method is essentially that of Harrison and Gair (Pharm. J., 1906, 94, 6) which is the official method of the Victorian Pure Food Regulations for testing commercial malt extracts.

Diastatic power of malt extract "A."

TABLE I.

Malt solution neutralised with N/100 acid or alkali.

Indicator.	Reaction.	Starch used in digestion	Potato	Soluble.
—	Un-neutralised	200	302	
Methyl orange	Faintly acid	120	150	
—	Neutral	164	207	
Methyl red	—	313	259	
Litmus	—	280	286	
—	Faintly alkaline	246	252	
Phenolphthalein	Neutral	120	185	

The results show a maximum about the region of methyl red, while the difference between the un-neutralised solutions is striking as showing the result of using two different starches in a normal estimation. The reaction of the starch was not corrected in the above series because of the indefiniteness of the endpoints.

However, the following measurements, where the fairly clear end-points alone are included, are not without significance:—

TABLE II.

*Diastatic power of malt extract "A."
Both starch and malt solutions neutralised.*

Indicator.	End-point.	Potato starch.
—	Un-neutralised	200
Methyl orange	Faintly acid	69
Methyl red	Neutral	189
Phenolphthalein	—	140

It should be remembered that the reactions of the un-neutralised solutions to methyl red are faintly acid for the malt extract, neutral for the soluble starch, and alkaline for the potato starch.

TABLE IV.

Diastatic power of diastase. Soluble starch digestion with added phosphates.

No.	Indicator.	Reaction.	Moles per litre.				Diastatic power.
			H ₃ PO ₄ .	NH ₄ H ₂ PO ₄ .	(NH ₄) ₂ HPO ₄ .	Ammonia.	
1.	Methyl orange	Faintly acid	0.0020	0.0174	—	—	0
2.	—	Neutral	0.0010	0.0174	—	—	7330
3.	(Intermediate)	—	—	0.0348	—	—	8630
4.	(Intermediate)	—	—	0.0174	—	—	8630
5.	Methyl red	Neutral	—	0.0051	—	—	8220
6.	(Intermediate)	—	—	0.0051	0.00023	—	7560
7.	Litmus	Neutral	—	—	—	—	75000
8.	—	Intermediate	—	0.0034	0.0015	—	Below 30
9.	—	—	—	0.0017	0.0015	—	0
10.	—	—	—	—	0.0015	—	0
11.	Litmus	Faintly alkaline	—	—	0.0030	—	0
12.	Phenolphthalein	Neutral	—	—	0.0030	0.0014	0

The addition of ammonium phosphates.

With the same malt extract and potato starch, the results shown in Table III. were obtained.

The preceding work suggested the advisability of a further series working with soluble starch and purified diastase, so as to reduce as far as possible the phosphates occurring naturally in the reaction mixture.

TABLE III.

*Addition of ammonium phosphates.
Final conc., mols. per litre.*

(NH ₄) ₂ HPO ₄ .	NH ₄ H ₂ PO ₄ .	H ₃ PO ₄ .	Diastatic power
0.076	—	—	200
0.038	—	—	0
—	0.044	—	172
—	0.0044	—	242
—	0.0088	—	233
—	0.0132	—	236
—	0.0176	—	205
—	0.0220	—	204
—	0.0310	—	188
—	0.0440	—	220 (?)
—	0.0660	—	176
—	0.0880	—	129
—	0.1760	—	0
—	0.0880	0.025	122

The diastase was prepared by Wroblewski's method as follows: 900 g. of air-dried, unkilned barley malt was ground and extracted once with 70% alcohol, the extract being rejected. It was then treated twice with 45% alcohol, and the diastase precipitated from this by adding 95% alcohol until the sp. gr. reached 0.890. The precipitate was washed with 95% alcohol, twice with absolute alcohol, and finally three times with freshly re-distilled ether. After drying in the air 2.5 g. was obtained.

This was not completely soluble in water, but the diastase readily dissolved from it and the preparation showed a Harrison and Gair value of 7450 when tested with soluble starch. The mixture of diastase solution and soluble starch solution was found to be faintly alkaline to methyl red and faintly acid to phenolphthalein. By titration with 1% solutions of H₃PO₄, NH₄H₂PO₄, (NH₄)₂HPO₄, and ammonia the quantities of these reagents necessary to bring the mixture to the different end-points were found, and these quantities were subsequently added when effecting the digestion.

In the results recorded as zero the reduction of 10 c.c. of Fehling's solution on boiling with 100 c.c. of digested starch solution was imperceptible. In the case of No. 8, there was a slight reduction which was judged by a comparative test to represent the diastatic power shown.

The figures show that a slight acidity to methyl red, as attained by the addition of ammonium dihydrogen phosphate, is the optimum reaction for the solution undergoing digestion, the activity falling off sharply when sufficient phosphoric acid is added to make the mixture neutral to methyl orange.

The amount of ammonium dihydrogen phosphate added alone varied in the above series from 0.0051 to 0.0348 mole per litre, or 0.06 to 0.40 g. per 100 c.c.

Further experiments were designed to show what was the maximum amount of phosphate allowable or the concentration of ammonium dihydrogen phosphate at which the diastatic power ceased to rise.

The difference in the "blanks" of the different series is partly due to the fact that different diastase solutions, prepared by filtration after shaking the diastase with water, were used. It is possible that differences in the glass of the flasks used may have been a contributory factor, although flasks of resistance glass were used.

TABLE V.

NH ₄ H ₂ PO ₄ added.		Diastatic power by Harrison and Gair method		10- ² Series III.
%	Mols. per litre.	Series I.	Series II.	
0	0	71	75	71
0.001	0.000087	68	—	—
0.0025	0.00022	65.7	—	—
0.005	0.00044	67.6	—	—
0.0075	0.00066	69.6	—	—
0.01	0.00087	72.8	—	—
0.025	0.0022	—	—	71.6
0.06	0.0052	—	82.2	—
0.10	0.0087	—	—	80.1 (b)
0.20	0.0174	—	86.3	—
0.30	0.0261	—	—	74.5
0.40	0.0348	—	86.3	—
0.80	0.052	—	—	70.0
1.00	0.087	—	—	63.6

An initial fall at very low concentration is followed by a rise to an apparent maximum at about 0.2 to 0.5% NH₄H₂PO₄.

To determine whether this fall was due rather to increase of molecular concentration than to increased hydrogen-ion concentration, a final series was undertaken in which diammonium hydrogen phosphate was also added in a small proportion.

TABLE VI.

NH ₄ H ₂ PO ₄		(NH ₄) ₂ HPO ₄		D.P. x 10- ²
%	Mols. per litre.	%	Mols. per litre.	
0	0	0	0	82.2
0.30	0.026	0	0	82.2
0.30	0.026	0.015	0.00114	54.4
0.50	0.044	0	0	86.6
0.50	0.044	0.005	0.00038	82.2
1.00	0.087	0	0	86.6
1.00	0.087	0.010	0.00076	82.2
1.00	0.087	0.050	0.0038	75.6

In all the above experiments, the titrations are correct to within 0.2 c.c. of sugar solution to 10 c.c. of Fehling's solution, representing an error of about 150 in the diastatic power.

Although the results in this table are not entirely in accord with those of the last, it appears that in general the diastatic activity increases with the addition of ammonium dihydrogen phosphate up to the region of 0.5% of the salt, and it is possible that, as suggested by previous investigators, the fall at higher concentrations occurs when a certain molecular concentration is exceeded.

It seemed reasonable, therefore, to test the addition of 0.5% of acid ammonium phosphate in the estimation of the diastatic power of some routine malt extracts.

TABLE VII.

Diastatic power of malt extracts with added acid ammonium phosphate.

No.	Potato starch.		Soluble starch.	
	No addition.	0.5% added.	No addition.	0.5% added
A.	200	—	302	—
B.	215	347	412	306
C.	518	871	611	909
D.	500	774	595	774
E.	—	—	463	614
F.	—	—	382	435

Hence the addition of phosphate, as well as raising the diastatic power by establishing a favourable and constant hydrogen-ion concentration, goes far to equalise the divergent results obtained with soluble and potato starches.

With malt extract "B" and soluble starch, the addition of phosphate causes a fall in D.P., an anomalous result which recurred in a series with different starches, also digested with sample "B."

TABLE VIII.

Diastatic power of malt extract "B" with and without 0.5% NH₄H₂PO₄.

Starch.		Without phosphate.		With 0.5%	
Soluble
Potato I.	..	215	..	347	..
" II.	..	411	..	335	..
Arrowroot	..	575	..	341	..
Rice	..	511	..	331	..

Summary.

1. The difference in the hydrogen-ion concentration of starches due to the acid salts they contain renders the estimation of diastatic activity by the digestion of starch uncertain.

2. When estimations are conducted by the Harrison and Gair method the diastatic power is at a maximum when the digesting solution has a faintly acid reaction to methyl red but is alkaline to methyl orange.

3. This condition may be secured by the addition of ammonium dihydrogen phosphate, and if 0.5% of the salt is added a constant acidity and, in most cases, an optimum activity are assured, and the difference due to the divergent reactions of different starches is neutralised.

The thanks of the author are due to Professor Masson, Dr. Heber-Green, and the members of the Malt Analysis Section for helpful criticism, and to C. S. Callaghan, who carried out most of the determinations of diastatic power.

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THE PREPARATION OF THIOPHOSGENE FROM THIOCARBONYL TETRACHLORIDE.

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When perchloromethyl mercaptan (thiocarbonyl tetrachloride) CCl₄.SCl undergoes reduction two chlorine atoms are removed and thiophosgene is obtained. The literature points to the use of tin and hydrochloric acid as employed by Klason (Ber., 1887, 20, 2380) as being the most promising method, although Kern and Sandoz (F.P. 5.430 of 1887; Monit. Scient., 1887, 4, 1328; and Friedländer's "Fortschritte der Teerfarben Fabrikation," 1877—1887, p. 96) employed either stannous chloride and hydrochloric acid, iron filings and acetic acid, or any mixture which produces hydrogen. These communications contain no information as to the yields to be expected.

By the action of silver dust on thiocarbonyl tetrachloride Rathke (Annalen, 1873, 167, 201) obtained thiophosgene in small amounts, as also James (Trans. Chem. Soc., 1887, 51, 268), by the action of chlorine on methyl thiocyanate. By heating carbon bisulphide and phosphorus pentachloride in sealed tubes at 100° thiophosgene and thiophosphoryl chloride are produced.

The formation of thiophosgene during the chlorination of carbon bisulphide has been noticed by Rathke (*loc. cit.*) and by three of the present authors (J., 1920, 257 T), but owing to the small quantity produced and the simultaneous presence of carbon tetrachloride, this method is entirely unsuited to its preparation even on a small scale.

The present paper contains a summary of the results we have obtained by the use of tin and hydrochloric acid as reducing agent. This method appears to be preferable to any other that we have investigated. The yield of thiophosgene usually obtained is 55—60%, while varying amounts (20—35%) of thiocarbonyl tetrachloride may be recovered.

Our method of experiment was as follows:—All the low-boiling impurities in the crude thiocarbonyl tetrachloride (J., 1920, 257 τ) were removed by fractionation with a 12-bulb column, and the residue boiling above 140° was used for the preparation of thiophosgene without further purification.

Granulated tin and hydrochloric acid in the desired proportions were heated in a large flask (not more than half-filled) until hydrogen was freely evolved. On addition of a little CSCl_4 from a dropping funnel a lively reaction ensued, thiophosgene distilling and being condensed in a series of well-cooled receivers. Very little CSCl_4 should be added until thiophosgene begins to distil over, otherwise the reaction may become too vigorous and efficient condensation impossible. When all the CSCl_4 had been added, the mixture was heated till distillation of thiophosgene ceased. After separation from the water, the thiophosgene was dried over calcium chloride and distilled, the fraction boiling below 110° being collected. After a second fractionation this yields practically pure thiophosgene. The residue boiling above 110° consists mainly of CSCl_4 , and may be used again.

The rate of addition of CSCl_4 to the mixture of tin and hydrochloric acid exerts some influence on the yield. Rapid addition (involving rapid formation of thiophosgene) gives rise to almost the same yield of thiophosgene as when the CSCl_4 is added slowly; a greater total yield of thiophosgene and CSCl_4 is, however, obtained. In experiments with 550 g. of CSCl_4 , 350 g. of tin and 1000 c.c. of concentrated hydrochloric acid, yields of 58, 61.5, and 57% of CSCl_4 were obtained when the time of addition of CSCl_4 was $1\frac{1}{2}$ hrs., 1 hr., and 20 mins., respectively; the amounts of CSCl_4 recovered were 8, 8, and 31%, respectively. With 550 g. of CSCl_4 , 175 g. of tin, and 750 c.c. of acid, the respective yields for the above times of addition were 46, 55, and 54%, with recoveries of 36, 25, and 33% of unchanged CSCl_4 . The CSCl_4 should, therefore, be added as rapidly as is compatible with efficient condensation of the distillate, although the rate of addition seems to have less influence when the proportion of tin to CSCl_4 is smaller.

Variation of the proportion of tin to CSCl_4 .—Using one atomic proportion of tin to one mol. of CSCl_4 and more hydrochloric acid than is required to dissolve the tin, yields of 54–60% of thiophosgene and 18–29% unchanged CSCl_4 were obtained. On the other hand, with half an atomic proportion of tin, one mol. of CSCl_4 ,* and excess of hydrochloric acid, the yield of thiophosgene was 50–53% and unchanged CSCl_4 33–37%. The total percentage yield (i.e., CSCl_2 plus CSCl_4) is, within certain limits, almost independent of any alteration in the ratio $\text{Sn}:\text{CSCl}_4$. In cases where this ratio is 0.5:1, however, it appears to be clearly established that (provided the rate of addition of the CSCl_4 and the other factors of the experiment remain unchanged) the amount of CSCl_2 obtained is slightly, but distinctly smaller than with higher ratios, a corresponding increase in the recovered CSCl_4 being noticed. By employing this smaller ratio, it is obvious, however, that a very great economy in the use of a rather expensive metal can be effected. Alteration of the ratio to 0.75:1 offers no special advantage, the yields of CSCl_2 and CSCl_4 being 56–58% and 30–31% respectively; the same would appear to be the case when 1.5 atomic proportions of tin are employed, the yields in one experiment being 59% and 20%, respectively.

* The proportion of $\frac{1}{2}$ atom tin : 1 mol. CSCl_4 is based on the assumption that both the nascent hydrogen and stannous chloride produced by action of hydrochloric acid on tin, are equally effective in carrying out the reduction; the proportion 1 atom tin : 1 mol. CSCl_4 assumes that only the stannous chloride or the hydrogen is effective in this way.

Variation in the concentration of the hydrochloric acid used.—In all the experiments in this series, 550 g. of CSCl_4 (1 mol.) and 175 g. of tin ($\frac{1}{2}$ atom) were employed. It was found that if ordinary commercial acid (28%) diluted with an equal volume of water is used for the reduction a yield of 53% of thiophosgene and of 38% of unchanged CSCl_4 may be obtained, if acid equal to 148% of the amount theoretically required to dissolve the tin (*viz.*, 500 c.c.) be employed. Using this amount of acid we find that dilution with $1\frac{1}{2}$ volumes of water reduces the yield of thiophosgene to 41%, with 45% of CSCl_4 recovered. Using no acid at all, but only tin and water, the yield is 25% of thiophosgene and 68% of unchanged CSCl_4 .

It appears to be of no advantage to use more than 500 c.c. of acid, the yield with 750 c.c. being only 52%. On the contrary, the reduction may be effected with less acid than is required to dissolve the tin; *e.g.*, when 250 c.c. of acid was employed a yield of 51% of CSCl_2 with 42% of unchanged CSCl_4 was obtained, whilst with only 100 c.c. (30%) the yields were 33% and 61%, respectively, but in this case the mixture became solid before all the tin had dissolved; under these circumstances increased yields were obtained by steam injection (*cf. infra*).

Use of steam for heating the reduction mixture.—Instead of heating the reaction mixture directly, steam may be introduced for this purpose with good results. Thus, with half an atom of tin to one molecule of CSCl_4 and only 30% of the amount of commercial hydrochloric acid theoretically required to dissolve the tin, a yield of 50% of thiophosgene with 39% of unchanged CSCl_4 has been obtained, solidification of the mixture being prevented.

Use of stannous chloride and hydrochloric acid as reducing agent.—A mixture of 200 g. of CSCl_4 , 270 g. of crystallised stannous chloride, 100 g. of 28% hydrochloric acid, and 70 g. of water was stirred continuously for 12 hrs. at 30° – 35° C. Yields of 56 and 58% of thiophosgene were obtained, but only 5 and 5.5% of CSCl_4 was recovered unchanged.

Use of iron and acetic acid as reducing agent (Patent specification of Kern and Sandoz).—Iron filings were slowly added to a well stirred mixture of CSCl_4 and dilute acetic acid at 35° – 40° . Yields of thiophosgene varying from 44 to 51% were obtained, but no CSCl_4 was recovered. The thiophosgene thus obtained may contain some carbon tetrachloride. We have not specially examined the product in this case. In the last two methods, the use of hot concentrated hydrochloric acid is avoided, but the yields are lower than those obtained by the use of tin and hydrochloric acid.

Use of hydrogen sulphide as a reducing agent.—By prolonged passage of hydrogen sulphide over CSCl_4 mixed with 1% of finely divided charcoal and a quantity of dried pumice at 100° – 105° , a yield of 51% of fairly pure thiophosgene and 38% of CSCl_4 was obtained; some sulphur chloride was simultaneously produced. The thiophosgene in this case contained a little carbon tetrachloride.

The action of metallic copper on CSCl_4 gives rise to considerable quantities of thiophosgene, although Rathke states that silver powder is more satisfactory. It would appear from our preliminary experiments that the best yields are obtained when one atomic proportion of copper and one mol. of CSCl_4 are employed, yields of 49, 65, and 71% of thiophosgene being obtained with 2, 1.5, and 1 atomic proportions of copper bronze (commercial finely-divided metallic copper) respectively. Another experiment with larger quantities and one atom of copper bronze gave a yield of only 49%, however, and the reaction proceeded much more slowly, giving rise to a by-product, probably hexachloromethyl disulphide, $\text{CCl}_3\text{S}_2\text{CCl}_3$.

In most of the experiments the copper bronze was gradually added to the thiocarbonyl tetra-

chloride contained in a flask fitted with a reflux condenser. The reaction occurred without the application of heat, but the mixture was heated on the water-bath for about an hour after all the copper bronze had been added. Water was finally added, when the temperature rose and thiophosgene distilled. When CSCl_4 , water, and copper bronze are mixed in the cold a mass of yellow crystals is obtained. On heating, these disappear, and thiophosgene begins to distil. Similar crystals are formed when water is added to a mixture of CSCl_4 and copper dust which has been heated.

When CSCl_4 is treated with zinc dust under various conditions small amounts of thiophosgene are produced. In one experiment a yield of about 20% was obtained, but all other attempts were much less satisfactory. Rathko (*ibid.*, 201) obtained very little thiophosgene by the use of zinc at 160°C .

Fractionation of crude thiophosgene.—About 2320 g. of crude thiophosgene (b.p. below 110°), obtained by the use of tin and hydrochloric acid, was submitted to several fractionations with the following result:—(1) Below 72°C , 34 g.; (2) 72° – 76° , 2063 g.; (3) 76° – 110° , 81 g.; (4) above 110° , 128 g. (chiefly CSCl_4). Fraction (2) had sp. gr. 1.509 at 15° . Billetter and Strohl (Ber., 1888, 21, 102) give the sp. gr. of thiophosgene obtained from CSCl_4 , stannous chloride, and hydrochloric acid as 1.5085 at 15° . Commercial thiophosgene obtained from the firm of Kern and Sandoz (Basle) is stated to contain some carbon tetrachloride (Bergreen, Ber., 1888, 21, 337).

The authors desire to express their thanks to the War Office for sanctioning the publication of this paper.

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CALCULATIONS INVOLVED IN THE PREPARATION OF ACID MIXTURES USED IN THE MANUFACTURE OF NITROCOTTON.

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In the process of preparing the nitration mixtures, spent or refuse acid from the manufacture of nitrocotton receives a preliminary adjustment by the addition of fuming nitric acid and oleum. This treatment corrects the composition of the refuse acid, bringing it somewhere near the composition specified for the nitration process. The corrected mixture, or "bulked acid," as it is called, may prove to fall within the limits allowed in the specification laid down for the nitrating mixture, but it may not do so and will therefore require further correction, which is effected by addition of nitric acid, oleum, or refuse acid, or usually of two of these acids, in calculated proportions. The use of water is avoided, except under special circumstances.

The following algebraic method has been used for the calculation of the proportional parts at a large explosives factory with satisfactory results. After trial it has proved much less laborious than the arithmetical method and more adaptable to changing conditions than the graphical method, and with certain justifiable assumptions it can be used for constructing tables from which the proportional parts may be written down.

The method is most easily demonstrated by the working of examples, and, for the sake of convenience, the abbreviations N/A, S/A, R/A, M/A and B/A will be used respectively for nitric acid, oleum, refuse acid, specified mixed acid, and the "bulked acid" referred to above.

Example 1: A batch of bulked acid is found to require correction by means of 90% nitric acid and

20% oleum to bring it to a certain specified composition. Let the required proportions be 100 parts of bulked acid, x parts of oleum and y parts of nitric acid, on mixing which $100+x+y$ parts of the specified mixed acid will be obtained. Two independent equations are available for finding the values of x and y . The example is worked out as follows:—

	Acid.	B/A.	S/A.	N/A.	M/A.
Analysis	(% HNO_3)	22.0	0.0	90.0	23.0
	(% H_2SO_4)	66.0	104.5	0.0	67.0
	(% H_2O)	12.0	-4.5	10.0	10.0
		100.0	100.0	100.0	100.0

The equation connecting HNO_3 contents is:—

$$100 \times \frac{22.0}{100} + x \times \frac{0.0}{100} + y \times \frac{90.0}{100} = \frac{23(100+x+y)}{100}$$

Rewritten, this equation becomes $-23x + 67y = 100$
Similarly, the equation from H_2O contents becomes $14.5x = 200$

Solving these equations, $x=13.793$ and $y=6.228$. The required proportions are therefore 100 parts of bulked acid, 13.793 parts of oleum, and 6.228 parts of nitric acid.

Example 2: A general case in which the correcting acids are refuse acid of known composition and 92% nitric acid. Proceeding as before, we have the following:—

	Acid.	B/A.	R/A.	N/A.	M/A.
Analysis	(% HNO_3)	a	19.0	92.0	23.0
	(% H_2SO_4)	b	68.0	0.0	67.0
	(% H_2O)	c	13.0	8.0	10.0
		100	100.0	100.0	100.0

The HNO_3 and the H_2O contents furnish two equations, viz.:—

$$4x - 69y = 100(a - 23) \dots (1), \text{ connecting } \text{HNO}_3 \text{ contents,}$$

$$\text{and } 3x - 2y = 100(10 - c) \dots (2), \text{ connecting } \text{H}_2\text{O} \text{ contents.}$$

Solving equations (1) and (2), we find

$$x = 34.673(10 - c) - 1.005(a - 23),$$

$$\text{and } y = 2.010(10 - c) - 1.508(a - 23).$$

These general formulæ for x and y may now be used for drawing up tables, on the assumption that the compositions of the correcting acids and the specified mixed acid are constant. This assumption is dealt with later.

Construction of tables:

In practice the terms $(10 - c)$ and $(a - 23)$ are usually of small magnitude, and a working table from which the proportional parts of correcting acids can practically be read off is easily constructed.

The following skeleton table is drawn up for the conditions given in Example 2:—

TABLE 1.

Parts of correcting acids per 100 parts of bulked acid.

$$\text{Parts refuse acid} = 34.673(10 - c) - 1.005(a - 23).$$

$$\text{Parts nitric acid} = 2.010(10 - c) - 1.508(a - 23).$$

$(10 - c)$ or $(a - 23) =$	0.01	0.02	0.03	0.1	0.2	0.3	1.0	2.0
$34.673(10 - c) =$	0.35	0.69	1.01	3.47	6.01	10.40	34.67	69.35
$-1.005(a - 23) =$	-0.01	-0.02	-0.03	-0.10	-0.20	-0.30	-1.01	-2.01
$2.010(10 - c) =$	0.02	0.04	0.06	0.20	0.40	0.60	2.01	4.02
$-1.508(a - 23) =$	-0.02	-0.03	-0.05	-0.15	-0.30	-0.45	-1.51	-3.01

In Example 2, if $a=22.65$ and $c=9.85$, then $(a-23)=-0.35$ and $(10-c)=0.15$. Using the formulae or the completed table it will be found that 5.55 parts of refuse acid and 0.83 part of nitric acid are required to correct 100 parts of bulked acid.

Similar tables can be constructed for correction (1) with oleum and nitric acid, and (2) with oleum and refuse acid.

If in using any table or formula negative results are obtained, correction is impossible with the acids represented in the table, and must be carried out by some other combination of acids. For instance, in Example 2, if $a=22.65$ and $c=10.55$, oleum and nitric acid are required for correction, and Table 1 would give a negative result.

The use of tables is possible on the assumption that the refuse acid, oleum, and nitric acid are constant as regards composition. In practice this is never the case, but variations within $\pm 2\%$ of the average compositions have little effect, as is shown in Table 2.

TABLE 2.

1000 lb. of nitric acid added to 100,000 lb. of mixed acid. Calculated effect of a 2% variation in the strength of the nitric acid on the composition of the resulting mixture.

Composition of the original mixed acid.	Composition after addition of		Variations in resulting composition.
	1000 lb. of 90% N/A.	1000 lb. of 92% N/A.	
22.0% HNO_3 68.0 % H_2SO_4 10.0 % H_2O	22.673 % HNO_3 67.327 % H_2SO_4 10.000 % H_2O	22.693% HNO_3 67.327% H_2SO_4 9.980% H_2O	+0.020% HNO_3 +0.000% H_2SO_4 -0.020 % H_2O
100.0	100.000	100.000	0.000

The limitations of the method will be obvious to chemists employed in acid-control laboratories.

The author desires to express his thanks to the Research Committee of Messrs. Nobel's Explosives Co. for their kind permission to publish this method, and especially to Mr. W. Rintoul and Capt. J. M. Weir for the interest they took in its development and application.

LABORATORY APPARATUS FOR PREPARING ELECTROLYTIC HYDROGEN.

BY WILLIAM GEORGE PALMER.

The apparatus consists of an unglazed earthenware pot, 3 in. \times 7 in., in the closed end of which three holes are drilled, one at the centre of about $\frac{1}{2}$ in. bore, and two at the outer edges each of about $\frac{1}{4}$ in. bore. For $2\frac{1}{2}$ in. from the open end of the pot vertical rows of small holes (about $\frac{3}{16}$ in. diameter) are also drilled. The material of the pot is made gas-tight by soaking in melted paraffin wax. A large sheet of parchment paper (which is to form the real diaphragm of the cell) is then closely wrapped round the pot so as to cover the vertical rows of holes, and held in position by rubber bands.

The electrodes are formed of stout sheet nickel beaten into cylindrical form. The cathode is supported with its lower edge well inside the pot, by leads passing through glass tubes cemented with sealing wax into the $\frac{1}{4}$ -in. holes in the base of the pot.

The centre hole of the pot having been provided with a rubber bung carrying a bulb delivery tube for the hydrogen, the pot (containing the cathode inside) is immersed with the open end downwards in about 300 c.c. of a 5–10% aqueous solution of caustic potash contained in a beaker of one litre capacity.

The pot is supported by a glass triangle standing on the bottom of the beaker. The cylindrical anode is now fitted into the beaker outside the pot so that its lower edge is about $\frac{1}{2}$ in. above the lower edge of the pot. A distance of about $\frac{1}{4}$ in. is left between the parchment paper and the anode.

It is important to steep the anode in concentrated nitric acid for $\frac{1}{2}$ hr. before making up the cell. If this is not done the anode readily becomes attacked when a large current is used. The cell is cooled by immersion in a large outer vessel of cold water. The strength of the electrolyte should not be less than 5%, and it is preferable to work at temperatures between 25° and 30° C.

A current of 8–10 ampères may be used without inconvenience, the anode remaining untarnished. The electrolyte gradually absorbs carbon dioxide from the air, but this does not interfere with the working of the cell. It is, however, advisable to renew the electrolyte once each month.

University Chemical Laboratory,
Cambridge.

London Section.

Meeting held at Burlington House on December 6, 1920.

DR. CHARLES A. KEASE IN THE CHAIR.

THE DYESTUFF INDUSTRY.

BY HERBERT LEVINSTEIN.

The importation of dyes into this country was in fact controlled by Proclamation from February 25, 1919, until December 17, 1919, when Mr. Justice Sankey decided that the Order was invalid. On the very next day the President of the Board of Trade, Sir Auckland Geddes, stated in the House of Commons that legislation to secure the necessary powers to re-impose restrictions would be pressed forward in the next session.

It might therefore reasonably have been anticipated that this Bill would have been passed into law many months ago. In that case we should have been spared the disorganisation of the aniline dye industry which was caused by the large importation of German dyes during August, September, and October of this year, the necessity of throwing out of employment large numbers of workpeople engaged both in manufacturing and in new construction, and the feeling of uncertainty engendered in the minds of producers and consumers alike by the controversy which has recently arisen with regard to the Government policy.

The Government policy was announced in the House of Commons by Sir Albert Stanley as far back as May 15th, 1918, in these words, viz., "that the importation of all foreign dyestuffs shall be controlled by a system of licences for not less than ten years after the war, in order to safeguard this industry against the great efforts which the great German firms are certain to make after the war to destroy all we have accomplished during the war and to make this industry again subservient to Germany."

As Sir Albert Stanley was careful to state, the proposal was made on the advice of a Cabinet Committee of Mr. Asquith's Government and therefore had the approval of Mr. Asquith and his Government. It is now practically four years since Mr. Asquith's Government went out of office. Since then the proposals have been approved and the pledge given to the industry has been renewed by Mr. Lloyd George and his two Governments with the full knowledge and silent acquiescence of this Parliament and its predecessor.

It will be seen therefore that this is not a casual pledge thrown idly across the floor of the House by a Minister in a rash moment. There is no question of stampeding the House of Commons; but, on the contrary, great and regrettable delay has occurred in carrying out a policy which was agreed upon long ago by statesmen of very different political views.

The debate on the second reading of the Government Bill for controlling the importation of dyes, which takes place to-morrow, is therefore one of great importance, for if pledges solemnly made, and on the faith of which a great industry has been built up and millions of pounds spent, are not ratified by Parliament or become a matter for acute controversy, with the issue doubtful, nobody in the chemical industry will have the least confidence in Parliament.

Consider that it takes five years at a university for a young man to become a research chemist. He is then, or should be, a good organic chemist, but he is not a dyestuff chemist. It will be a further

two years before he is valuable as a dyestuff chemist for the purpose of research, and as a rule longer than that before he is useful on the manufacturing side. Our university chemical laboratories are filled with students at the present moment, a large number of whom have been attracted to this science by recognition at long last of the extraordinary importance of the dye industry and by the promise of the Government support. Years must elapse before the training of those who went to the universities after the Armistice is complete.

Those chemists who have been asked to do something which is vital to the State are entitled to say to Parliament and the public, "Having made up your mind that you want this industry, be consistent and do all that may be necessary to see it through. You want to attract our young men, the cream of our profession, and you can only do this by giving them an assured career."

I do not propose to deal with the merits of the Bill. I am solely concerned with the preservation of the industry. I am indifferent as to the means selected so long as they are adequate, but this I will say:—No alternative scheme likely to command support has been put forward. The Bill has the great merit in my eyes that it has the support of the great majority of dye users. That is very important. Further, it must be remembered that alternative schemes had been carefully considered by two Governments previous to this one, by four Presidents of the Board of Trade, and that the present Bill embodies the unanimous conclusion of many different men of great political sagacity and experience representing every grade of political thought of the House of Commons.

There is nothing in the assistance asked by the dye industry which brings it even remotely within the realms of party politics or of the acute political economic controversies of pre-war days; at any rate, it is supported by Mr. Asquith and Mr. Runciman, as well as by their successors and by such authorities as John Stuart Mill and Adam Smith. The former (*Political Economy*, Book IV, Chapter 10) says that the only case in which on mere principles of political economy protective duties can be defensible is when they are imposed temporarily in the hope of neutralising a foreign industry in itself perfectly suitable to the circumstances of the country. "Defence," says Adam Smith, "is of much more importance than opulence."

When I last addressed a meeting of the Society on the dye industry (in July, 1919; cf. J., 1919, 246 *r*) I gave four reasons why a large dye industry was vital to this country:—(1) It is a key industry. (2) It gives national security as a guarantee of peace. (3) The development of new industries is encouraged by organic research. (4) It is of political importance.

Key Industries.

As Lord Moulton said on December 8, 1914:—"We have had peace in England, and never thought of danger, industrial or national. Even though there may not be another war, there is perpetual war going on industrially. It is based on what, in the industrial world, corresponds to war in the political world."

In this industrial war, to use Lord Moulton's phrase, the struggle is for the control of the key industries, just as in political war, the struggle is for key positions, by the mastery of which dominance is secured over large tracts of enemy territory.

This point of view has been extremely well put by Mr. Vernon Clay, the Chairman of the Colour Users' Association, and is very clearly enunciated in the first instance by Mr. Runciman in a speech in the House of Commons which has been frequently quoted.

The German Interessen Gemeinschaft (I.G.) is making desperate attempts to recover the monopoly, because having once re-established it, they control our textile trade. They can refuse supplies, they can quote high prices to other countries, or they can use the advantage that they hold for the purpose of political bargaining. This is not a fanciful statement. I will give two instances:—

(1) Shortly after the armistice, the supply of dyes in Alsace became a question of difficulty to the French, and this difficulty was used by the Germans greatly to their political advantage. The great war development of our industry in England saved us during the Peace Conference from similar embarrassment. The Germans were unable in our case to use their predominance in the supply of dyes for bargaining purposes.

(2) The first practical method for the manufacture of Alizarin was patented almost simultaneously in Germany and in England in June of 1869. The English patentee was Perkin, the German patents were acquired by the Badische Co. Both patentees exchanged licences, so that Perkin obtained a monopoly for the manufacture of Alizarin in this country. In 1874 Perkin retired from business. His works were sold to Brook, Simpson, and Spiller, who in turn sold out later to Burt, Boulton, and Heywood. Perkin actually manufactured a ton of Alizarin Red in 1869, and in the following year he produced 40 tons, while the Germans only commenced manufacturing in 1871. In that year they produced 150 tons, while Perkin manufactured 220 tons. Two years later, viz., in 1873, the Germans already manufactured $2\frac{1}{2}$ times as much as Perkin, and a few years later they completely dominated the supply. This was due to the commercial enterprise of those directing the German factories, not to their superiority in science or large-scale manufacture. The German manufacturers themselves travelled, establishing agencies all over the world, while the British rested content with issuing circulars warning their customers against using inferior foreign-made goods. In this way by 1881 the Germans had secured so great a hold on the market that they combined together and formed the first Alizarine Convention with a view to raising prices. There was no Patent Law in Germany in those days. That is why there was more than one manufacturer in Germany. As a result, the Germans cleared £1,000,000 on this dye alone in one year. The next year, 1882, the year before the expiration of the patent, the Germans issued a famous circular in which they threatened to stop supplies to British consumers unless they agreed to extend their contracts at a fabulous price for 12 months after the expiration of the patent in 1883. The Scottish Turkey Red dyers replied to this coercive circular by going on to short time in order to conserve their stocks. They then formed a new company, a co-operative company to buy Messrs. Burt, Boulton, and Heywood's works. As a result, the first Alizarine Convention came to an end in 1885. Prices, which had been dropping ever since the formation of the British Alizarine Company, were then reduced to an unremunerative level. The extraordinary profits made by the German dye industry through the Alizarin monopoly enabled them to write off the cost of their works, to reconstruct them with huge laboratories, and to man them with skilled research chemists. At that time about one-third of the entire consumption of Alizarin was used in this country, so that these profits were made to a great extent out of the British consumer. In 1886, in an address to this Society, my father spoke as follows:—

"It is the development of the Alizarin industry in this country that our printers and dyers have to thank for a cheap and unlimited supply of Alizarin. It was the development of this new industry which the combination attempted to crush

in the germ, and this fact accounts for the present unremunerative price; and this endeavour on the part of some powerful continental colour-manufacturing companies to crush British competition by hook or by crook is not the only instance of such action. Now some may say that with all this reduction in the price of the article, our dyers and printers are not one iota better off to-day than they were when it was much higher. I would just invite such persons to consider what their position would have been if the combination had not been broken up. Turkey Red dyers, printers, and merchants, each and all of them, would have been at its mercy; and if it had so pleased the German combination, it might even have stopped our export trade in Turkey Red or Alizarin dyed goods by exacting prohibitive prices from our consumers, and thus diverted this important branch of the dyeing and printing trade into the hands of German manufacturers and merchants."

The expression "key industry" had not been invented, but already so early as 1881, it was clear that the manufacture of Alizarin Red was not one which could be safely left in the hands of a German trust. The manufacturers of dyes are now combined together in the I.G., which controls the manufacture not only of this important dye, but of every other dye. I need say no more on this point. Past history and past experience are surely sound guides to policy.

The formation of the British Alizarine Company saved the Turkey Red dyers. What was the result? The Germans first did all they could to put this company out of business, and finally in 1900 they made a Convention which included the British Alizarine Company. Under this Convention the British Alizarine Company was allowed to manufacture a certain quantity of Alizarin Red, and to sell it at a fixed price. This quantity was not a large quantity, but the sale was guaranteed. It was quite inadequate to supply the needs of this country and of the Empire, and when war broke out there was in consequence an acute shortage of Alizarin. The agreement entirely restricted development. The company just made the proportion of Alizarin which they were permitted to by the Germans, and sold it at the price fixed by the Germans. I say this because the position of the British Alizarine Company has been cited as an example of how aniline dyes can be made in this country in defiance of the Germans, and the business developed successfully in the face of competition. There were people who did develop their business in this country without making arrangements with the Germans, but their growth, like that of the British Alizarine Company, was restricted by the overwhelming competition which they met. The country is under a great debt to those energetic and able consumers who founded the British Alizarine Company when threatened by the German Alizarine Convention, and their action was, as far as I know, the first one taken by consumers to render themselves, if only in one dyestuff, not entirely dependent upon the German Convention.

National Security as a Guarantee to Peace.

The importance of the dye industry for national defence is, I think, now generally admitted. I see from an answer given by Mr. Churchill to Mr. Grattan Doyle on November 25 that the reports of the Commission which went to Germany early in 1919, and of Brigadier H. Hartley, may possibly be published in the near future. Of course, as Mr. Churchill admitted, the War Office is well aware of the fundamental part played by the German dyestuffs in keeping the German army in the field, but I will repeat that every toxic substance put in the field by the Germans during the war, with an insignificant exception, was made by the German Dyestuffs Trust, the I.G.. These substances were made by

methods devised by the I.G. in plant suitable for the manufacture of aniline dyes and of intermediate products. They contributed to the chemical warfare organisation of Germany:—

(1) Great, flexible plants suitable for making almost any organic substance on a large scale at a short notice.

(2) A highly-skilled personnel, capable of successfully conducting the large scale manufacture of complicated organic substances.

(3) A large research staff, experienced in making laboratory processes suitable for technical scale work.

It is said by some that the next war may not be a chemical war, but a bacteriological or a physical war. If we cannot produce a counter, the next war will certainly be a chemical war, and the results will be extraordinarily quick and decisive. Why was the last war in its later stages a chemical war? After the battle of the Marne the Germans selected chemistry as their most likely means of effecting a surprise because they were the sole belligerent possessing a large dyestuffs industry. Chemical warfare only failed to bring them a decision partly because they did not believe in their own weapon, and therefore were not prepared to follow up the results obtained, *e.g.*, the use of chlorine at the second battle of Ypres, and partly because they used their products on too narrow a front to secure a strategic result from the initial surprise, *e.g.*, the use of mustard gas.

All the belligerents would have used chemical means to effect their military objects on a more stupendous scale than was already the case in 1918 had the war dragged on to a further Spring campaign. Already at the end of 1918 70% of the casualties were caused by gas.

So long as there is no counter to the rapid production of toxic substances in Germany, the world is not safe; the menace of the Rhine factories lies, not only in their geographical position, but in the fact that they are unique.

That the next war will begin as a chemical war is certain, because it is an arm which has now been tried, the principles of which are well known to soldiers. Large appropriations have been made by Congress in America for carrying out chemical warfare research, and very drastic steps have been taken in the United States to safeguard their dye industry.

Research.

It is said by some people that the only thing necessary to enable the British dyestuffs industry to hold its own against the German I.G. is to give it sufficient money for research. There is no stronger protagonist of research than I am, but it is my opinion that the mere endowment of laboratory research—and this I presume is meant—would not lead to any practical results. The creation of a great industry from a laboratory reaction or invention is not an automatic process. Organised industrial research can only be successfully carried out—and spasmodic research is of no importance—by great organisations willing to devote sums of money not only to employing a large scientific staff, but for experimental work on a large or semi-large scale, and to erect great factories. This presupposes a great industrial organisation with technical experience and a commercial organisation of sufficient insight and imagination, extending over the markets of the world.

The cost of the laboratory work is a comparatively small charge in the development of a new industry. To endow laboratory research for industrial purposes, unless the conditions for large-scale exploitation exist, would seem a curious procedure. What

does the inventor of a new laboratory reaction do if he has no such organisation behind him and if he is not backed by a large manufacturing concern able to assist in its technical development on the large scale? He can only sell his invention to those who have. What is required for this technical development? A staff of experienced technologists, a plant design section, a constructional engineering section, and, above all, the money to carry out the work on a large scale. The laboratory chemist is a pure research chemist and has, in my experience, neither the knowledge nor the desire for large-scale work. The qualities which make for success in these two branches are different and seldom combined in one person.

Thus the process of fixing atmospheric nitrogen in the form of ammonia was in a sense invented by Professor Haber, but its technical development was entirely due to the Badische Co., and its commercial value is enormously enhanced by the fact that it is exploited by a dye trust. Could Haber have developed this invention himself without the aid of the Badische Co.? The process should really be called the Haber-Bosch, not the Haber process. The first Badische synthetic Indigo was not discovered at Ludwigshafen, but by Henmann, professor of chemistry at Zürich. Could Henmann have developed his invention? The reaction was technically valueless and only became a commercial success after a great deal of money had been spent upon it by the able technologists of the Badische Co.

There are numerous other examples, *e.g.*, incandescence mantles, argon and the 3-watt lamp, helium and airships. The ammonia-soda process was patented by Dyar and Henning in 1838; yet it was only in 1865 that Ernest Solvay, the creator of the industry, was able to start a works near Charleroi. Even after Solvay had shown the way in 1873, Ludwig Mond, a man of outstanding talent, who obtained a licence from Solvay, risked all the capital that he and his partner had at Northwich, and only after incredible anxiety, assiduity, experimentation and failure the great firm of Brunner Mond emerged successfully.

Did the discovery of viscose by Cross and Bevan create the artificial silk industry which Messrs. Courtaulds now dominate? Everyone knows that that is not so.

We have to-day magnificent research organisations, directed by some of the most brilliant and original minds in the country, staffed by the best young chemists obtainable. We spent on these organisations, exclusive of capital, over £100,000 last year; we employed last year 100 academically trained research chemists. Could we have spent more? I think not at this stage of our development. We hope in due course to employ far more and to spend greater sums on research. But the development of research must be proportionate to the capacity of the factory organisation for making a profitable use of scientific work, just as the development which the factory can undergo is purely a function of the success of the commercial organisation in promoting sales. That is the essence, the vital point is harnessing science, industry, and commerce.

Conclusion.

If we desire our textile trades to flourish, unhampered by conditions or restrictions imposed upon them by a foreign trust; if we desire to carry on our lives peacefully, without a constant menace from these great factories on the Rhine, potential arsenals; if we desire to train our young men in the pursuit of chemistry to organise progress in the inventive industries, and not merely to rely on our staple industries while other nations more progressive launch out into the unknown; if we desire

these things, or any one of them, we will, as a nation, take any measures which may be necessary now and during the next few years to establish the coal tar colour industry in this country.

DISCUSSION.

SIR WILLIAM PEARCE, M.P., said that he had been a member of Lord Balfour of Burleigh's Committee, which nearly five years ago had first made the recommendation that key industries should be supported at any risk and at any cost; that had been the beginning of Government policy. One of the difficulties of the present Bill was that in the few years before the war the textile industry in this country had been treated by the Germans with the greatest consideration. The industry had not only had the most favoured clause with regard to prices but he believed dyes had been sold in this country to the textile industry at prices lower than in Germany or any other country in the world. Moreover, the big German organisations had sent their agents over here to give full information with regard to the application of a colour. The prices of German dyes having been so low, it had been no encouragement for anyone to embark on the dye industry here on a large scale without Government help. Dr. Levinstein had made one very good point as to how the textile industry, if it went back to these conditions, might find itself some day with its supply of dyes refused in the interests of the German textile industry, but even apart from that, there was something national at the back of the German dye industry. In 1913 the Germans had made 100,000 tons of ammonia from atmospheric nitrogen at a cost of roughly £5 per ton, and in 1914 the whole of that had been placed on the European market at £11 per ton. The Germans had known that if they went into a world war their stocks of sodium nitrate would soon have been exhausted, and they could not have carried on for two years but for the Haber process. What had been our position compared with Germany when the war broke out? The first thing that had come into prominence in the war had been anhydrous sulphuric acid. The total demand for anhydrous sulphuric acid in this country before the war had been under 500 tons per week, but the Germans had been producing 10,000 tons a week and possibly more. Directly we had come to manufacture nitroglycerin or gun-cotton or T.N.T. in large quantities we in this country had been in the greatest distress owing to the lack of sufficiently large plant. In regard to high explosives, we had again been met with the fact that there had been not only insufficient oleum, but no manufacture of T.N.T. on any scale at all in this country. At the time the present Prime Minister had stated from the Front Bench that, thanks to Lord Moulton, we had secured an ample supply of high explosives for ourselves and our allies, there was not 20 tons of T.N.T. per week being made in this country. Our requirements at the end of the war had been about 2000 tons. In the same way there had been at that time no plant in this country capable of producing a large quantity of chlorine, or even of hydrogen. He believed we had nearly lost the war because we had no mustard gas shells. The risk we ran had been frightful. Who could say that the same thing would not happen again if we did not possess a large organic and synthetic chemical industry in this country? It was necessary in the interests of this country, from the peace point of view, quite apart from the possibilities of war, that this industry should be allowed to establish itself. It was imperative that the Government should pass this Bill in the interests of the nation.

PROF. H. E. ARMSTRONG said he hoped that a copy of Dr. Levinstein's address would be sent to

every member of the House of Commons. It was unfortunate that it had not been delivered in time to produce an effect in connexion with the second reading of the Bill, because he could not imagine anything more likely to lead to a sweeping vote in favour of it. On the whole, they would be inclined to agree with Dr. Levinstein that research, after all, was no good if carried out merely from the academic point of view; that it had to be put into practice to make it of value. At the same time, he felt that Dr. Levinstein had, he was sure unconsciously, very much under-rated the value of ideas. The whole trend of late years had been very much in the direction of carrying out research work for the sake of the money to be made out of it. We should be raising the wrong type of man if we put that view forward, as had been the case lately. Over and over again he himself had expressed the view that the failure in this country was a commercial failure, due to lack of commercial insight, lack of breadth of outlook, on the commercial side, and that was one of the points which ought to be brought before the House. If there had not been this entire lack of foresight on the part of members of the House as a whole we should not be in the parlous position we were to-day, because they had not been sufficiently informed to appreciate the value of science. It was the very fact that the Germans had had this foresight that had given them their pre-eminence. There was no better evidence of this than the fact that the Badische Company had offered Dr. Emil Fischer £5000 a year when he, a young and unknown man, had discovered the nature of Rosaniline. Dr. Levinstein's main argument was a sound one, that this country was bound to fail if it did not develop the particular type of industry which he represented, but the subject must be looked at from a very much broader point of view than that which Dr. Levinstein had put before them. If we were to develop our agricultural resources and intensive culture to the necessary extent to meet the requirements of our growing population it would be through the agency of the organic chemist, and we must convince our legislators of the absolute need to develop this particular industry and of the importance of organic chemists for its progress.

MR. F. H. CARR agreed as to the vital importance of the organic chemical industry to this country, but pointed out that it was not alone the dye industry or that part of applied organic chemistry that was concerned with the next war, but the development of biochemistry, the advance of our knowledge of the vital elements for the preservation of life and for the destruction of life; that was the type of work which was bound to develop, that branch of our industry which was concerned with the production of drugs and chemicals of that type. It was for that reason he was convinced that it was of vital importance that not only the dye industry, but the whole of the organic chemical industry should be preserved in the interests of peace as well as war. With reference to what Prof. Armstrong had said as to the value of ideas, he had not understood Dr. Levinstein to speak disparagingly of the value of ideas at all, but rather to the effect that ideas, even with a well-paid chemical staff, cost less in money and less in patience than did the technical scale experiments, which required not merely large sums of money and good chemists, but large sums of money for workers on the engineering side, and so forth. He alluded to this because the British Dyestuffs Corporation and other dyestuffs companies which were operating in this country at the present time had not had the least chance of establishing their industry. Even if, during the few years they had been at work, they had had full opportunities, the time would not have been sufficient to put them in a position to

compete openly with an industry so highly developed as it was in Germany, but these companies had been working under war conditions and had not had anything like the proper opportunities. Therefore, not only must we expect to spend a lot of money on the technical scale experiments, but we must expect to exercise great patience in seeing a lot of mistakes made, and it was only by overcoming these mistakes that ultimate success would be obtained.

MR. R. WADDINGTON, M.P., said that he, together with many of his colleagues in the House of Commons, was satisfied that the Bill which had been presented by the Government was the best solution of the problem and would give the greatest satisfaction to the textile users, and at the same time be the best means of building up the dye industry in this country.

SIR CLEMENT KINLOCH COOKE, M.P., said that he personally was in favour of the Bill, and several other members of the House of Commons who had spoken to him on the subject were all agreed that the Bill was a good one and that they would support it. He had no doubt whatever that the second reading would be passed by a large majority.

DR. M. O. FORSTER said that Prof. Armstrong had referred to the question of commercial incentive to organic research. On that point he agreed with Mr. Carr's remarks. He had not understood Dr. Levinstein to say anything diminishing in any sense whatever the importance of ideas. What Dr. Levinstein had done was to emphasise the need of incentive to research; he regarded that as of tremendous importance. His own view was that a subsidy for the dye industry on the scale which would be required, i.e., a scale which would practically maintain the industry whilst it was not selling any goods at all, was impossible. He could not imagine anything more demoralising to the staff of the Corporation from the scientific standpoint than such a situation, because, delighted as they all were to do what was called pure research, and splendid as it was as a mental development, nevertheless, it was an incentive to these workers to think that they might hope that something useful would arise from what they were doing. If they went on year after year seeing nothing useful arise from what they did in the laboratory, if they merely saw their records filed and no goods sold, he could not imagine that any more young men would be inclined to go in for the study of organic chemistry. In his opinion the dye industry was not really a key industry, but merely a branch of the textile industry. Had the textile manufacturers fifty years ago taken that much broader view of the dye industry, and subsidised it by setting aside a percentage of their profits, they would have enabled an industry to be built up in this country just as it had been built up in Germany.

MR. REGINALD B. BROWN said that the correspondence, not only in *The Times* of London, but also in *The Manchester Guardian* during the last fortnight or so, had shown a most remarkable diversity of opinion as to the advisability of the measure now being brought forward and as to the precise terms of the Bill which would meet with the fullest support. One argument which was certain to be advanced, but which, perhaps, had not been considered sufficiently that evening, was that if we could not get constant supplies of German colours our textile industries here would suffer severely, and therefore we should suffer in competition in foreign markets. That was probably an argument of which the most would be made in the House of Commons. He believed he was correct in saying that the strongest opposition would come from the Calico Printers' Association, which represented users

of specially fast dyestuffs which were not yet produced in this country. Whilst that was to some extent correct, it was necessary to point out that the Bill did not entirely prohibit the importation of these special German dyestuffs at present, but merely said that they were to be imported under licence, and that if the Committee, which consisted of a majority of colour users, could prove to the satisfaction of the Board of Trade that these colours were necessary for their particular industry and were not being produced in sufficient quantity or of a sufficiently good quality in this country, then they could be imported. As Dr. Levinstein had said, there were vast numbers of chemists being trained in the colleges in this country, and it might reasonably be hoped that the industry, given a few years in which to build itself up, would, with the aid of these young chemists, be brought into such a position and gain such experience on the commercial side that all the needs of this country could be met. It was necessary to build up the industry by the co-operation of science, technology, and commerce, and only in that way would it be possible for the full range of colours required by the textile industry to be made available. He did not believe in the argument that the Germans would refuse to sell colours if the Bill went through, but he did believe that they would do all they could to kill the dye industry in this country if the Bill was not passed. He did not agree with Dr. Forster that the dye industry was a branch of the textile industry. It was much wider than that, because it touched many trades, such as the leather trade, the paint trade, the paper trade, and others which strictly would not be called textile industries. As an industry itself, the dye industry was of the greatest importance for our national industries and national life, and he hoped that those Members of Parliament who were present that evening would be convinced of the urgency of the Bill and be strengthened in their conviction of the necessity for it.

DR. STEPHEN MIALl strongly deprecated the manner in which this matter had been left until the very last minute. Owing to the disgraceful management of this business by successive Governments during the last few years, the country had been brought to such an extraordinarily dangerous position. It was far more serious than had been indicated in the discussion. The trade had now got into a state that only the very promptest measures could save it from absolute extinction.

MR. WALTER F. REID recalled the important part which Dr. Levinstein's father had played, not only in developing the dye industry in this country, but also in fighting the German monopoly and in regard to patents. Patents had been the means of controlling the German interests in this country and fighting the British. Mr. Ivan Levinstein had been a member of a deputation to the Board of Trade at the time Mr. Lloyd George had been President, and he (Mr. Reid) had represented the Society of Chemical Industry on that occasion. One of the points brought before Mr. Lloyd George had been that the fixation of nitrogen from the atmosphere was then being developed abroad, and that if it was not established in this country before the outbreak of a war we should find ourselves in a very awkward position. Mr. Lloyd George had at once asked for a small committee to be nominated to confer with him on the matter. The result of that had been the introduction of the compulsory working of patents in this country. That had had one small effect, that the Germans had erected a model factory in this country. Nevertheless, it had had some practical effect, but the Courts of Law had given a decision which rendered this absolutely useless in the same way as a recent judgment had done in regard to licences for importation. Chemists

ought to take some steps to see that the regulations that were passed and the advice given to the Government by the law officers was advice that was really of some use. With regard to research, he quite agreed that research alone was of little use; it was only those who were in actual contact with industry and who had the means of utilising the raw material who could put it to proper use. An inventive head was required, and this was a matter which should be thoroughly considered in the agitation for large sums to be spent on research work.

Mr. W. MACNAB said that a great deal had been made of the fact that in spite of our unpreparedness we had won the war. That was perfectly true, but it was not realised that the cost in money of the improvisation that had to be resorted to and in the suffering had been caused by our lack of preparedness. That was an aspect which should not be overlooked in regard to the future.

Mr. W. J. U. WOOLCOCK, M.P., said that it must not be imagined that the Government's Bill was purely and simply one for the prohibition of the import of foreign dyes into this country: it was a Bill for the establishment of a dye industry in this country. It was not proposed to prohibit straight away the whole importation of German dyes into this country without any consideration for the textile manufacturers; neither were they asking, as dye makers, for protection which would give no spur whatever to progress in the industry. On the contrary, the dye makers said that if they had the Bill they were prepared that the policy of the Licensing Committee should be to ask the British dye maker to demonstrate that he could produce the dyes required before there was any thought of cutting off the necessary supplies. That, to his mind, was one of the best features of the proposals, and he personally would never be a supporter of any protection for any industry which would cause that industry to go to seed and rest on protection. Such a course would not establish a dye industry in this country.

Mr. E. V. EVANS said that it was impossible under the present conditions to stand up against the form of competition that Germany was able to provide at the moment, and would be even more able to do in the future, unless some form of legislation were brought in to deal with it.

Dr. LEVINSTEIN, in reply, said that the degree of unanimity which had been shown with regard to the paper was greater than was usually obtained in a meeting of chemists. They were fortunate in having Sir William Pearce as their protagonist in the House of Commons for the Bill, and he had every hope that the result in the House of Commons would be as unanimous as the present meeting.

Communications.

THE DYEING OF ARTIFICIAL SILK.*

BY THE LATE LEONARD PHILIP WILSON AND MARJORIE IMISON.

The dyeing of artificial silk has, as a rule, been considered to be practically identical with the dyeing of cotton, but since it has a greater affinity for dyestuffs, and unevenness in shade is occasion-

ally observed in the dyed material, it was thought desirable to investigate the dyeing of viscose artificial silk from a fresh standpoint.

It has hitherto been assumed that all direct cotton colours are as suitable for use with artificial silk as with cotton, but recent research has clearly shown that only a limited number can be considered to be eminently suitable from the point of view of giving even effects. The reason for this is that many of these colours are extremely sensitive to those small differences of dyeing affinity which are at present liable to occur occasionally in artificial silk, as indeed they do in all textile fibres, but which in the case of short fibres, such as cotton and wool, are concealed by the mixing of a very large number of fibres to form a thread.

Among the sulphur colours also there are many which are very sensitive to variations in the affinity for dyes of the artificial silk fibres, but fortunately the "even" or less sensitive colours of this class include shades which help to fill in the gaps in the range of "even" direct cotton colours. By the term "even" as applied to dyestuffs in this connexion is meant the property of the dyestuff of giving an equal depth of colour on artificial silk fibres of different affinities.

Tannic acid, which is used as a mordant for basic dyestuffs, is taken up by the thread like a dyestuff and behaves to a certain extent in the objectionable manner of many of the direct cotton and sulphur colours; thus, if there are irregularities in the artificial silk, the basic colours when used with a tannic acid mordant may give uneven results. Where they are used for topping other dyestuffs, the final result is dependent upon the effect obtained with the bottom colour.

Sky Blue F.F. is one of the dyes which is frequently connected with complaints of uneven dyeing, but recent research has shown that if there are some conspicuous offenders in this respect, there are others, e.g., Chrysophenine G., of which no complaint can reasonably be made since they consistently give even shades when used in the orthodox manner.

Methods have been devised which serve to measure the extent to which a dyestuff can exhibit unevenness. A large number of dyes have been tested and, according to the degree of evenness which they show with fibres of known different affinities, when dyed under standard conditions, have been classified as "even," "moderately even," and "uneven."

No superiority in respect of evenness can be claimed for the products of the German dyeworks. A given dyestuff, whether it be labelled Diamine, Oxamine, Dianol, or Paramine, will be classed as "even" or "uneven" (as the case may be) according to its constitution and behaviour, which are independent of its German or British prefix. For example, Diamine Sky Blue F.F. (Cassella), Chlorazol Sky Blue F.F. (Brit. Dyestuffs Corp.), and Chloramine Sky Blue F.F. (Sandoz) are all "uneven," as are also Diamine Black B.H. (Cassella), Oxamine Black B.H. (Badische), Melantherine B.H. (Soc. Chem. Ind. Basle), Melantherine B.H. (Clayton Aniline), and Chloramine Black B.H. (Sandoz). On the other hand, Sultan Yellow G. (Brit. Dyestuffs Corp.) and Chrysophenine G. (various makers) are "even," while Cotton Red 4 B. (Soc. Chim. Ind. Basle), Benzopurpurin 4 B. (Sandoz), Benzopurpurin 4 B. (Brit. Dyestuffs Corp.), and Diamine Red 4 B. (Cassella) are "moderately even."

It has been found, however, that evenness (at normal dyeing temperature, 50° C.) varies according to the molecular weight of the dyestuff used;

* The reading of this paper at the meeting of the Birmingham Section on October 21st was cancelled owing to the sudden death of Mr. Wilson on October 16th.

the arrangement of the groups in the molecule and their nature have, apparently, no real effect. The dyestuffs of low molecular weight give very even results under normal dyeing conditions, although the silks may vary considerably; while with the same irregular silks the dyestuffs of high molecular weight give uneven results. For example, Rosophenine 10 B. (mol. wt. 600) is even dyeing, whereas Dianol Sky Blue (mol. wt. 992) is very uneven; Indoine Blue (mol. wt. approx. 521) is very even, but Dianol Fast Pink B.K. (mol. wt. 944) again is very uneven; Diamine Green B. (mol. wt. 812) is fairly uneven, and so on. The result is that fabric made up of artificial silk of varying affinities will show variations in shade when dyed with dyestuffs of high mol. wt. and a practically even effect if dyestuffs of low mol. wt. are employed.

The lists given below show the direct cotton colours which have been tested classified as those showing good evenness, those of moderate evenness, and the uneven ones, determinations having been made by dyeing at 50° C. The lists may include the same dyestuff under more than one name and must, for the present, be regarded as provisional and subject to alteration as research proceeds. A large number of German dyes which have been tested are not included.

EVEN DYES (DIRECT COTTON COLOURS).

Red.—Chlorazol Red A., Chlorazol Pink R.D., Chlorazol Fast Bordeaux B., Dianol Fast Red K., Dianol Fast Red F.G., Congo Corinth G.W., Congo Rubine (all British Dyestuffs Corporation), Chloramine Red B., Chloramine Fast Red F. (Sandoz), Rosophenine 10 B. (Clayton Aniline), Rosanthrene B., Rosanthrene R., Rosanthrene Bordeaux B., Cotton Red C. (Soc. Chem. Ind. Basle).

Brown.—Chlorazol Brown G.R. and Dianol Brown G.M. (British Dyestuffs Corp.), Trisulphon Brown M.B. and Chloramine Brown 2 R. (Sandoz), Cupranil Brown B. and Chlorantine Pure Brown 3 G.L. (S.C.I. Basle).

Orange.—Congo Orange R. (British Dyestuffs Corp.), Paramine Direct Orange R. (Holliday).

Yellow.—Chrysophenine G. (Sandoz), Sultan Yellow G. (British Dyestuffs Corp.), Cotton Yellow C.H. (S.C.I. Basle).

Blue.—Indoine Blue (Badische: this colour dyes artificial silk without a mordant).

Violet.—Chlorazol Violet W.B. (British Dyestuffs Corp.), Direct Violet 2 B. (S.C.I. Basle).

MODERATELY EVEN DYES.

Red.—Brilliant Congo Red R. (Sandoz).

Brown.—Paramine Direct Brown G. (Holliday), Dianol Brown 2494 (British Dyestuffs Corp.).

Orange.—Stilbene Orange J R. (Clayton Aniline).

Yellow.—Titan Yellow G. (British Dyestuffs Corp.).

Green.—Dianol Dark Green N. (British Dyestuffs Corp.).

Violet.—Direct Violet 2 R. (S.C.I. Basle).

Black.—Vulcan Black 2 G.W. (British Dyestuffs Corp.), Direct Fast Black B. (S.C.I. Basle).

UNEVEN DYES (DIRECT COTTON COLOURS).

Red.—Direct Fast Scarlet S.E. (S.C.I. Basle), Chloramine Brill. Red 8 B (Sandoz), Dianol Fast Pink B.K. (Brit. Dyestuffs Corp.).

Brown.—Trisulphon Brown B. and Trisulphon Bronze B. (Sandoz), Cupranil Brown G. (S.C.I. Basle).

Orange.—Pyrazole Orange G. (Sandoz).

Yellow.—Dianol Fast Yellow A.R.X. and Afghan Yellow G.X. (Brit. Dyestuffs Corp.), Paramine Direct Yellow R. (Holliday), Oxypheanine R. and Oxypheanine G.G. (Clayton Aniline).

Green.—Dianol Green B.G. (Brit. Dyestuffs Corp.), Direct Green G. and Direct Green B. (Sandoz), Direct Green B. (Clayton Aniline).

Blue.—Sky Blue F.F., Dark Blue B., Blue 2 B., Fast Blue G. (all makers), and many others.

Violet.—Trisulphon Violet B. (Sandoz), Direct Violet R. (Clayton Aniline), Chlorazol Violet B.N., Dianol Violet R. 9068 K., and Dianol Violet R. (Brit. Dyestuffs Corp.), Direct Violet N. (S.C.I. Basle).

Black.—Black B.H. (all makers), Chlorazol Black E. Extra and Dianol Black No. 1 (Brit. Dyestuffs Corp.), Chloramine Black H.W. and Chloramine Black E.X. Extra (Sandoz), Melantherine T.H. and Indigene Black B.W. (S.C.I. Basle).

From these lists it is apparent that there are many direct cotton dyestuffs which, when employed in the usual manner, will give unsatisfactory results if there is any unevenness in the artificial silk, but, on the other hand, a considerable number will give good and even shades in most colours. The work of investigation into the evenness of dyestuffs is still in progress, but up to the present only one or two satisfactory direct blues have been found, and for most blue shades other methods must be adopted to ensure more even results. It may, however, be possible now that the test of evenness has been ascertained, for manufacturers to produce blue dyestuffs of low molecular weight, and investigations have been started with this end in view.

It has been found, moreover, that it is possible by dyeing at other temperatures to obtain more even results with dyestuffs which, when tested at 50° C., were classified as uneven. With higher temperatures of the dyebath, the amounts of dye absorbed by two silks of very different affinities gradually approach one another, so that whereas when dyed at 35° C. one silk might be six times as dark as the other, when dyed at 75° C. there is very little difference between them; the silks, of course, were immersed in the bath at the proper high temperature and not put into a cool bath and subsequently heated.

When using mixtures of direct cotton dyestuffs, even results can only be obtained if all the dyestuffs in the mixture are even; a dyebath composed of both even and uneven dyes almost invariably gives worse results than the uneven dyes alone. It has also been found that when material dyed with an uneven dye and showing variation in shade is bleached and afterwards re-dyed with an even dye, the final result will frequently be uneven.

It has already been stated that some of the sulphur colours will help to supply the deficiencies in the direct colours. The sulphur dyes as a class cannot, however, be said to give even results, as many of them do not, but it is fortunate that among the blue sulphur colours some have been found which give even shades on artificial silk of varying affinity. It is possible, therefore, by the use of these to extend considerably the range of colours which do not show unevenness, and the following dyestuffs can be recommended to this end.

EVEN DYESTUFFS (SULPHUR COLOURS).

Thionol Sky Blue P.X. (Brit. Dyestuffs Corp.), Pyrogene Direct Blue R.L., Pyrogene Direct Blue (green shade), and Pyrogene Indigo (S.C.I. Basle), Thionone Brilliant Blue G. and Thionone Navy Blue B. (Holliday).

The sulphur colours named below have been found to give uneven results:—

UNEVEN DYES (SULPHUR COLOURS).

Blue.—Thionol Navy Blue R., Thionol Navy Blue 9055 K., Thionol Direct Blue S. (Brit. Dyestuffs Corp.), Thional Dark Blue S. and Thional Dark Blue R.L. (Sandoz), Thionone Indigo R. and Thionone Navy Blue 2 R. (Holliday).

Black.—Thionol Black O.G., Thionol Black XXN Conc., and Thionol Black S.G.G. (Brit. Dyestuffs Corp.), Thionone Black Paste (Holliday).

Green.—Thionol Green D.Y. (Brit. Dyestuffs Corp.), Pyrogene Green 3 G. (S.C.I. Basle), Thional Brill. Green 2 G. (Sandoz).

Brown.—Thionol Brown O. (Brit. Dyestuffs Corp.).

The basic colours find considerable application on artificial silk where great fastness in the dyed silk is not essential. In addition to producing extremely bright shades, basic dyes as a class possesses the great advantage of giving the most even dyeing results. In fact, almost without exception, these colours can be made to give shades which show no sign of unevenness when used to dye artificial silks of very different affinities. It should be noted that these dyes are of low molecular weight.

With the usual method of dyeing, *i.e.*, with a preliminary mordanting with tannic acid and tartar emetic, unevenness may, as stated above, sometimes result when the material is dyed, but to a much smaller extent than with many of the direct colours. The most even results are to be obtained by dyeing first in a neutral dyebath, and then fixing in tannic acid and tartar emetic.

The fact that basic colours are, on the whole, even dyeing, enhances their value for topping direct or sulphur colours and extends the range of shades which can be produced, and which might be curtailed by the reduction in the number of dyes available if only those which fulfil the requirements of even dyeing are to be used.

It has thus been found that, among all classes of dyestuffs investigated, there are some which under normal conditions of dyeing exhibit great sensitiveness to variations in artificial silk, and special methods have been indicated for dyeing with some of these colours which tend to make them less sensitive. On the other hand, there are many dyes of undoubted even-dyeing properties covering, in one class or another, a full range of colours; and it is hoped that the extended use of these and the restricted or more careful use of uneven colours will tend further to eliminate irregularities in dyed viscose silk.

A CONTRIBUTION TO THE THEORY OF POLYMERISATION IN FATTY OILS.

BY ARTHUR H. SALWAY, D.S.C., PH.D.

The phenomenon of polymerisation is best observed with highly unsaturated oils, notably Chinese wood oil (tung oil) and linseed oil, but is not

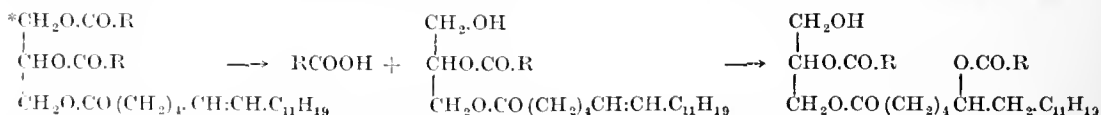
Polymerisation of an oil is accompanied by an increase in the density, viscosity, refractive index,* and mean molecular weight of the oil, and a decrease in the iodine absorption. The melting point of the total fatty acids is raised slightly by polymerisation, whilst the proportion of free fatty acid is also increased to some extent (Wolff, *Farbenzeit.*, 1913, 18, 1171). Furthermore, it is stated that the glyceryl radicle remains unchanged during polymerisation, whilst the saponification value of the oil is not materially affected.

Several attempts have been made from time to time to explain the physical and chemical changes attending the process of polymerisation (*e.g.*, Kronstein, *Ber.*, 1902, 35, 4153; Kitt, *Chem. Rev. Fett- u. Harz-Ind.*, 1905, 12, 241; Normann, *Chem.-Zeit.*, 1907, 188; Wolff, *loc. cit.*; Morrell, *J.*, 1915, 105; Fahrion, *Chem. Umschau*, 1917, 24, 103), but none of the theories so far advanced can be considered to provide an entirely satisfactory explanation of the change. Hitherto it has generally been considered that the unsaturated fatty acid radicle in the fatty oil is the seat of the polymerisation. Morrell (*J.*, 1918, 181) has, however, recently shown that the fatty acids (or methyl esters) obtained from polymerised tung oil are of normal molecular weight, from which it may be concluded that depolymerisation occurs with surprising ease, or that polymerisation is not entirely conditioned by the unsaturated acids. In fact, it seems necessary, for a satisfactory explanation of the observed experimental data, to assume that the glyceryl radicle is also involved in the polymerisation. It is stated, however (Lewkowitsch, *J.*, 1903, 592), that the glycerol is unaltered during polymerisation, although Kitt (*loc. cit.*) observed that an apparent loss of glycerol occurred in polymerising linseed oil. Kitt attached no special theoretical significance to this result, for, according to his theory, polymerisation of fatty oils consists first in the production of free fatty acids, which then condense with the production of inner anhydrides or lactenes.

There is much to be said for this view, especially if it be so modified to include a change in the glyceryl radicle. For this reason the author advances the suggestion that the mechanism of the process is as follows:—At the high temperature necessary for polymerisation, there is first a liberation of one or more fatty acid radicles, which then condense with the unsaturated linkages of the fatty oil. This condensation takes place between the free acidic groups on the one hand and the unsaturated linkages of fat on the other hand, and the result is a partial saturation of the fat. This change may in general be represented as shown below†.

It will be seen that as a result of the reaction formulated above, one glyceryl hydroxyl group becomes free. At the high temperature required for polymerisation, however, such a compound might be expected to condense with the formation of a polyglycerol derivative as represented below, thus accounting for the increased weight of a polymerised oil.

According to this view, polymerisation consists in an internal rearrangement of fatty acid radicles

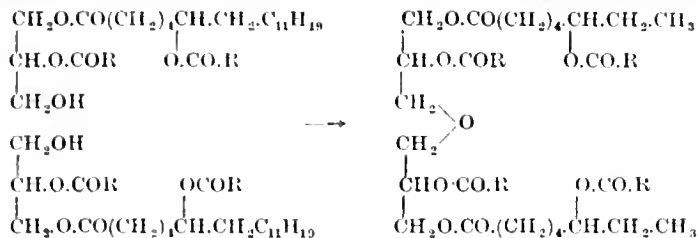


entirely peculiar to highly unsaturated oils, for most fatty oils can be made to undergo the change to a greater or less extent. For example, olive, arachis and cottonseed oils contain a considerable proportion of glyceride which gelatinises like tung oil if the non-polymerisable portion of the oil is removed by distillation (Kronstein, *Ber.*, 1916, 49, 722).

* A decrease in refractive index sometimes occurs when tung oil is polymerised.

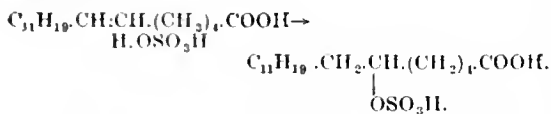
† These formulae must not be regarded as representing the exact change of any particular oil; they are only intended to indicate the way in which the unsaturated linkages and a fatty acid radicle are involved in the process. The change is not necessarily limited to one fatty acid radicle and one unsaturated linkage; numerous combinations are possible.

involving a partial saturation of the molecule and the production of a polyglycerol fat. A change of such a character accounts satisfactorily for the observed increase in molecular weight, density, and viscosity of the oil, as well as the decrease in iodine value. It would explain why polymerised oils have substantially the same saponification value before and after polymerisation and why the fatty acids



from a polymerised oil have a normal molecular weight. The two main reactions which occur during polymerisation of oils according to this theory are (a) the union of a carboxylic group of a fatty acid with an unsaturated linkage or series of unsaturated linkages, and (b) the condensation of a so-called diglyceride with the formation of a polyglyceride.

With regard to the first reaction, it is well known that unsaturated fatty acids readily combine with sulphuric acid, with the formation of sulphuric esters according to the following scheme:—



A similar reaction undoubtedly occurs when a highly unsaturated fatty acid is heated alone (Fahrión, *Farben-Zeit.*, 1913, 18, 2111), for in this case, instead of the sulphuric acid, a second molecule of the fatty acid is involved in the condensation, an O.CO.R group taking the place of the O.SO₃H in the above scheme. The result is a reduction both in iodine value and acid value.

Experimental evidence in support of this conclusion was obtained by heating fatty acids from linseed oil and palm oil separately at 270° C. for 6 hours in the presence of carbon dioxide and then observing the change in iodine and acid values of the fatty acids. The results were:—

		Linseed oil fatty acids.	Palm oil fatty acids.
Iodine value	Before polymerisation	200.7	55
	After	176.9	54.5
Acid value	Before	191.5	199
	After	181.1	198
Sap. value	Before	196.1	200
	After	198.5	201

It is clear from these figures that in the case of the linseed oil fatty acids, a reduction in iodine value is accompanied by a reduction in acid value, whilst with the comparatively saturated acids of palm oil there is no appreciable reduction either in iodine value or acid value. This is a fact of some significance from which it may be concluded that the reduction of acidity is not due to anhydride formation, R.CO.OH + (R.CO)₂O, but is correlated with the reduction of iodine value and brought about in the manner formulated above.

Since fatty acids are thus shown to be capable of combining with highly unsaturated linkages, it is only reasonable to conclude that a similar reaction would occur between a fatty acid and an unsaturated glyceride in the manner suggested above in the first stage of the polymerisation of fatty oils. The free fatty acid required for that reaction

may be originally present in the fat or it may be produced during polymerisation. There is, indeed, reason to believe that all fats when heated to the usual polymerisation temperature undergo a kind of dissociation with the liberation of fatty acid, for it is precisely at this temperature, 250°–270° C., that heated fats begin to evolve visible amounts of volatile substances. Kronstein's observation that

olive oil, arachis oil, cotton seed oil, etc., are polymerised when distilled is of interest in this connection (cf. Kraft, *Ber.*, 1903, 36, 4343, and Bömer and Baumann, *Z. Unters. Nahr. Genussm.*, 1920, 40, 97, on the temperature of decomposition of fats when distilled in a high vacuum).

If this view of the first stage in the polymerisation is correct the introduction of free fatty acid should accelerate the reduction in iodine value. On the other hand, if the formation of free fatty is retarded by any means, the reduction in iodine value on heating should also be retarded. That this is actually the case is shown by polymerising whale oil in the presence of an excess of fatty acid on the one hand, and in the presence of an excess of glycerin on the other hand. In the former case, the rate of reduction of iodine value was found to be accelerated; in the latter case retarded. In the experiments to demonstrate this fact, a sample of crude whale oil was refined by means of caustic soda and fuller's earth, after which the oil was washed and dried at 100° C. under diminished pressure. For the polymerisation, samples of the oil were heated for 8 hours at 250°–270° C. in sealed tubes in the presence of carbon dioxide. The tubes were heated side by side in an oil bath under identical conditions. Two series of experiments were made, in one of which known amounts of fatty acid from whale oil were added before polymerising, whilst in the other series glycerin was added in known amounts. The results of the experiments were:—

Effect of free fatty acid.

		Before treatment.	Iodine value. After treatment.
Whale oil (neutral)	..	121	106.8
	.. + 1% F.A.	121	100.5
	.. + 2% F.A.	121	99.8
	.. + 3% F.A.	121	97.6

Effect of glycerin.

		Before treatment.	Iodine value. After treatment.	Reduction %
Whale oil (neutral)	..	121	94.8	21.8
	.. + 10% glycerin	110	93.6	15.1
	.. + 20% ..	101	89.9	11.0

It is thus evident that the reduction of iodine value is accelerated by the addition of free fatty acid and retarded by the addition of glycerin.

Passing on to the second stage of the above theory of polymerisation, namely, the stage involving a condensation of the glyceryl radicle, the evidence is less convincing. According to this view the glycerol obtained from a polymerised oil should contain polyglycerol, but no direct evidence has yet been obtained that this is the case. It is, however, well known that glycerides containing free glyceryl hydroxyl groups (such as the so-called mono-glycerides) when heated to high temperatures readily undergo change with elimination of water.

Thus monoacetin loses water at a comparatively low temperature and is converted into polyacetin (diacetyldiglycide). The present author has also observed a similar change with monostearin. If monostearin is heated at 180° C. for several hours, the amount of free hydroxyl in the substance is considerably reduced, as can be shown by determinations of the acetyl value of the product.

There is thus considerable evidence to show that the reactions here formulated may readily proceed under the conditions of polymerisation of fatty oils.

The author recognises, however, that further experimental verification of this theory is needed, especially regarding the formation of a polyglycerol fat during polymerisation, before the theory can be accepted without reserve.

The experiments recorded in this paper were undertaken by Mr. L. V. Cocks, F.I.C., to whom the author's best thanks are due.

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NOTES ON THE U.S. GOVERNMENT METHOD OF DETERMINING PHENOLS IN SHEEP DIPS.

BY K. B. EDWARDS, B.S.C., AND G. A. FREAK, B.S.C., F.I.C.

In the U.S. Government method of determining phenols in sheep dips the phenols are isolated from the acidified dip by steam distillation and eventually obtained in benzene solution.* This solution is shaken in a graduated tube with a 1:3 sodium hydroxide solution, and, after the lapse of sufficient time to allow of complete separation, the increase in volume of the alkaline solution is measured. Each cubic centimetre increase in volume is then taken as representing 1 g. of phenols.

Chapin states* that with weighed amounts of pure phenols the coefficient weight of phenols ÷ volume increase of NaOH is constant for a given phenol irrespective of the amount measured or of the presence or absence of other coal tar hydrocarbons in addition to benzene. In support of this the following coefficients, obtained with different weights of "fairly pure cresylic acid," are given:—1.055, 1.078, 1.079, 1.076, 1.070.

In connexion with this coefficient, Chapin says:—"This coefficient is not the same for all phenols, but varies in the same direction as the specific gravity of different phenols, though in greater ratio, accordingly varying inversely as the molecular weights of the different phenols and in approximately equal inverse ratio. For the mixtures of phenols ordinarily occurring in commercial cresylic acid, and in the grades of coal tar creosote oils commonly used for making dips, the average coefficient proved to be unity as nearly as could be determined." He gives, however, no figures obtained with commercial creosote oils or cresylic acids, and, in view of the fact that the commercial products vary to a great extent, it is important to know whether the variation exhibited by the coefficient is large or sufficiently small to be neglected.

TABLE 1.

Phenol.	Coefficient	Weight of phenols Increase in vol. of NaOH*			Apparent % phenols taking coefficient as 1.
		I.	II.	Mean.	
Phenol..	..	1.081	1.075	1.078	92.7
<i>o</i> -Cresol..	..	1.058	1.064	1.061	94.2
<i>m</i> -Cresol..	..	0.995	0.990	0.992	100.7
<i>p</i> -Cresol..	..	0.971	0.950	0.975	102.5

This point has therefore been investigated with results as shown in Table 1. These tests and those

on purified cresylic acid, to be described later, were carried out in the prescribed apparatus, using 10 g. of the pure phenol in 60 c.c. of benzene.

These figures show clearly that the nature of the phenol is all-important, ordinary phenol showing an excess of 8% and *p*-cresol a deficiency of 2.5%. The various xylenols were not available for test at this time, but results of tests made by one of us some nine years ago with pure xylenols indicated that the increase in the molecular weight would result in still further discrepancy. This is confirmed by the results obtained with different fractions of purified cresylic acid. Commercial cresylic acid was purified by dissolving it in an excess of sodium hydroxide solution, distilling with steam in order to remove neutral oils and bases, and recovering the phenols by treatment with acid. These phenols were then divided into fractions by distillation from a flask fitted with a four-bulb Young fractionating column. In addition, a portion of the acid was completely distilled until decomposition commenced in the flask, the product being designated below as "complete mixed distillate."

Table 2 shows the results obtained:—

TABLE 2.

Fraction.	Apparent % of Coefficient. phenols (coefficient 1).	
Below 195° C. ..	1.052	95.0
195°-197° ..	1.042	96.0
197°-200° ..	1.026	97.5
200°-205° ..	1.015	98.5
205°-215° ..	0.990	101.0
215°-225° ..	0.943	105.5
225°-235° ..	0.922	108.5
235°-250° ..	0.900	111.5
Above 250° (to about 300°) ..	0.851	117.6
Complete mixed distillate ..	0.985	101.6

Comparison of Table 2 with Table 1 shows that the cresylic acid fractions behave in a manner analogous to the isolated phenols, i.e., the coefficient is less for the higher-boiling compounds.

It is therefore obvious that the method is open to serious objection, inasmuch as a low-boiling acid, which will contain a large proportion of phenol and *o*-cresol, may yield results considerably below the truth.

It may be contended that an error of about 5% is not a serious one in a method designed to estimate cresylic acid in sheep dips containing, say, 20% of phenols. Unfortunately, however, this United States Government method has been recommended as the standard test in evaluating cresylic creosotes, and cresylic acids bought for sheep dip and disinfectant purposes, and American buyers appear invariably to test material by this process. Cresylic acid for the American market is almost invariably supplied to a specification as to percentage and boiling points, and in our experience the specifications usually indicate a demand for cresylic acid having a large percentage of *o*-cresol. It will be seen from the above figures that when such an acid is supplied the method of testing must show the purity as being 4-6% below the true content of phenols; such a difference on a very large shipment represents a considerable sum of money. The authors have in mind a specification which demanded that 60-70% of the cresylic acid supplied should boil between 185° and 195° C. This specification invariably was the cause of claims for impurity to a very large sum, inasmuch as acids containing 97% of cresols showed 92%, according to this test. It should also be noted that the test is only designed for dealing with phenols boiling between 185° and 250°, inasmuch as the phenols have to be steam distilled, and phenols boiling above 250° are extremely difficult to volatilise with steam. The British test upon which creosote oils are evaluated does not make any such distinction, and phenols are usually estimated by removal from the oil with caustic soda solution of sp. gr. 1.10. There are many

* The analysis of coal-tar creosote and cresylic acid sheep dips.
R. M. Chapin. Bulletin 107, Bureau of Animal Industry, U.S. Dept. of Agri., 1908.

creosotes produced to-day in which a considerable proportion of the phenol content boils above 250° C., and such creosotes are likely to increase in number, owing to the various forms of low-temperature carbonisation employed. Such creosote oils will show extremely erratic results when tested by the American method as compared with the usual method adopted in this country, and the American specification will shut out any creosote containing a large proportion of these valuable disinfectant substances.

It will be clear from the figures given in this paper that phenols boiling above 250° C. show a considerably higher degree of purity than is actually present. Against this, however, must be placed the fact that such phenols will not volatilise with steam within the limits for practical estimation. There are, therefore, two balancing factors which will tend to produce results which in isolated cases may be somewhere near the truth but are by no means to be relied upon.

There is at present a very great need for a simple and accurate test for the determination of the purity of cresylic acids. The standard method of the text-books, namely, extraction by ether or petroleum spirit from an alkaline solution, is by no means reliable in the presence of very high-boiling phenols or in the presence of relatively large percentages of bases which are apt to volatilise in the removal of the solvent. In our experience the percentage figures attached to cresylic acid obtainable in the open market are purely nominal, there being extremely few makes of cresylic acid which can be relied upon to be of 98% purity, although practically all pale cresylic acid is sold as 97–99%. A 97% acid is difficult to obtain in actual practice except at a greatly enhanced price, and very many of the so-called 97–99% cresylic acids contain not more than 94% of pure phenols. A recent sample of white “99% cresylic acid” examined by the authors contained less than 97% of the pure phenols.

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THE DISTRIBUTION OF ENZYMES AND PROTEINS IN THE ENDOSPERM OF THE WHEAT BERRY.

BY F. J. MARTIN, M.A.

In the separation of flour by the process of gradual reduction, now in general use in the milling industry, different parts of the wheat berry tend to segregate at various points of the mill; it is therefore of great interest and importance to the miller to know how far and in what manner the proteins and enzymes that occur in the wheat berry are liable to vary.

Jago (“Technology of Bread-making,” p. 371) describes a method by which the uneven distribution of the protein in the wheat berry may be demonstrated qualitatively. The starch is removed from a cross-section of the grain, which is then stained with Millon’s reagent; the gradual concentration of the gluten in the endosperm as the cortex is approached is evidenced by the vivid coloration of the protein fibrils. Richardson (*ibid.*, p. 351), who made a very complete examination of the mill products from various mills, shows in his analyses that there is a very wide variation in the gluten content of flours produced at various parts of the mill. As experience does not show that the flour from the tail of the mill is of much greater “strength” than flour from the other reductions, an examination of the milling products, representing flour from different parts of the endosperm, was carried out.

The work recorded in this paper was carried out to investigate the nature and extent of the variations, if any, in the flour from different parts of

the wheat berry with respect to enzymic activity and quantity and quality of gluten.

In order to avoid any segregation effects due to the milling of a mixture of wheats, a single wheat, Barusso Plate, was milled for this experiment. Samples of flour were taken as follows:—

(a) Reduction flours:—

(1) From ABC reductions, corresponding to the central portion of the grain.

(2) From DEG reductions corresponding to a region exterior to the central portion.

(3) From K reductions corresponding to a layer nearer the cortex than DEG.

(4) From MN reductions corresponding to the region nearest the cortex.

(b) Break flours:—

(1) From BM₁, a flour from the central portions of the grain.

(2) From Br₁, a flour having its origin near the cortex.

(c) Straight grade flour—representing the entire endosperm.

Enzymic activity and quantity of gluten.—The germ is always regarded as the seat of enzymic activity, but since in the process of modern milling the germ is practically all eliminated and collected separately, an examination of the various flours was made to see how far the endosperm was homogeneous as regards diastatic strength. The enzymic activity was measured as suggested by Wood (J. Agric. Sci., 1907, 2, 139) by the amount of gas the flour was capable of producing in 24 hours when fermented with yeast. A dough was made up of 10 g. of flour and 5 g. of water, with 1·2% of salt and 1·0% of yeast; the temperature was maintained at 29° C. and the carbon dioxide collected for 24 hours. The results are given in Table I.

The gluten in all the samples was estimated by first washing away the starch and then weighing both while wet and after drying at 100° C.

TABLE I.

Flour.	Amount of CO ₂ in c.c.	Wet gluten. %	Dry gluten. %
a 1 ..	85	40·0	13·8
a 2 ..	93	46·5	15·9
a 3 ..	115	50·2	17·4
a 4 ..	122	53·6	17·9
b 1 ..	58	45·4	15·9
b 2 ..	83	63·5	21·6
c ..	88	45·3	15·6

It will be seen that the enzymic activity of the flour increased from the centre to the exterior of the endosperm, the variation being represented by 58 c.c. and 122 c.c. in the extreme cases.

The quantity of gluten is least in the centre of the grain and increases towards the exterior of the endosperm. In the case examined the extremes stated as dry gluten were 13·8% and 21·6% respectively.

Quality of gluten.—The rather large difference in the amounts of gluten present in the various flours examined suggested that these differences would be reflected in the baking tests; but previous experience had not borne this out. It was therefore thought that the quality of the gluten from the various parts of the grain might vary. In handling the glutens separated by washing it was noticed that the gluten removed from the flour obtained from the centre of the grain was of a more elastic and rubber-like consistency than that from the flour from near the cortex, the differences in characteristics between the extremes being very marked.

In order to test the gas-retaining capacity of the gluten from various parts of the wheat berry the

flours from the different points of the mill were mixed with a calculated amount of wheat starch so that each mixture of flour and wheat starch contained the same amount of gluten. Doughs were made up of flour and wheat starch 20 g., water 11 g., salt 1.2%, yeast 1.0%, and fermented at 29° C. in graduated cylinders, the carbon dioxide generated being collected over brine. The volumes of the doughs and the gas generated were read at frequent intervals. Curves constructed from these figures gave the gas-retaining power of the gluten (Martin J., 1920, 146 r). The gluten of the flour from the ABC reductions (a1) showed the greatest capacity for gas-retaining, and for the purpose of this paper the gas-retaining power of this gluten is taken as 1.00 and the other glutes referred to this standard; the results are given in Table 2.

Gas-retaining capacity of the flours.—The gas-retaining capacity of the flour was calculated by multiplying the percentage of gluten in the flour by the gas-retaining capacity of the gluten; the results are also given in Table 2.

TABLE 2.

Flour.	Relative gas-retaining capacity of gluten.	Dry gluten. %	Gas-retaining capacity of flour.
a 1	1.00	13.8	13.8
a 2	0.93	15.9	14.8
a 3	0.86	17.4	14.9
a 4	0.77	17.9	13.8
b 1	0.86	15.9	13.7
b 2	0.64	21.6	13.8
c	0.90	15.6	14.0

There is thus a considerable difference in the gas-retaining capacity of the gluten from different parts of the wheat berry, the better qualities of gluten being found in flour from the central portions of the endosperm, but the variations in the quantity of the gluten, which increases from the interior to the exterior of the endosperm, are practically counterbalanced by the variations in the quality of the gluten, which progressively deteriorates from the centre to the outer parts of the endosperm, and flour of a more or less uniform "strength" is produced. The flours from DEG and K reductions (a2 and a3), however, appear to be slightly stronger than the others.

Confirmatory trials.—The results of the above tests were confirmed by baking trials. It was not thought advisable to bake the Barusso Plate flours by themselves on account of their low gas-producing capacity and high gluten content. Each sample of flour was therefore mixed with an equal weight of flour from English wheat, of high diastatic capacity and low gluten content. The English flour had a

gas-retaining capacity of 8.0 when referred to the standard used for the Barusso Plate, so that the gas-retaining powers of the mixtures, by calculation, were as shown in Table 3. The gas-producing capacity of the English flour was 253 c.c. when estimated under the same conditions as the Barusso Plate flour. The calculated diastatic capacities of the mixed flours are also given in Table 3. The addition of a percentage of another flour had the effect of reducing proportionally any differences originally present in the Barusso flours, but this could not be avoided.

The loaves were baked on the short straight dough process, the fermentation in the troughs lasting four hours. Sufficient flour was included in each trial to make ten "cottage" and two "tin" loaves. Bakers marks were awarded by Mr. A. E. Humphries, under whose supervision these trials were made (Table 3). It will be seen that the different batches were so alike with respect to "strength" that they were awarded the same mark.

TABLE 3.

Flour 50% English and 50% as below.	Calculated gas-producing power of mixed flour in c.c.	Calculated gas-retaining power of mixed flour.	Baker's mark.
a 1	169	10.9	82
a 2	173	11.4	82
a 3	184	11.5	82
a 4	188	10.9	82
b 1	156	10.9	82
b 2	168	10.9	82
c	171	11.0	82

Summary.—(1) The distribution of the enzymes and proteins in the wheat berry has been investigated. It has been found that the enzymic activity, as shown by the evolution of carbon dioxide on fermentation, increases progressively from the interior to the exterior of the endosperm.

(2) The quantity of gluten increases in the same manner.

(3) The quality of the gluten varies considerably, being good in flour from the interior of the endosperm and deteriorating progressively in flour from regions approaching the cortex.

(4) The effect of the combination of the increasing quantity with decreasing quality of gluten tends to produce flours of a common "strength" from various parts of the wheat berry.

In conclusion, the author wishes to express his thanks to Mr. A. E. Humphries for permission to make use of figures obtained while working at Coxes Leck Milling Co., Weybridge.

Wellcome Tropical Research Laboratories,
Khartoum.

Yorkshire Section.

Meeting held at Leeds on May 3, 1920.

MR. W. MCD. MACKEY IN THE CHAIR.

A GRAPHICAL METHOD FOR THE INTERPRETATION OF FLUE GAS ANALYSES.

BY GILBERT B. HOWARTH.

[Department of Coal Gas and Fuel Industries, the University, Leeds.]

Complete tests of the thermal efficiency of boilers, furnaces, and similar appliances generally involve a series of laborious calculations intended to determine:—

(1) The ratio of the amount of air supplied for combustion to the minimum amount of air theoretically required for complete combustion, and

(2) The ratio of the amount of heat in the hot flue gases to the amount of heat liberated by the combustion of the fuel.

If these calculations can be generalised in such a way that the results may be represented on a system of co-ordinates, the determinations may be effected rapidly from the smallest possible number of experimental data.

The combustion with excess of air of any fuel containing only carbon, hydrogen, oxygen, and nitrogen may usually be regarded as producing flue gases composed of a mixture of carbon dioxide, water vapour, oxygen, and nitrogen. In the usual processes of gas analysis water is eliminated and the composition of the dry gas obtained. The ratio of the volume of water vapour in the flue gases to the volume of carbon dioxide is equal to half the ratio of hydrogen atoms to carbon atoms in the fuel.

The composition of the dry flue gases may be completely defined by a statement of the percentages of any two of the dry constituents (say oxygen and carbon dioxide), the percentage of the third (in this case nitrogen) being obtained, if required, by difference. These quantities may be calculated for the combustion of any given fuel with any given percentage excess of air by constructing a chemical equation. Thus there are four variable quantities:—

P=Percentage of oxygen in the dry flue gases.

Q=Percentage of carbon dioxide in the dry flue gases.

x =Extra air, expressed as a percentage of the minimum quantity of air theoretically required for complete combustion.

n =A quantity depending on and to be calculated from the composition of a fuel, and which is the same for those fuels (and no others) which, when burnt with a given percentage of extra air, give flue gases of the same dry composition,

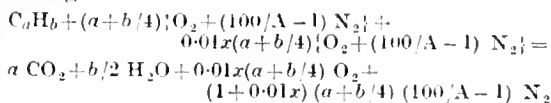
and from any given values of n and x it is possible to construct a chemical equation, and from it to calculate corresponding values of P and Q.

Suppose that the values of P and Q calculated for a number of values of n and x are plotted on a system of rectangular co-ordinates, and that lines are drawn through all points obtained from the same value of n , and another series of lines through all points obtained from the same values of x . The

two series of lines thus drawn will constitute an irregular grid superimposed on the original rectangular grid. This double grid constitutes the basis of the "graphical method" to be described in this paper. It will be seen that for any point on the diagram the four quantities, P, Q, n , and x may be read off at once on the two grids. Moreover, any two of the four quantities are sufficient to define a point on the diagram from which the other two quantities may be obtained at once by inspection.

The commonest use of the diagram for industrial purposes is the following:—The "fuel factor" (n) being known from the composition of the fuel, the only observation required is the percentage of carbon dioxide in the dry flue gases in order to obtain accurately from the diagram the percentage of oxygen in the dry flue gases and the percentage of extra air. Moreover, if the composition of the fuel is not known, the value of n can be found from the diagram by a single determination of carbon dioxide and oxygen in the dry flue gases.

The calculation of the co-ordinates of multitudes of points from a corresponding number of chemical equations (as supposed in the preceding paragraph) is not actually required in practice. The chemical equation representing the combustion of C_aH_b with $x\%$ of extra air containing $A\%$ of oxygen is the following:—



From this it is seen that

$$a + 0.01x(a+b/4) + (1+0.01x)(a+b/4)(100/A-1)$$

volumes of dry flue gases contain a volumes of CO_2 and $0.01x(a+b/4)$ volumes of O_2

Then percentage of oxygen = $P =$

$$\frac{x(1+b/4a)}{1+0.01x(1+b/4a) + (1+0.01x)(1+b/4a)(100/A-1)}$$

and percentage of carbon dioxide = $Q =$

$$\frac{100}{1+0.01x(1+b/4a) + (1+0.01x)(1+b/4a)(100/A-1)}$$

Since the values of P and Q for a given value of x depend only on the ratio b/a , we may accept this ratio as fulfilling the function of the quantity n mentioned above. Hence we may put $n=b/a$. Then if $A=21.0$, from these two expressions, by the elimination of x , the following general equation for the "fuel lines" is obtained:—

$$P + (1+0.1975n)Q = 21.0$$

which is the general equation of a series of straight lines (one for every value of n), all of which pass through the point (21,0).

By the elimination of n from the same expressions, the following general equation for the "extra air lines" is obtained:—

$$(376/x + 4.76)P + Q = 100$$

which is the general equation of a series of straight lines (one for every value of x), all of which pass through the point (0,100).

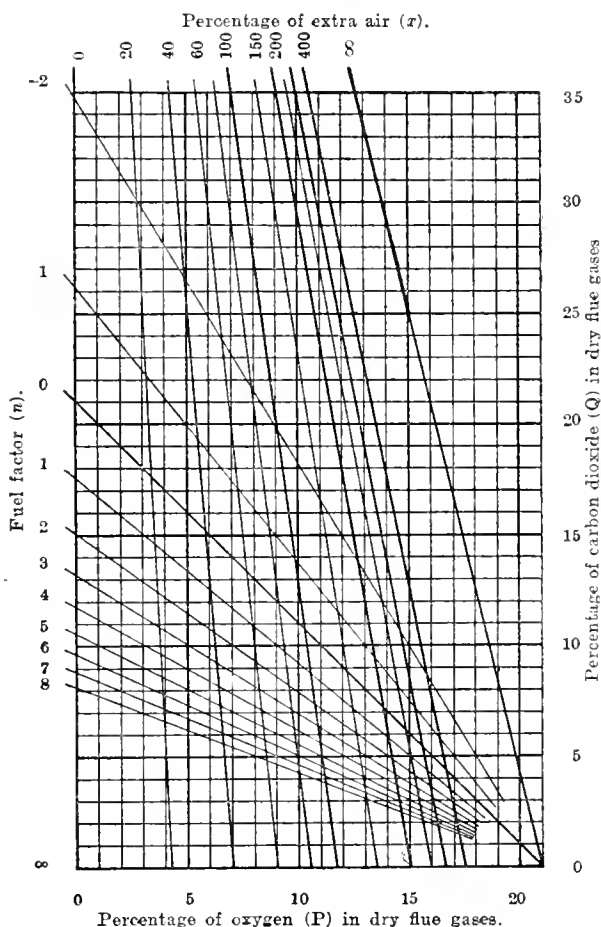
These results, shown graphically in the extra air diagram (fig. 1), are quite in accord with the disposition of the lines that might be expected from general considerations.

It should be noted that when $P=0$ (i.e., complete combustion without excess of air) the equation of the fuel lines becomes

$$Q = 21.0 / (1 + 0.1975n)$$

thus giving the percentage of carbon dioxide obtainable in such ideal conditions.

It has been shown above that any hydrocarbon may be completely defined for the present purpose by the ratio of the number of atoms of hydrogen to the number of atoms of carbon accompanying them, i.e., $n = H/C$. Thus, for example, n is 1 for benzene or acetylene, 2 for olefines, 2.4 for pentane.



The Extra Air Diagram.

FIG. 1.

Other methods of defining hydrocarbons (e.g., by the gravimetric percentage of one of the elements, etc.) may be used, provided that the numbers attached to the fuel lines on the "Extra air diagram" are amended accordingly.

It is evident that the addition to or removal from a fuel of oxygen and hydrogen in the proportion to form water will have no influence whatever on the composition of the dry flue gases. Thus, to obtain n we must subtract from the number of atoms of hydrogen two atoms of hydrogen for every atom of oxygen in the fuel and divide the remaining number by the number of atoms of carbon, i.e., $n = (H - 2O)/C$. Where a fuel is rich in oxygen, the value of n thus obtained may be negative; thus for carbon monoxide $n = -2$, and for carbon dioxide $n = -4$.

The majority of pure combustibles may be dealt with by the formula $n = (H - 2O)/C$. For example, n is 2 for alcohol or ether, and 0 for carbohydrates.

Nitrogen in the fuel is assumed to be associated with a portion of the oxygen in the proportion to

form air. Thus, for example, the products obtainable by the complete combustion of $C_aH_bO_cN_d$ without excess of air will have the same composition as those obtainable by the complete combustion of $C_aH_bO_{c-d/3.76}$ without excess of air. This suggests that the value of n is the same for both substances and is equal to $\{b - 2(c - d/3.76)\}/a$ or, using chemical symbols, $n = (H - 2O + 0.532N)/C$. But the minimum quantity of air required for the combustion of a molecule of $C_aH_bO_cN_d$ is $a + b/4 - c/2$ volumes and for the combustion of a molecule of $C_aH_bO_{c-d/3.76}$ is $a + b/4 - (c - d/3.76)/2$ volumes, and any extra air supplied is measured as a percentage of one or other of these two quantities according to the formula used to represent the combustible. Therefore the percentage of extra air given by the chart (which utilises the second expression) must be converted into the units obtainable by actual measurement (according to the first expression) by multiplying by

$$(a + b/4 - c/2 + d/7.52)/(a + b/4 + c/2).$$

In other words, the chart readings (x) must be increased by a certain fraction, $\delta x/x$, viz.,

$$\frac{\delta x}{x} = \frac{d/7.52}{a + b/4 - c/2} = \frac{0.532d}{4a + b - 2c}$$

or, reverting to the use of chemical symbols, the correction to be added to the percentage of extra air given by the chart

$$= \delta x = \frac{0.532N}{4C + H - 2O} \cdot x$$

In the same way, if the percentage of extra air is included in the data, this ($x + \delta x$) must first be reduced to the chart readings (x) by subtracting the following correction from the percentage of extra air as given:—

$$\delta x = \frac{0.532N}{4C + H - 2O + 0.532N} \cdot (x + \delta x)$$

For many industrial purposes (for example, routine tests to regulate the supply of secondary air to a gas-fired furnace) absolute values of the extra air are not essential, and relative values are almost as useful in controlling the approach to the theoretical minimum. In this case the correction δx need not be applied, the figures obtained being regarded as relatively correct. The errors thus introduced become smaller as the theoretical minimum of air is approached. Or an approximation may be made by using a simple empirical formula applicable to a given class of fuel.

An alternative method of dealing with nitrogen is based on the fact that the addition of nitrogen to a fuel reduces the proportions of oxygen and carbon dioxide in the flue gases in the same proportion. Though this treatment is not usually convenient in practice, it provides an interesting confirmation of the arguments used above and a simple method of verifying the accuracy with which the diagram is drawn.

We may consider that the combustible sulphur in the fuel burns completely to sulphur dioxide. Atoms of sulphur should be added to atoms of carbon, the SO_2 being recorded as CO_2 in the flue gas analysis. The molecular ratio of the two gases will evidently be the same as the atomic ratio of the elements. In practice the proportion of sulphur in the fuel is usually small, and the presence of this element may be ignored, or its quantity assumed to have a suitable average value for all fuels of a given type.

The above formulæ deal with the atomic composition of the fuel. It will be clear that they may be adapted to suit gravimetric composition, or, in the case of gases, volumetric composition. These formulæ are summarised in Table I.

The part of the "Extra air diagram" above and to the right of the line corresponding to $n = -4$ and $x = \infty$, and the part below the P-axis ($n = \infty$) do not appear to be applicable to ordinary industrial fuels.

The region to the left of the Q-axis involves negative values for the extra air and oxygen. Evidently this area covers the important case of *incomplete combustion*, where the negative percentage of extra air implies a deficiency of air and the negative percentage of oxygen implies the presence of an equivalent quantity of unconsumed combustibles. It seems possible that this region might be utilised by considering separately the fuel factor of the fuel burnt, and the fuel factor of the combustible material remaining in the flue gases. But the process becomes very complex when combustion is seriously incomplete.

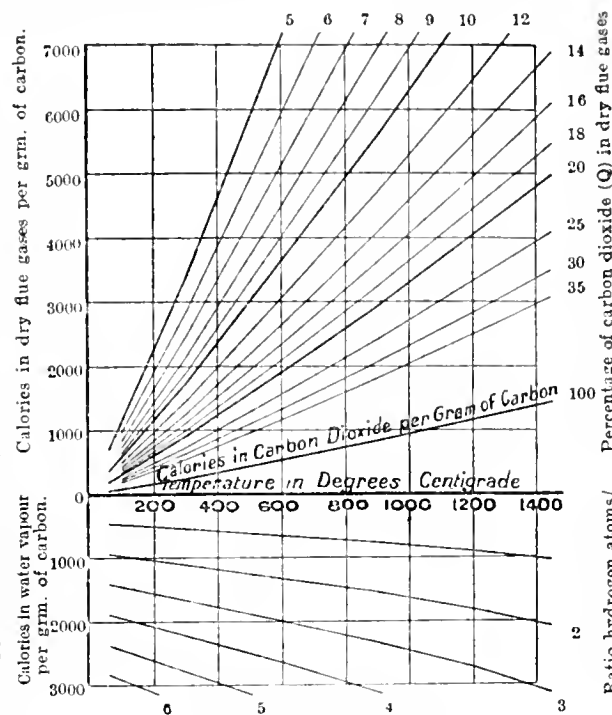
For any physical properties of the dry flue gases that may be calculated from their composition (e.g., density, heat capacity over any stated range of temperature, etc.) further series of lines may be drawn on the "Extra air diagram." The determination of carbon dioxide in the dry flue gases may then be replaced by a determination of any physical property for which curves have been prepared. In practice it may be more convenient to make the measurements on the flue gases saturated with water vapour at a pre-arranged temperature, this modification being taken into account in the calculation of the curves to be superimposed on the extra air diagram.

The author has no difficulty in using an extra air diagram photographically reduced from the original large and complex diagram to a size of about 2 in. \times 3½ in. For industrial purposes it is suggested that a diagram about 30 in. \times 18 in. with many more lines should be reproduced by lithography in a single colour (to avoid registration troubles). From such a diagram, suitably mounted on sheet zinc, or three-ply board, any draughtsman could trace the lines in distinct colours, the tracing being confined to that part of the diagram that was of particular interest for the purpose in view.

The composition of the dry flue gases being known, either by actual analysis or by the use of the diagram already described, the thermal capacity of the dry flue gases over any given range of temperature may be calculated from the mean specific heats at constant pressure of the various constituents over the given range.

From the large number of specific heat determinations that have been made the calorimetric measurements up to 1400° C. carried out by Holborn and Henning may be selected. Their results are so arranged as to give for each gas the mean specific heat at constant pressure from 0° C. to t° C. in the form of a linear or quadratic expression in t . (For water vapour 100° C. forms the lower limit.) Since oxygen and nitrogen have practically the same molecular specific heats, Q and t are the only data required for the calculation of the heat contained in a given amount of the dry flue gases. Moreover, since the weight of carbon burnt in the formation of this quantity of dry flue gas can easily be found, the quantity of heat in the dry flue gases formed during the combustion of unit weight of carbon may be obtained. The equation connecting this quantity with Q and t is the general equation of the curves forming the upper part of the "Thermal diagram" (fig. 2). It will also be recognised that, of this quantity of heat per gram of carbon, the quantity of heat per gram of carbon contained in the carbon dioxide alone is given by the curve corresponding to $Q=100$.

The lower part of the thermal diagram shows the total heat (sensible plus latent) contained in the water vapour accompanying the above quantity of dry flue gases. For any given temperature this may be expressed in terms of K , which is the ratio of hydrogen atoms to carbon atoms in the fuel burnt. Evidently the total heat in the whole flue gases per gram of carbon burnt is the sum of these two quantities (i.e., for dry flue gases and for water vapour), and is obtained by measuring along the ordinate corresponding to the observed temperature the intercept between the curve corresponding to Q (the percentage of carbon dioxide in the dry flue gases) and that corresponding to K (the ratio of hydrogen atoms to carbon atoms in the fuel burnt).



The Thermal Diagram.

FIG. 2.

According to Holborn and Henning the mean specific heats at constant pressure from 0° C. to t° C. (in calories per gram) are as follows:—

$$\begin{aligned} \text{N}_2 & 0.2350 + 0.000019t \\ \text{CO}_2 & 0.2010 + 0.0000742t - 0.000000018t^2 \\ \text{H}_2\text{O (from 100° C.)} & 0.4669 - 0.0000168t + 0.000000044t^2 \end{aligned}$$

Calculation from these gives:—

$$\begin{aligned} \text{Heat in dry flue gases per gram of carbon burnt} \\ = (54.83t + 0.00443t^2)/Q + 0.189t + 0.000228t^2 \\ - 0.000000066t^3 \end{aligned}$$

from which the upper part of the thermal diagram may be plotted. Also total heat in water vapour produced during the combustion of 1 g. of carbon

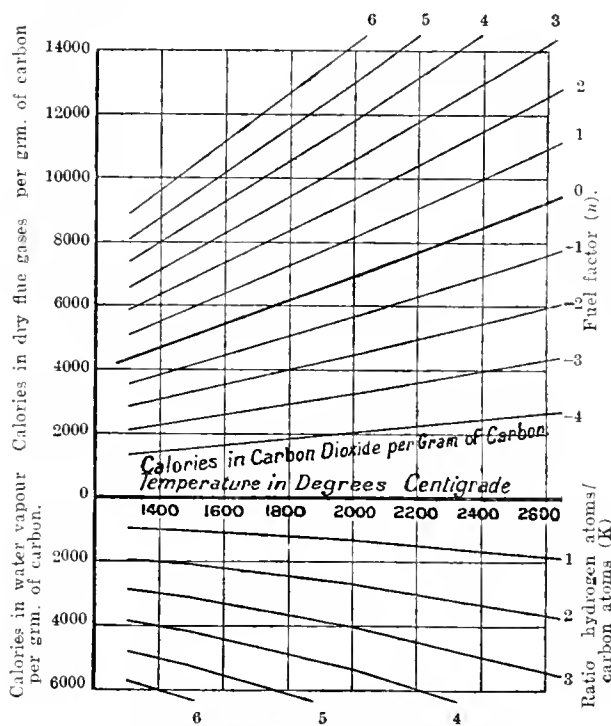
$$= K(445.0 + 0.3515t - 0.0000159t^2 + 0.000000033t^3)$$

from which the lower part of the thermal diagram may be plotted.

The expression "Theoretical flame temperature" is often used to denote that temperature at which, when a fuel is burnt with the minimum quantity of air, the heat in the flue gases is equal to the heat developed by the combustion of the fuel. Occasionally a specified fraction of the heat developed by combustion is employed, in order to

allow for radiation losses from the flame. Evidently this temperature may be found by means of a thermal diagram resembling that already described. However, in general the "theoretical flame temperature" of industrial fuels lies between 1500° C. and 2500° C.—beyond the scope of Holborn and Henning's specific heat determinations. The most convenient data to use are those collated by Lewis and Randall (J. Amer. Chem. Soc., Sept., 1912), who modified a large variety of experimental results to agree with theoretical requirements, thus obtaining simple formulæ likely to be reasonably accurate over a wide range of temperature, certainly up to 2000° C.

As in this case the fuel is always supposed to be burnt with the theoretical minimum quantity of air, we may substitute for Q the value given previously—when $P=0$, $Q=21.0/(1+0.1975n)$, so that the lines in the upper part of the diagram may be labelled with values of n instead of with values of Q . Then, n and K being known from the analysis of the fuel, and the calorific value of the fuel per gram of carbon being obtained, the "theoretical flame temperature" may be read from the diagram (fig. 3).



The Flame Temperature Diagram.
FIG. 3.

As before, the heat contained in the carbon dioxide alone is given by the curve corresponding to $n = -4$.

According to Lewis and Randall the specific heats at constant pressure at T° C. absolute (in calories per g.-mol.) are as follows:—

$$\begin{aligned} \text{N}_2 \text{ and O}_2 & 6.5 + 0.0010T \\ \text{CO}_2 & 7.0 + 0.0071T - 0.00000186T^2 \\ \text{H}_2\text{O} & 8.81 - 0.0019T + 0.00000222T^2 \end{aligned}$$

Calculation from these gives:—

Heat in dry flue gases per gram of carbon burnt

$$= n(0.531t + 0.0000392t^2) + 2.854t + 0.000411t^2 - 0.00000052t^3$$

from which the upper part of the flame temperature diagram may be plotted.

Also total heat in water vapour produced during the combustion of 1 g. of carbon

$$= K(445.0 + 0.3523t - 0.00001458t^2 + 0.0000000308t^3)$$

from which the lower part of the flame temperature diagram may be plotted.

TABLE I.

SUMMARISED FORMULÆ.

From atomic composition.

$$n = (H - 2O + 0.532N) / (C + S)$$

$$\delta x / x = 53.2N / \{4(C + S) + H - 2O\} \%$$

$$K = H / C$$

From gravimetric composition.

$$n = (4H - \frac{1}{2}O + 0.152N) / (\frac{1}{3}C + \frac{1}{8}S)$$

$$\delta x / x = 15.2N / \{4(\frac{1}{3}C + \frac{1}{8}S) + 4H - \frac{1}{2}O\} \%$$

$$K = 12H / C$$

From volumetric composition of gases.*

$$n = [6C_3H_8 + 4\{CH_4 - (CO_2 + O_2)\} + 2(H_2S + H_2 - CO) + 1.064N_2] / (3C_3H_8 + CH_4 + CO + CO_2 + H_2S)$$

$$\delta x / x = 53.2N_2 / (9C_3H_8 + 4CH_4 + 3H_2S + CO + H_2 - 2O_2) \%$$

$$K = (6C_3H_8 + 4CH_4 + 2H_2 + 2H_2S) / (3C_3H_8 + CH_4 + CO + CO_2)$$

Approximations (subject to amendment).

For coals (gravimetric composition).

$$\delta x / x = \frac{1}{3}N\% \text{ but is practically always negligible.}$$

For industrial gases over 400 B.Th.U. (gross) per cub. ft.

$$\delta x / x = (300 \times N_2) / (2 \times \text{cal. val.}) \%$$

(from volumetric composition).

For industrial gases under 400 B.Th.U. (gross) per cub. ft.

$$\delta x / x = (700 \times N_2) / (4 \times \text{cal. val.}) \%$$

(from volumetric composition).

TABLE II.

Values of n , $\delta x / x$ and K for various solid industrial fuels.

	Lignite	S. Yorks. bituminous.	S. Wales bituminous.	S. Wales steam.	S. Wales anthracite.	Gas coke.
C ..	59.5	75.9	79.1	87.6	90.4	87.2
H ..	6.0	5.2	4.5	4.1	3.3	1.2
O ..	27.3	9.3	6.8	1.2	2.5	2.3
N ..	0.5	1.3	1.0	2.8	0.9	0.8
S ..	1.5	2.8	0.9	0.8	0.9	1.2
Ash.	5.2	5.5	7.7	3.5	2.0	7.3
	100.0	100.0	100.0	100.0	100.0	100.0
n ..	0.52	0.63	0.56	0.55	0.40	0.13
$\delta x / x$..	0.08%	0.17%	0.13%	0.32%	0.10%	0.10%
N_2 ..	0.06%	0.16%	0.13%	0.35%	0.11%	0.10%
K ..	1.21	0.82	0.68	0.56	0.44	0.17

* C_3H_8 is the assumed average composition of the unsaturated hydrocarbons.

TABLE III.

Values of n , $\delta x/x$, and K for various gaseous industrial fuels.

	American carburetted water gas.	Coal gas, horizontal retorts.	Coal gas, vertical retorts.	Debenzolisied coke oven gas.	Present day town's gas.	Blue water gas.	Producer gas (bituminous coal—no steam).	Mond gas.	Blast furnace gas.
Cal. Val. ^a H, Th. U. per c. ft.	713	625	587	526	480	316	153	152	106
C ₆ H ₆ ..	3.0	2.5	2.5	2.5	4.0	4.5	1.0	17.0	10.0
CO ..	27.0	5.5	10.0	6.5	12.0	43.0	29.0	11.0	30.0
C ₂ H ₄ ..	12.5	3.5	3.5	2.0	1.0	—	—	—	—
CH ₄ ..	19.5	33.0	27.0	25.0	28.0	0.5	2.0	3.0	—
H ₂ ..	30.5	49.0	52.0	55.0	34.0	48.0	10.0	24.0	1.0
O ₂ ..	1.0	0.5	0.5	0.5	1.0	—	—	—	—
N ₂ ..	6.5	6.0	4.5	8.5	20.0	4.0	58.0	45.0	59.0
	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
n ..	1.73	4.55	4.12	5.15	3.48	0.21	0.87	0.58	—0.83
$\delta x/x$..	1.46%	1.47%	1.19%	2.33%	6.44%	2.25%	65.7%	50.9%	101.5%
Approx.† ..	1.37%	1.44%	1.15%	2.42%	6.25%	2.22%	65.5%	51.8%	97.5%
K ..	2.46	4.88	4.60	5.55	3.96	2.04	0.88	0.52	0.05

^a C₂H₄ is the assumed average composition of the unsaturated hydrocarbons.

† Using the formulæ given in Table I.

DISCUSSION.

Mr. HUDSON pointed out that the quality of fuel was very variable even throughout the same commitment, and expressed a doubt as to the applicability of the method to ordinary works conditions.

The CHAIRMAN asked if the method could be adapted to the ordinary fuel analysis results expressed as volatile matter etc.

Mr. TUTTON said that it was most important in works practice not to rely entirely on the percentage of carbon dioxide in the flue gas. Unless carbon monoxide was estimated the deductions made only from the dioxide figure might be very misleading.

Prof. COAN said that the present scheme was a great advance on the work of Bunte, who had had to construct a separate table for each fuel. The author's graphs were applicable to practically all fuels and resulted in an enormous saving of time and trouble when it was necessary to calculate flue gas analyses in terms of heat loss, excess of air, and flame temperature.

The AUTHOR, in reply, pointed out that the values of n for all the coals given in Table II., ranging from lignite to anthracite, lay between 0.63 and 0.40, representing a difference of only about 0.5 in the CO₂ percentage, and that the much smaller accidental variations in quality could usually be ignored. The conversion of fuel analyses from terms of volatile matter etc. into ultimate analyses was quite a different problem and was not touched on in the present paper.

portion dealing with research carried out at the Research Department, Woolwich, has been published in the Journal of the Chemical Society and in this Journal by Drs. Gibson and Masson, Messrs. McCall, Spencer and Barrett, and Miss Jacobs; the second portion forms the subject of this paper.

In the chemical industries employing cellulose as their fundamental raw material the systematic scientific attention given to the physical properties of the cellulose and the relation of these to the physical properties of its derivatives required for producing the ultimate finished product was, until recently, small in relation to its importance. It was of course known that certain types of cellulose gave better results from the physical point of view than others, and a selection as the result of experience was thus made. The desired results were not then obtained through scientific knowledge of the properties of the cellulose used, but by the methods of manufacture of its derivatives. These methods also were adopted as the result of long and expensive experience rather than by a scientific knowledge admitting explanation of the way in which the various factors in the processes affected the properties of the derivatives. The ultimate products of the cellulose chemical industries comprise mostly the results of the further treatments of the cellulose derivatives prepared. From the choice of the raw material to the application of the finished product the colloidal nature of the cellulose is generally a controlling factor.

From this point of view the stages of manufacture are all inter-connected, and generally it is advisable to control them all as far as possible, commencing at the bottom with the selection of the raw cellulose material and the treatment to be given to it in its purification. It is in this particular section that published information is most lacking and practice actually most backward. The essential physical property of cellulose and its derivatives from the practical point of view is viscosity, and in the processes for the manufacture of artificial silks, celluloid and plastics, cellulose varnishes, lacquers, etc., viscosity determinations of the cellulose derivatives have already become important, though the methods followed and the results obtained have not been traced back to show definite relationships throughout.

During the war the nitrocellulose industry in its particular application to propellant explosives manufacture became pre-eminent, and the results which had been obtained in this work to the date of the Armistice have been concerned with this particular section of the industry, which was under

Communications.

COTTON CELLULOSE.

THE APPLICATION TO INDUSTRY OF RECENT RESEARCHES ON ITS VISCOSITY.

BY R. A. PUNTER, B.SC., A.I.C.

Introduction.

In carrying out the work now under report, the closest co-operation between business, technology and research was maintained. For publication it has been thought desirable to divide the report into two sections, namely, research, and the application of the results of this research. The

the directorship of Sir F. L. Nathan, K.B.E., Department of Explosives Supply, Ministry of Munitions of War.

For the manufacture of nitrocotton for propellants before the war, cotton waste chiefly from the home spinning mills was used after purification. The purification was carried out by a number of small firms who supplied their products direct to propellants manufacturers, and the product supplied was required to conform to a Government specification dated March 23, 1908. This specification gave considerable latitude to the mills both in the choice of the raw materials and in the purification treatment which could be followed. It aimed at the preclusion, to what was considered a sufficient extent, of materials other than normal resistant cellulose without any reference to the effect which the treatments employed for attaining this object might have on the colloidal properties of the cellulose and on subsequent manufacture.

The importance of this side of the question had been realised by the Research Department, Royal Arsenal, Woolwich, before the war, but as the research was comprehensive it had to give place to other more urgent problems after a preliminary report dealing with viscosity was issued in 1914. Until towards the end of 1915 nothing further was done, and in the interval the production of cotton waste for propellants, largely in the hands of a company (the British and Foreign Supply Association, owning eleven out of eighteen cotton waste purification mills and producing 80–90% of the total production in agreement with the Ministry of Munitions) had been increased to about 1000 tons per week. To meet this production about 1500 tons of raw materials had to be treated per week, necessitating the heavy importation of a large variety of cotton cellulose materials. Very crude methods of treatment were in general followed without any technical supervision. The purified wastes, although complying with the specification, were very different in physical properties, which affected subsequent manufactures, and the price of the waste had risen in the beginning of 1916 to £75 per ton from the pre-war figure of £44 per ton.

In 1916 a shortage of bleaching powder, difficulties connected with the colloidal properties of the materials, experienced in the manufacture of the new cordite R.D.B., and the decision to undertake the manufacture of the American nitrocellulose powder in this country caused the research investigations to be renewed.

In May, 1917, satisfactory methods for the determination of the viscosity of cellulose prepared from cotton wastes and of nitrocellulose had been established by the Research Department, Woolwich (*cf.* Gibson, Spencer, and McCall, *Chem. Soc. Trans.*, 1920, 117, 479; Gibson and Jacobs, *ibid.*, 473), and from these it was found that:

(1) Considerable variations in the viscosities of the purified cottons from the different mills existed.

(2) The causes of the variations in these viscosities were due to some extent to the purification treatments followed, the main factors being the conditions of the soda boiling treatment.

(3) The viscosity of cotton cellulose bore a direct qualitative relation to the viscosity of the nitrocellulose prepared from it in the laboratory.

It was already known that the viscosity of the nitrocotton influenced the amount of solvent required in the actual manufacture of propellants, but little attention had been paid to this point in practice.

The stage had therefore been reached at which useful experimental work on a large scale could be undertaken, and Sir Frederick Nathan, with Mr. P. Tainsh and the author, arranged, in collaboration with the Research Department, Woolwich, for a comprehensive series of experiments with the

object of providing a more uniform, cheaper, and more suitable cotton cellulose for the manufacture of cordite R.D.B.

At the same time a provisional treatment pending the result of these experiments and based on the results already available was agreed upon, and issued to the mills. The following is the treatment which was issued:

1. *Blending*.—The blending of the raw materials is to be done carefully in the proportions in which they are received. Where possible, blending of the raw materials is to be carried out before kiering.

2. *Kiering*.—Each kier is to be charged with a fixed quantity of cotton and kiered with a definite proportion of alkali solution of constant strength. The solution is to be prepared outside the kier and the sediment allowed to settle; it should give 3.0% free caustic soda and should contain sufficient sodium carbonate to render the liquor at the end of the boil alkaline to the extent of approximately 1% as NaOH.

The time and pressure of the boiling is to be always the same for each kier and as far as possible is to be 10 hours at 40 lb. At certain mills where kiers boiling at two or three different pressures are in operation, the products from these kiers are to be blended in the ratio of the output of the kiers. After kiering the alkaline liquor is to be displaced from the cotton before the latter is removed from the kier.

3. *Black washing*.—As at present.

4. *Bleaching*.—The cold bleaching liquor, prepared apart from the cotton and settled, is to be of definite strength, approx. 0.1% available free chlorine. Circulation during bleaching to be as effective as possible. Bleaching to be continued for a definite period, and the liquor to be run off and the cotton washed before souring.

5. *Souring*.—The acid solution is to be of sufficient strength to reduce the ash of the cotton to specification (about 0.5% as H_2SO_4 , or lower if possible).

6. *Washing, etc.*—Cotton to be washed till neutral, hydro-extracted, wet-willowed where possible, dried at 95–100° C., dry-willowed, and teased.

None of the results obtained from the work done by the Research Department, Woolwich, up to this stage have been given, as these are being included in the publications referred to above.

Work on the preparation of a cotton suitable for nitrocellulose powder followed on similar lines to the work done for cordite R.D.B., but at a later date, and was guided by the latter. It is dealt with in a subsequent section of this paper.

Experimental work on the preparation of cotton cellulose for cordite R.D.B.

Throughout the mills various types of plant were in operation, particularly in regard to kiers, comprising open vessels up to vessels passed for and worked at 80 lb. steam pressure, though of material boiled at pressures higher than 40 lb., the output could be but small. It appeared from previous work that 40 lb. pressure would be sufficient to give, from the raw materials to be used, a purified cotton with a viscosity as low as it was thought would be required.

Plant capacity determined the maximum initial ratio of solution to cotton in the kiers at 4 to 1.

The following experiments were arranged to determine a method of preparation of cotton waste most suitable for the manufacture of nitrocotton for cordite R.D.B.:—

Series 1.

It is known that the character of the cotton waste used is related to the resistance offered by the

cordite R.D.B. dough in the presses and in consequence to the quantity of solvent required for incorporation, and it is thought that this again is related to the viscosity of the nitrocellulose and possibly also to that of the raw cotton. It was decided to ascertain empirically the effect on the character of the cordite dough of the various factors under control in the preparation of the waste, and, if possible, to obtain a test which when applied to the waste would afford an indication of its suitability for cordite manufacture in this respect.

The raw materials available were:—

- (A) English spinners' sweeps, degreased.
- (B) American spinners' sweeps, non-degreased.
- (C) Eastern spinners' sweeps, non-degreased.
- (D) American willowed fly.
- (E) Eastern card fly.
- (F) American cops.
- (G) Eastern cops.

The English spinners' sweeps contained 17·3% of oil and was, therefore, initially degreased by extracting three times with benzene, followed by steaming off to remove the residual solvent.

Each of these classes of raw material was submitted to the following treatment:—Degreased if necessary; kiered with a completely causticised 3% NaOH solution at 40 lb. for 10 hours, the propor-

D 25, E 25, F 5, G 5, and with this blend the following samples were prepared, the treatment given being that described above unless otherwise specified.

When the specification tests of these samples were known, a suitable treatment was applied to a uniform blend of cotton of the composition given above, and from the resulting product the following samples were prepared:—No. 27, no bleaching or souring; 28, bleached with 0·1% available free chlorine solution, soured with 0·5% acid solution, and thoroughly washed; 29, unbleached but soured with 0·5% acid solution (weaker if possible) and thoroughly washed.

The specification tests were carried out according to standard methods.

The following tables show the results of tests:—

TABLE I.
Tests of raw materials.

Sample no. ..	A.	B.	C.	D.	E.	F.	G.
Viscosity C.G.S. units ..	435	781	192	995	163	1512	1677
Oil, % ..	17·3	13·5	11·5	0·5	2·2	0·62	0·9
Ash, % ..	4·67	8·3	6·9	2·2	3·3	1·20	1·0

TABLE II.

Tests of purified materials.

Sample no. ..	A.	B.	C.	D.	E.	F.	G.	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26
Viscosity C.G.S. units ..	9	16	10	128	53	57	20	332	193	317	256	226	79	34	95	35	156	48	4	129	17	172	162	73	24
Oil, % ..	0·24	2·7	2·5	0·19	0·19	0·35	0·46	0·61	0·56	0·66	0·87	0·66	0·81	0·60	0·73	0·65	0·74	0·47	0·44	0·53	0·53	0·59	0·68	0·95	1·00
Ash, % ..	0·81	1·0	1·4	0·60	0·82	0·56	0·54	0·60	0·63	0·87	0·71	0·62	0·72	0·80	0·87	0·88	1·14	0·87	1·14	1·12	1·23	1·18	0·72	1·06	0·71
Copper number ..	0·32	0·20	0·24	0·25	0·37	0·28	0·25	0·36	0·30	0·37	0·41	0·33	0·30	0·38	0·37	0·39	0·38	0·22	0·21	0·22	0·21	0·16	0·19	0·30	0·35
Solubility in 3% NaOH, % ..	2·7	2·3	2·4	2·3	1·4	2·3	2·0	2·7	2·4	2·9	2·7	2·8	2·5	2·8	2·7	2·8	2·9	2·0	2·8	2·8	3·7	2·8	3·0	2·7	3·5

Specification: Oil, 0·6%; solubility in 3% NaOH, 5·0%; copper number, 1·0; ash, 0·8%.

tion of solution to cotton being 4:1; washed with water in the kier till the alkaline liquor was completely displaced; black washed till neutral, using plentiful quantity of water; hydro-extracted; dried at 95°—100° C.; willowed once.

Sample no.	Pressure, lb. per sq. in.	Strength of alkali.		Time, hours.
		NaOH, %	Na ₂ CO ₃ calc. as NaOH, %	
9	0	4	—	10
10	0	4	—	20
11	0	2	—	10
12	0	2	—	20
13	20	2	—	10
14	20	2	—	20
15	20	4	—	10
16	20	4	—	20
17	20	2	—	10
18	20	2	—	20
19	40	4	—	10
20	40	4	—	20
21	40	2	—	10
22	40	2	—	20
23	40	1	3	10
24	40	1	3	20
25	40	2	—	10
26	40	2	—	20

Samples A, B, E, and F were nitrated for manufacturing trials.

For the soda boil a uniform blend was prepared of the following composition:—A 10, B 15, C 15,

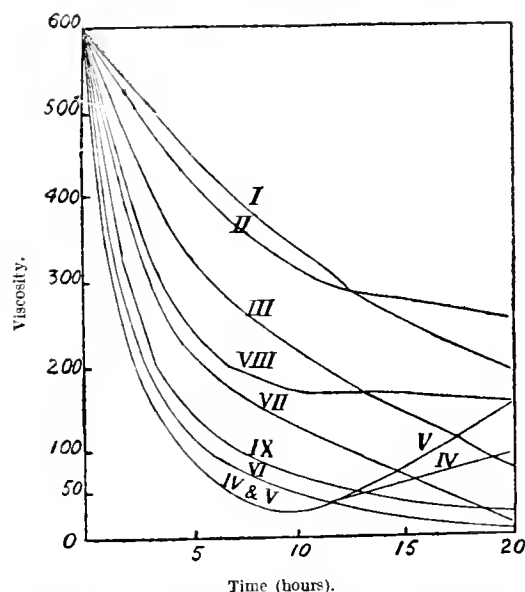
The viscosity results of samples B, C, D, E, F, and G were abnormal, and on investigation it was found that the steam pressure during the soda boiling had been too low owing to the position of the gauge on the steam supply pipe. A standard gauge was fitted to the lid of the kier and the temperature in the kier was determined by the introduction of tubes containing organic substances of known m.p. The correct pressure was then maintained and the boils repeated.

The only point of interest in the tests of the samples prepared from the individual raw materials A to G is the apparent ease of reduction of the size of the molecular aggregate in the case of the samples of sweeps and cops compared with the reduction in the case of the samples of fly. The viscosities of the materials before treatment are only approximate owing to the impurities in the samples. From the tests of the samples prepared from the blends 9—26 of the raw materials the removal of the oils is more complete with higher temperature and stronger alkaline solutions and is not affected by extending the time of the boil over ten hours. The ash content is not affected by the conditions of kieriing, but the copper number appears to be slightly reduced with the higher temperature of boiling. Pressure of kieriing up to 40 lb. does not appear to affect the yield to any extent.

The results given for viscosity are the mean of several determinations. The individual tests shown in some cases give different results, due probably to the difficulty of sampling a blend of materials

each having different viscosities. These results are shown in Graph I. below.

- GRAPH I.
- I. Atmospheric, 4% NaOH.
 - II. Atmospheric, 2% NaOH + 2.65% Na₂CO₃.
 - III. 20 lb. .. 2% NaOH.
 - IV. 20 lb. .. 4% NaOH.
 - V. 20 lb. .. 2% NaOH + 2.65% Na₂CO₃.
 - VI. 40 lb. .. 4% NaOH.
 - VII. 40 lb. .. 2% NaOH + 2.65% Na₂CO₃.
 - VIII. 40 lb. .. 1% NaOH + 4.0% Na₂CO₃.
 - IX. 40 lb. .. 2% NaOH.



These graphs clearly show the effect of the concentration of caustic soda, the temperature and the time of boiling on the viscosity of the resultant cotton.

From these results it was impossible to choose conditions for kieriing at 20 lb. pressure which could be relied upon to give a regular product equal to that of a suitable 40 lb. boil. The irregular results given by the 20 lb. boils were thought to be due to the different effects of the boils on each constituent of the blend, some of the constituents being very incompletely broken down. Repeat boils were, therefore, carried out, but the results were again quite irregular, a rise in viscosity after an initial fall during the boiling again being shown. This unexpected development seemed to be well confirmed in spite of every precaution, and it was decided to investigate more closely the change in viscosity during the progress of boiling at 20 lb. pressure. The following programme of experiments was accordingly carried out:—

Series II.

The same blend of raw materials was used as in the first series of tests, but the materials were taken

from different consignments; also the three types of spinners' sweeps were degreased before kieriing, otherwise the conditions and methods for carrying out the boils and the finishing treatments were as before. Table III. gives the results obtained under varying conditions of treatment:—

TABLE III.

Tests of purified materials.

No. of expt.	Pressure, lb. per sq. in.	Strgth. of NaOH soln., %	Time hrs.	Grease %	Ash %	Copper numb.	Solubility in 3% NaOH %	Viscosity, C.G.S. units.
1	20	4	2	0.24	1.17	0.29	2.6	26
2	20	4	6	0.25	1.05	0.33	2.9	9
3	20	4	10	0.23	1.10	0.38	3.7	33
4	20	4	12	0.20	1.00	0.30	2.8	4
5	20	4	14	0.22	0.97	0.36	2.6	76
6	20	4	16	0.18	1.07	3.42	2.7	7
7	20	4	20	0.20	0.80	0.33	2.9	15
8	20	4	25	0.20	1.08	0.30	2.9	14
9	40	3	2	0.29	1.02	0.33	3.8	27
10	40	3	4	0.26	1.06	0.27	3.0	22
11	40	3	6	0.23	1.05	0.29	3.0	32
12	40	3	8	0.23	0.96	0.34	2.8	31
13	40	3	10	0.23	1.17	0.29	3.0	22
14	40	3	15	0.23	1.14	0.33	2.6	12
15	40	3	20	0.21	1.18	0.26	2.5	17
16	20	5	2	0.21	0.99	0.18	2.5	22
17	20	5	6	0.21	0.99	0.20	2.4	12
18	20	5	10	0.21	1.06	0.24	2.8	27
19	20	5	12	0.23	0.93	0.31	2.0	18
20	20	5	14	0.23	1.06	0.37	1.7	12
21	20	5	16	0.21	0.97	0.35	1.7	16
22	20	5	20	0.19	1.10	0.37	2.0	16
23	20	5	25	0.24	0.90	0.26	2.1	20
24	20	3	2	0.22	0.94	0.20	2.0	73
25	20	3	6	0.25	0.90	0.24	3.0	85
26	20	3	10	0.25	0.90	0.26	2.0	64
27	20	3	12	0.23	0.81	0.19	2.0	92
28	20	3	14	0.21	0.88	0.13	2.1	42
29	20	3	16	0.20	0.95	0.17	2.5	47
30	20	3	20	0.26	0.90	0.20	2.0	22
31	20	3	25	0.25	1.01	0.15	2.0	29

The changes in strength of solution were very similar for each boil. From the figures it appeared that the total alkali fell by a fairly constant amount during the period of bringing the temperature to a set figure and the first hour of the boil. Throughout the remainder of the boil it remained fairly constant in each case. The initial reduction in strength was due mainly to the dilution of the solution by the condensation of the steam, and to a very small extent only to neutralisation by the impurities in the cotton. Up to the end of the first hour's boiling the strength of the caustic soda decreased and the sodium carbonate increased by a fairly constant amount. Allowing for the amount of dilution determined from the value for total alkali, the loss in caustic soda was approximately equal to the gain in sodium carbonate. Under existing working conditions with unlagged kiers the actual conditions throughout the kier cycle have been roughly calculated and the results are given in Table IV.:—

TABLE IV.

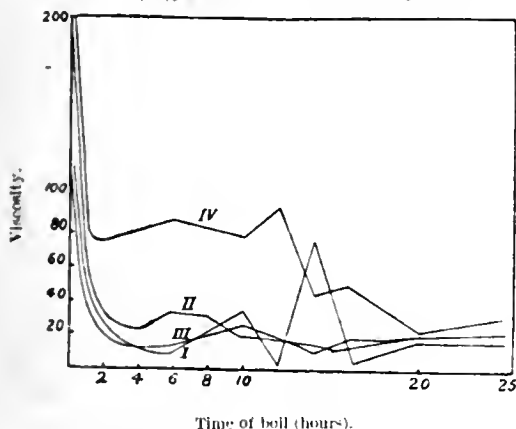
Expt No	Pressure lb	From start to end of first hour.				From end of first hour to end of 20th hour.			
		Ratio. Solution: cotton.	NaOH, %	Na ₂ CO ₃ as NaOH, %	Total as NaOH, %	Ratio. Solution: cotton.	NaOH, %	Na ₂ CO ₃ as NaOH, %	Total as NaOH, %
1-8	20	4-5.5	4.01-2.20	0.12-0.80	4.13-3.0	5.5-5.9	2.20-1.49	0.80-1.29	3.0-2.78
9-15	40	4-5.2	3.07-1.10	0.10-1.38	3.17-2.48	5.2-5.3	1.10-0.54	1.38-1.86	2.48-2.40
16-23	20	4-4.9	5.05-2.92	0.11-1.35	5.15-4.27	4.9-4.6	2.92-1.67	1.35-3.08	4.27-4.75
24-31	20	4-4.3	3.03-1.00	0.08-1.83	3.09-2.83	4.3-4.7	1.00-0.24	1.83-2.37	2.83-2.61

To ensure the presence of free caustic soda throughout the boil it is, therefore, necessary under present conditions and with the raw materials used to use a solution, the initial strength of which is not less than 3% NaOH. With the exception of the ash content the specification tests call for no comment. The figures for the ash content are slightly above the specification limit, but for economy in production and to save plant costs a series of experiments was carried out to examine as to the possibility of omitting the souring treatment altogether.

It was found that at those mills possessing plant by which the kierung liquor could be prepared from soda ash and lime separately from the kier instead of adding both soda ash and lime to the cotton in the kier, and by paying greater attention to mechanical treatment, the ash content of the purified unbleached unsoured cotton could be reduced to less than 1% which included approximately 0.20% of Na_2CO_3 formed from sodium hydroxide retained by adsorption by the cotton. Consequently permission was given to omit the souring treatment, thus removing also a possible source of objection if traces of acid were left in the purified cotton. The viscosities given in Table III. are plotted in Graph II. below:—

GRAPH II.

I. 20 lb., 4% NaOH. III. 20 lb., 5% NaOH.
II. 40 lb., 3% NaOH. IV. 20 lb., 3% NaOH.



The irregularities in the curves are probably due to the mixed nature of the materials boiled. The effect of boiling on the viscosity of each constituent of the blend was investigated by the Research Department, Woolwich, in the laboratory. The results confirmed previous large scale results from the first series of experiments indicating that the cellulose in materials of the nature of fly is more resistant to the action of alkali, other things being equal, than cellulose in normal cotton fibres.

The composition of the alkaline liquor after kierung has been carefully investigated by Dr. Denham. The full report of this work will be published later, but it is of interest to note that he finds that no decomposition of the cellulose occurred during boiling, that recovery from the liquor of any substances other than alkali was not practicable, and that the liquors could not be rendered suitable for re-use by filtration, though by subsequent work, when purer raw materials, i.e., sliver cotton, were used, the latter method was applied profitably.

In addition to the stoppage of souring referred to above, tests were carried out on samples of treated cotton from the mills to ascertain if the mild bleaching treatment given to the materials boiled at 20 lb. (or under) steam pressure affected

the viscosity of the cellulose. Table V. gives the results obtained.

TABLE V.

Sample no.	Viscosity.	
	Bleached.	Unbleached.
1	6.4	7.3
2	7.5	8.2
3	7.3	8.4
4	41.0	43.0
5	35.0	55.0
6	26.0	21.0

The viscosities having been reduced so considerably by the soda boil, the mild bleaching treatment carried out by the circulation of the cold bleaching solution was without appreciable effect on the viscosity and, as the colour of the cotton was of no consequence to the manufacture of propellants, the bleaching operation was omitted.

When this stage had been reached in the experimental work, circumstances had arisen whereby it was desirable to ascertain the most suitable conditions for boiling at 80 lb. steam pressure. A series of experiments was therefore carried out with a blend of American sweeps 10, Eastern sweeps 10, American willowed fly 35, Eastern card fly 35, American cops 5, Eastern cops 5%, 9000 lb. being used for each boil, and an initial proportion of 4 to 1 of completely causticised 3% NaOH solution.

In carrying out these tests the conditions were as in Series I with one important exception: namely that the steam supply pipes fitted to the kiers were small, in consequence of which the times taken for bringing the contents of the kier to the required temperature were very long. The times of the boils cannot therefore be taken to have any significance. After boiling, the cotton was finished according to the standard method, but in the case of Nos. 1 to 4 watering off was done with hot water. The results of the tests are given in Table VI.

TABLE VI.

Sample no.	Steam pressure, lb. per sq. in.	Time of boil, hrs	Viscosity.
1	80	2½	20.1
2	80	5	16.0
3	80	7½	8.4
4	80	10	22.5
5	40	10	27.2

The results for samples 1 to 4 are all lower, as was to be expected, than the standard 40 lb. boil, but conclusions are deferred until the later section in which the results of nitro-cotton and cordite manufacture are considered together. The two series of samples were subjected to Barrett's test with Malachite Green for the detection of ligno-cellulose, oxycellulose, etc. (*cf.* J., 1920, 81 T). Those boiled at 80 lb. pressure contained less dyed materials than samples boiled at 40 lb. pressure. This is important particularly as the blend of raw materials used in these experiments contained larger amounts of fly than the blend used in the earlier programmes. The quantity of seed husk originally present was therefore larger, but was more satisfactorily disintegrated and removed at the higher temperature.

The specification tests were uniformly satisfactory.

The experiments at the waste mulls were carried out by Mr. J. W. Black, of the Propellants Branch, Ministry of Munitions of War; Mr. F. L. Barrett, who represented the Research Department, Wool-

wich; and Mr. A. Pinkerton, Chemist of the British and Foreign Supply Association.

The preparation of the nitrocottons and the cordites was carried out by Messrs. Nobel's Explosives Company, Glasgow, at their Ardeer Factory, and they also followed up the preparation of the cottons at their own cotton waste purification mill. The examination of the nitrocottons and cordites was undertaken by the Research Department, Woolwich.

Preparation and examination of nitrocotton samples.

The nitrocottons were prepared according to the service methods followed at the Ardeer Factory. The cotton was teased, willowed, dried, and nitrated by the displacement process. The time of nitration was one hour. The nitrated cotton was boiled five times (twice for 12 hrs. and three times for 4 hrs.), each commencing with fresh cold alkaline solution containing 0.27% Na_2CO_3 . After boiling the samples were pulped, washed, and centrifuged in the usual way. Samples were taken during the boiling process and samples of the finished nitrocotton were taken from the centrifuged batches. As a control batches were also prepared from service cotton waste.

The results are given in the following tables:—

TABLE VII.
Specification tests.

Sample No.	Cotton.		Nitrocotton.		
	Pressure of boil. lb.	Soly. in E/A. %	Nitrogen (absolute). %	Organic matter insol. in acetone. %	Presence of unnitrated cotton in E/A solution.
A.	40	98.7	12.30	0.33	Nil
B.	40	98.9	11.94	0.23	Nil
E.	40	98.8	12.24	0.42	Very little
F.	40	99.0	12.15	0.13	Nil
9	Atm.	98.3	12.13	0.80	Large amount
10	Atm.	98.3	12.18	0.86	Small amount
11	Atm.	98.8	12.36	0.46	Fair amount
12	Atm.	98.9	12.19	0.80	Fair amount
13	20	98.8	12.17	0.43	Large amount
14	20	98.0	12.16	0.32	Very little
15	20	98.7	12.12	0.50	Very little
16	20	98.7	12.12	0.61	Large amount
17	20	98.9	12.08	0.36	Large amount
18	20	99.0	12.14	0.49	Fair amount
19	40	98.8	12.13	0.57	Very little
20	40	98.9	12.08	0.49	Nil
21	40	98.4	12.14	0.35	Nil
22	40	98.9	12.07	0.44	Nil
23	40	98.5	12.06	0.49	Nil
24	40	98.6	12.02	0.26	Nil
25	40	98.7	12.12	0.47	Small amount
26	40	98.6	12.13	0.34	Nil

The usual stability tests—Will, Bergmann and Junk—were normal and disclosed no difference between the various samples of nitrocotton.

The results of the specification tests were normal, the important difference between the samples being brought out in the last two columns. The blended cottons boiled at 40 lb. pressure gave nitrocottons slightly low in nitrogen, but these nitrocottons were much more free from unnitrated material than the nitrocottons prepared from cottons boiled at lower temperatures.

Although the stability tests were all normal it cannot be concluded that the different treatments given to the cotton waste do not affect the stability of the nitrocotton. It would appear possible that the same degree of stability might be produced by a shorter stabilising treatment for nitrocotton prepared from some samples of cotton waste than for nitrocotton prepared from other samples.

The determination of the viscosity was carried out by the falling sphere method (*cf.* Gibson and Jacobs, *loc. cit.*) in ether-alcohol solution. It was found necessary to carry out this test under very carefully chosen conditions in order to render the results for different samples comparable. The conditions were established by the Research Department, Woolwich, after considerable work, from which it was demonstrated that an optimum solvent composition, *i.e.*, ratio of ether to alcohol, exists for each nitrocotton (*cf.* Gibson and McCall, *J.*, 1920, 172 r). The nitrocotton dissolved in its optimum solvent gives a minimum viscosity, the variation of the composition of solvent, either by a greater proportion of alcohol to ether or *vice versa*, causing gelation with consequent increasing viscosity, and the degree of gelation increases with time. The optimum solvent is independent of the concentration of the solution, concentration and viscosity being connected, within a certain range, by the formula of Arrhenius, $\log(\text{viscosity}) = \text{constant} \times \text{concentration}$. The composition of the optimum solvent is, however, dependent upon the nitrogen content of the nitrocotton, the ether concentration increasing with increased nitrogen content.

TABLE VIII.
Viscosity determinations.

Sample	After 1st displacement. 2% soln.	After 1st 12-hour boil. 3% soln.	After 2nd 12-hour boil. 3% soln.	Finished nitrocellulose.		
				In optimum E/A solvent.		In acetone 0.5% soln.
				5% soln. η .	Solvent E/A by volume.	
A.	3.5	11.3	6.7	20.2	1.38:1	2.10
B.	8.20	19.2	6.1	33.2	1.00:1	2.28
E.	15.7	29.0	7.1	21.5	1.22:1	2.15
F.	7.9	28.1	8.8	19.4	1.22:1	2.15
9	30.5	61.7	26.1	54.3	1.22:1	3.16
10	17.2	43.3	15.8	56.7	1.13:1	3.12
11	32.6	38.6	11.4	67.5	1.22:1	2.97
12	41.1	52.1	19.4	61.6	1.22:1	3.33
13	18.2	43.0	13.8	38.7	1.22:1	2.42
14	12.7	40.0	11.3	34.5	1.50:1	2.73
15	11.7	19.4	16.7	31.5	1.38:1	2.71
16	19.8	52.7	14.7	49.3	1.08:1	2.96
17	5.6	25.5	9.1	35.6	1.33:1	3.10
18	11.5	21.3	16.1	29.8	1.22:1	2.43
				46.4	1.22:1	
19	12.2	49.9	19.0	29.0	1.22:1	2.37
				16.4	1.22:1	
20	1.9	6.2	2.4	10.6	1.22:1	1.89
				43.3	1.0:1	
21	12.5	29.6	11.3	38.8	1.08:1	2.85
				33.8	1.0:1	
22	5.6	17.1	10.0	30.6	1.22:1	2.34
				75.3	1.0:1	
23	16.5	39.8	11.0	43.4	1.22:1	2.86
				61.9	1.0:1	
24	35.8	46.7	7.8	48.0	1.13:1	2.97
				61.7	1.0:1	
25	14.0	30.2	9.2	33.2	1.22:1	2.85
				46.9	1.0:1	
26	3.0	19.9	9.0	24.9	1.22:1	2.51

In view of these results it was decided that comparison of viscosities of nitrocottons could only be made when the viscosities were determined for each nitrocotton dissolved in its optimum solvent, other conditions, *i.e.*, temperature and concentration, being equal. As the object of the whole of this work was a reduction in the consumption of solvent in propellant manufacture, it was advisable in manufacture to use the optimum composition as far as possible if the viscosity of the nitrocotton affected solvent consumption.

Hitherto acetone had been adopted almost universally as the best solvent in which to determine the viscosity of nitrocellulose, although in 1918 Messrs. Du Pont, of the United States, had changed from this solvent to ether-alcohol. It was there-

fore desirable to ascertain if the results given by solutions in acetone were comparable with those given by ether-alcohol solutions, ether-alcohol being used in the manufacture of cordite R.D.B. From the work done a direct relationship was established, and it was found that acetone-water solvent gave gelation results similar to ether-alcohol, the optimum solvent containing about 7% of water (*cf.* Masson and McCall, *Chem. Soc. Trans.*, 1920, 117, 819). From these investigations it was decided to adopt ether-alcohol as the solvent to be used in this work.

Whether the viscosity of the nitrocotton would bear any direct relationship to the viscosity of the nitrocotton-nitroglycerin dough and thus to solvent consumption, required further investigation. It was found that for solutions in ether-alcohol of nitrocotton and nitroglycerin in the proportions in which they are used in cordite R.B.D. manufacture, the optimum composition of the solvent remained the same as if nitroglycerin were absent (*cf.* Gibson and McCall, *J.*, 1920, 172 τ). The viscosity was, however, higher than for the corresponding solution of nitrocotton, but it was approximately equal to that of the nitrocotton solution if in determining the concentration nitrocotton and solvent alone were considered, disregarding the nitroglycerin present. It therefore appeared that the direct determination of the viscosity of nitrocottons alone in ether-alcohol would give satisfactory results for establishing any relationship between viscosity and solvent consumption which might exist.

TABLE IX.

Specification tests of finished nitrocotton.

Expt. No.	Cotton.	Nitrocotton		
	Pressure of boil. lb.	Soly. in E.-A.	Nitrogen (absolute) %	Organic matter insol. in acetone, %
1	80	99.7	12.22	0.11
2	80	99.8	12.10	0.22
3	80	99.6	12.26	0.30
4	80	99.7	12.20	0.24
5	40	99.5	12.19	0.20
Service batch	40	99.5	12.10	—

The results of the examination of the nitrocotton samples from Series III. are given in the following tables, special attention having been directed to the examination of the change in viscosity and stability of the nitrocotton during the boiling treatment.

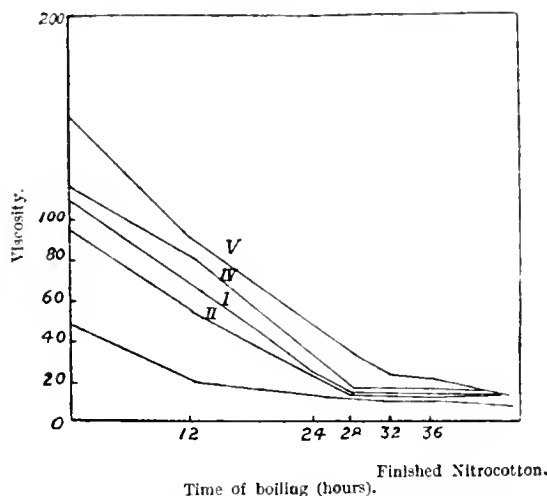
ture at which the cotton is kiered the lower the content of organic matter insoluble in acetone in the resultant nitrocotton.

The stability results given in Table X. show that the finished nitrocottons have all been reduced to conditions of equal stability. The process samples permit of no differentiation as regards the different treatments of the cotton, but as far as the Bergmann and Junk test may be taken as a reliable indication of stability, relatively little change occurs in the stability of the nitrocottons after the first four-hour boil.

Following the remarks made on the results from the experiments of Series I. as to the effect of the stabilising treatment on the viscosity, the results given in Table XI. are of importance, and have been plotted in Graph III., below.

GRAPH III.

Effect of boiling nitrocotton on its viscosity.



From this graph it is clearly indicated:—(1) That the full boiling process is effective in reducing to comparatively small proportions the initial large differences in viscosity of the various samples. (2) That reduction in viscosity is more readily effected in nitrocotton prepared from cotton boiled with caustic soda at high temperatures than at low temperatures. (3) That the viscosity is reduced to a practically constant figure after a given period of boiling. (4) That the reduction in viscosity to this constant figure is approximately directly proportional to the time of boiling and is not influenced by the quantity of acid present in the water during the boil. This is clear from the reduction in vis-

TABLE X.
Stability tests.

Dulong stabilisation—Bergmann & Junk test							Finished nitrocotton.				
Expt. No.	After 1st displacement	After 1st 12 hour.	After 2nd 12 hour.	After 1st 4 hour.	After 2nd 4 hour.	After 3rd 4 hour.	B. & J. test.	Will test.	Heat test.	Paste test.	Fume test.
1	32.9, 31.4	23.4, 22.2	1.7, 1.8	1.7, 1.9	1.55, 1.4	1.2, 1.3	0.9, 0.8	4.4	18, 20	15, 16	69
2	32.5, 30.7	20.0, 22.1	2.0, 1.8	1.8, 1.9	1.4, 1.4	1.1, 1.2	0.8, 0.8	4.2	17, 20	15, 15	60
3	32.4, 34.3	17.1, 16.7	1.8, 1.8	1.4, 1.4	1.4, 1.3	1.2, 1.3	0.9, 1.0	4.5	17, 18	15, 16	69
4	Samples exploded	9.9, 10.5	2.0, 1.8	1.6, 1.7	1.6, 1.5	1.2, 1.2	1.0, 0.9	4.6	18, 19	17, 17	60
5	41.9, 45.7	2.7, 2.5	1.5, 1.5	1.3, 1.4	1.3, 1.3	1.0, 1.0	1.0, 1.0	4.2	17, 19	16, 17	60
Service	45.0, 40.1	1.8, 2.0	1.5, 1.6	1.4, 1.3	1.2, 1.3	1.0, 0.9	0.8, 0.9	4.6	17, 18	17, 16	69

Of the results in Table IX. only those in the last column call for comment in confirming those obtained under series I. that the higher the tempera-

cosity during the first and second twelve-hour and the first four-hour boils when studied with the results given in Table XII.

TABLE XI.
Viscosity tests.

Expt. no.	During stabilisation.							Finished nitro- cotton.
	In E.-A. (1:22:1)—3% solution Nobel's.							In E.-A. (1:22:1) —5% solution.
	*1	2	3	4	5	6	7	Woolwich.
1	109	68	26	14.1	13.1	12.5	10.8	31.6
2	94	57	25	12.7	12.8	11.0	10.6	29.9
3	49	20	18	12.0	10.6	10.4	6.8	20.8
4	116	81	34	16.7	15.6	15.3	12.5	29.7
5	149	92	48	34.0	23.0	19.8	13.1	40.1
Service	105	68	26	13.7	13.3	12.8	10.9	39.0

* 1 after 1st displacement. 4 after 1st 4-hour boil
 2 .. 1st 12-hour boil. 5 .. 2nd ..
 3 .. 2nd .. 6 .. 3rd ..
 7 Finished sample.

TABLE XII.
Reaction of wash waters after each boil.

Sample no.	Congo red as indicator.	
	Acidity as H_2SO_4 .	Alkalinity as $CaCO_3$.
1.		
1st 12 hr.	0.040	—
2nd 12 hr.	—	0.0041
1st 4 hr.	—	0.0063
2nd 4 hr.	—	0.0018
3rd 4 hr.	—	0.0022
2.		
1st 12 hr.	0.028	—
2nd 12 hr.	0.0001	—
1st 4 hr.	—	0.0009
2nd 4 hr.	—	0.0009
3rd 4 hr.	—	0.0010
3.		
1st 12 hr.	0.035	—
2nd 12 hr.	0.0006	—
1st 4 hr.	—	0.0020
2nd 4 hr.	—	0.0019
3rd 4 hr.	—	0.0024
4.		
1st 12 hr.	0.038	—
2nd 12 hr.	—	0.0009
1st 4 hr.	—	0.0008
2nd 4 hr.	—	0.0009
3rd 4 hr.	—	0.0008
5.		
1st 12 hr.	0.035	—
2nd 12 hr.	0.0005	—
1st 4 hr.	—	0.0009
2nd 4 hr.	—	0.0005
3rd 4 hr.	—	0.0004
Service blend.		
1st 12 hr.	0.036	—
2nd 12 hr.	0.0007	—
1st 4 hr.	—	0.0005
2nd 4 hr.	—	0.0006
3rd 4 hr.	—	0.0006

The conclusion under (2) is important, as if stabilisation could be effected in a shorter time of boiling than is at present followed, the cotton from which the nitrocotton is prepared should be boiled at a high temperature.

The viscosity of the finished nitrocottons prepared from cotton wastes boiled at 80 lb. steam pressure are lower than those prepared from cotton wastes boiled at lower pressures.

Preparation and examination of cordite samples.

Nitroglycerin was added to the nitrocottons in the proportions of 52 to 42, and the mixture was hand-mixed, sieved, and incorporated in the usual way

in a closed mixing machine, the full quantities of ether and alcohol (1.4 pts. ether to 1 pt. alcohol) being added to the mixture in this machine. After mixing had continued for the normal time the dough was transferred to presses and pressed through dies by hydraulic pressure into cords (size 16). The routine observations made during manufacture called for no comment. The following table gives the classification made by Messrs. Nobel's as to the minimum solvent required by each sample to give a satisfactory, good, clear, and smooth cord. The full results of the stability tests of the samples of cordite with and without mineral jelly are not yet available, the initial stability tests not having been included, as they were all normal and showed no difference between the samples.

TABLE XIII.

Sample no.	Minimum solvent required—%.		
	1st nitration.	Repeat nitration.	
		1st trial.	Confirmation.
A.	59.5	—	—
B.	55.6	—	—
E.	55.6	—	—
F.	55.6	—	—
9	59.5	—	—
10	65.6	—	—
11	65.6	—	—
12	59.5	—	—
13	59.5	—	—
14	65.6	—	—
15	59.5	—	—
16	65.6	—	—
17	59.5	—	—
18	59.5	—	—
19	55.6	59.5	59.5
20	55.6	55.6	55.6
21	55.6	55.6	—
22	55.6	55.6	—
23	55.6	55.6	55.6
24	55.6	55.6	55.6
25	55.6	55.6	55.6
26	55.6	55.6	55.6
Service batch no.			
1	65.6	—	—
2	65.6	—	—
3	65.6	—	—
4	65.6	—	—
5	65.6	—	—
6	65.6	—	—
7	—	55.6	55.6
8	—	55.6	55.6

The results of these trials clearly showed that a reduction in solvent consumption could be effected by the use of a properly prepared cotton waste. The production of cordite R.D.B. varied between 1000 and 1400 tons per week throughout 1918. During this period the conditions for recovery of solvent underwent considerable improvement, but, taking the conditions existing at the time at which these results became available, the application of the results to manufacture would have resulted in a net reduction in the consumption of solvent of 80 tons of ether and 36 tons of alcohol per week, at an approximate value of £11,000.

The results actually obtained in manufacture and the consideration of the relation of solvent consumption to the viscosities of the nitrocotton and the cotton waste are further considered in the summary below.

The results given by the manufacture of cordite R.B.D. from the samples of cotton waste prepared under Series III. indicated that no further reduction in the quantity of solvent required would be possible, but the trials cannot be accepted as final.

Consideration of experimental results.

It is convenient, in considering the results given above, to subdivide this section as follows:—

(a) Empirical relations; (b) viscosity relations. The conclusions are subject to possible modification later, when the results of the stability tests of the cordite become available.

(a) *Empirical relations:*

To determine the most suitable of the treatments given to the various samples of cotton waste from the point view of economy of solvent in cordite R.D.B. manufacture, the following table has been prepared, the samples being arranged in order of merit. The conditions of manufacture of the nitro-cotton were service in each instance, so that reference to the intermediate nitro-cotton stage has been excluded from the table. The effect of the boiling treatment on the nitro-cotton is of the same nature as the effect of the soda boiling treatment on the cotton.

TABLE XIV.

Cordite.	Cotton.				No.
Minimum solvent required. %	Soda boil.				
	Pressure. lb.	Time. Hours.	Solution.		
			% NaOH.	% Na ₂ CO ₃ .	
55-6	40	10	3-0	—	B, E, F.
55-6	40	10	4-0	—	19
55-6	40	20	—	—	20
55-6	40	10	2-0	2-65	21
55-6	40	20	2-0	2-65	22
55-6	40	10	1-0	4-0	23
55-6	40	20	1-0	4-0	24
55-6	40	10	2-0	—	25
55-6	40	20	2-0	—	26
59-5	40	10	3-0	—	2
59-5	20	10	2-0	—	13
59-5	20	10	4-0	—	15
59-5	20	10	2-0	2-65	17
59-5	20	20	2-0	2-65	18
59-5	atm.	10	4-0	—	9
59-5	atm.	20	2-0	2-65	12
65-6	atm.	20	4-0	—	10
65-6	atm.	10	2-0	2-65	11
65-6	20	20	2-0	—	14
65-6	20	20	4-0	—	16

From this table it is clear that cotton wastes boiled at atmospheric and at 20 lb. pressure do not give the most satisfactory results even when the length of the boil and the concentration of the soda solution are increased. At 40 lb. pressure the samples from the individual raw materials and from the blend were satisfactory, independently of the time of boil and the concentration of alkali. Economically a ten-hour boil with the smallest quantity of caustic soda is to be preferred, but with the necessity of freeing the waste from ligneous matter it is not advisable to reduce the concentration of caustic soda below 3% at any rate for the class of raw materials which were used. It is important for the latter reason also to emphasise the necessity of a drastic soda boil in order to ensure uniformity.

(b) *Viscosity relations:*

The most suitable treatment for cotton waste can be laid down from the preceding section. As a check upon the treatment and to ensure at the mill the issue of a satisfactory product the practicability of adopting the viscosity test and fixing the maximum for viscosity is considered in the following table.

From these results it is seen that there is generally a qualitative agreement between viscosity of cotton, viscosity of nitro-cotton, and solvent consumption. On the other hand the individual results are very variable. Under simpler conditions of production, *e.g.*, the use of one class only of raw material and standard treatment, the qualitative agreement now established might be more definite. Further observations as a matter of

routine at the mills and at the propellant factories should, however, be made.

From this point of view it is of interest to study the relationships through viscosity step by step.

Raw and purified cotton.—The determination of the viscosity of the raw cotton or cotton waste is attended with considerable difficulty. The results given in Table II., samples Nos. A to G, show that in different samples the cellulose molecular aggregate is widely different and different types of cotton are differently affected by the kiering treatment. This is seen in comparing the initial and final viscosities of cotton cops and flys when each is subjected to the same kier treatment.

TABLE XV.

Minimum solvent required. %	Viscosity of nitro-cotton in optimum E.-A.		Viscosity of cotton in Schweitzer's reagent.	Experimental number.
	Before boiling. 2% sol'n.	Finished 5% sol'n.		
55-6	8-20	371-2	16	B
"	15-7	21-5	53	E
"	7-9	19-4	57	F
"	12-1	29-0	48	19
"	1-95	10-6	4	20
"	12-55	38-8	129	21
"	5-5	30-6	19	22
"	10-5	43-4	172	23
"	35-8	48-0	265	24
"	14-0	33-2	73	25
"	3-0	24-9	24	26
Mean	12-1	30-3	73	—
59-5	3-5	20-2	89	A
"	18-2	38-7	240	13
"	11-7	31-5	37	15
"	5-6	35-6	36-5	17
"	11-5	29-8	156	18
"	30-5	54-3	332	9
"	41-1	61-6	256	12
Mean	17-4	38-8	152-4	—
65-6	17-2	56-7	193	10
"	32-6	67-5	317	11
"	12-7	34-5	94	14
"	19-8	49-3	94	16
Mean	20-6	52-0	174-5	—

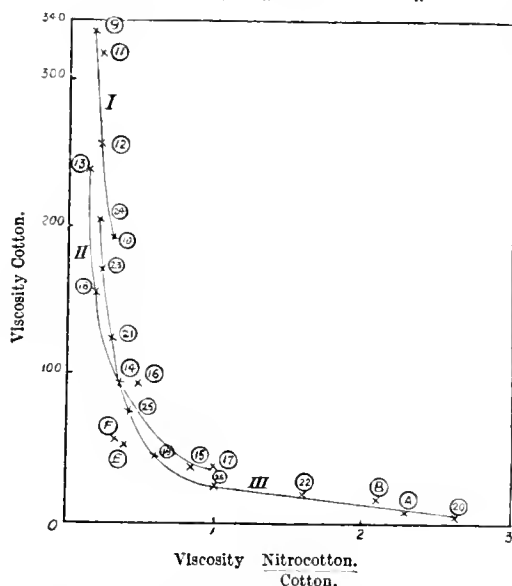
The temperature of this treatment is the most important factor in reducing the viscosity, while reduction is accelerated by caustic soda and by duration of the treatment. It has not been found possible to determine an equation showing the relation between the viscosity and the temperature, time, and alkalinity of the boiling, but for the economical production of a purified cotton of the lowest viscosity and to ensure uniformity it is necessary to adopt a high temperature boil for the minimum time and with the minimum concentration of caustic soda.

Purified cotton and nitro-cotton.—The relationship between the viscosities of the cottons and of the finished nitro-cottons was carefully considered with the Research Department, Woolwich. The effect of the boiling treatment of nitro-cotton on its viscosity during the boiling, as shown in Graph III., has been already considered. In passing through each stage of the conversion from raw cotton to finished nitro-cotton the cellulose undergoes progressive changes. Assuming that initially the cotton contains cellulose in different highly associated states, changes occur during the boiling of cotton, the rate of change being dependent chiefly on temperature, which results in producing a definite degree of dissociation which does not change under atmospheric conditions of temperature and humidity. Ultimately a state of dissociation is reached beyond which any further dissociation is not possible without disintegration of the

cellulose. The products of nitration prepared under exactly similar conditions from cellulose in different stages of dissociation retain their colloidal nature and may then be related to each other by their viscosities, given that the nitrogen contents of the nitrocottons are the same. The process of nitration, however, as well as introducing ONO_2 groups may affect the degree of dissociation, partly depending on the state of the cellulose initially. The nitration products can undergo by boiling further dissociation, the extent depending also on the degree of dissociation of the celluloses from which they were prepared. Ultimately a definite stage is reached beyond which dissociation measured by viscosity cannot be reduced without decomposition of the nitrocellulose. Plotting then the ratios, viscosities of finished nitrocottons ÷ viscosities of purified cottons, against the viscosities of the purified cottons, Dr. Masson, of the Research Department, Woolwich, obtained the curves given in Graph IV. below:

GRAPH IV.

- I. Cotton boiled at atmospheric pressure.
 II. " " " 20 lb. " "
 III. " " " 40 lb. " "



With the exception of the three points for No. 16, F, and E, all the points lie on regular curves, each curve being distinct for the temperature at which the cotton was boiled. A definite viscosity relationship thus appears to be possible between cotton and nitrocotton when all the factors contributing to the changes are taken into account. Initial differences in the viscosities of purified cottons are minimised on conversion into nitrocottons if the nitrocottons are given drastic boiling treatments. For a nitrocotton of minimum viscosity it is obviously most satisfactory to produce a cotton of low viscosity and so reduce to a minimum subsequent treatments of the nitrocotton. This becomes essential in certain industries in which the nitrocotton used does not require to be boiled unless for viscosity reasons. To obtain nitrocottons of higher or intermediate viscosity a study of the viscosities of the cottons is clearly necessary.

Nitrocotton and cordite manufacture.—A qualitative agreement is apparent from Table XV. between the solvent required for satisfactory manufacture and the viscosities of the nitrocottons contained in the cordite. It is necessary to take the average values for viscosities of groups of nitrocottons, the groups depending on the solvent

required. Individual results are not reliable, and further work is required to establish relations between the concentration of solution used in the laboratory determinations of viscosity with the solution and the consistency of cordite dough. That such relations exist would seem to be clear when the close relation between the curves in Graph IV., the solvent required in cordite manufacture, and the temperature of boiling the cotton is considered.

These results, while not definitely establishing viscosity determinations of the purified cellulose as a means of determining the suitability of the cellulose for cordite manufacture, have established to a large extent the most suitable treatments to be given to cellulose raw materials. Certain relationships have also been established as to the effect of treatments of the cellulose on its viscosity, effect of treatment of the nitrocellulose on its viscosity, and between the viscosities of cellulose and nitrocellulose.

Results from the manufacture of cordite R.D.B.

The results of the experimental work as they became available were applied to the methods of the preparation of the cotton at the mills, and with the cotton then prepared to the manufacture of nitrocotton and cordite R.D.B. at Messrs. Nobel's factories and at H.M. Factory, Gretna.

The method of preparation ultimately instituted at the mills fixed the preliminary picking and degreasing treatments necessary for certain raw materials, the blending of the materials in the proper proportions when charging the kiers, and the proper kier treatment, followed by washing, drying, and the usual mechanical treatments without either bleaching or souring.

The kierung treatment was the essential factor in determining the quality of the resultant cotton, and it was accordingly laid down that after the correct blended quantity of known weight of the cellulose materials had been charged into the kier, 30% NaOH solution, fully causticised and settled, was to be run into the kier in the proportion by weight of four parts of solution to one of cotton. Before closing the kier the air in it was to be displaced by steam, the kier then closed and the contents subjected to a boil at 40 lb. steam pressure for ten hours, the kier being slowly rotated. The hot alkaline liquor was then blown off and the cotton discharged. As the cotton issued from the kier it was immediately "drowned" by adequate sprays of cold water, so that oxidation should not take place.

Some time was taken in obtaining the necessary conditions at the mills to ensure that the treatment was followed. At each mill a technical staff was placed and scientific methods were then adopted in operating the mills. These methods covered studies of capacity of plant in relation to production of materials, efficiency, including steam production, and monthly process costing. The complete re-organisation was effected by June 1, 1918, and through the efforts of the new staff the average total cost of production fell month by month, until in October, 1918, a reduction of £13 per ton had been effected, making the comparison on an equal basis with the average total cost for the six months ending March 31, 1918.

The products supplied to Messrs. Nobel's, who used them admixed with materials of their own manufacture, also prepared according to the above method, and to Gretna, enabled these factories, the total production of which was some 800 tons of cordite R.D.B. per week, to reduce the mixed solvents they used for incorporation by 11% and 7½% respectively on the previous figure of 65 parts of solvent per 100 parts of cordite. At the same time the additional uniformity of the purified waste removed some difficulties experienced in the manu-

facture. Substantial confirmation was therefore obtained of the large-scale experimental results. Routine observations of viscosity were made on all the products at the mills, but whereas generally the viscosities were reduced to a low figure there were irregularities in the results which were not eliminated.

Experimental work on the preparation of a cotton cellulose suitable for nitrocellulose powder.

Towards the end of 1916 it was decided to manufacture nitrocellulose powder in this country and to adopt the process used by Messrs. E. I. du Pont de Nemours and Company.

Nitrocellulose powder, consisting almost entirely of nitrocellulose, has for its basic raw material prepared cellulose, generally cotton cellulose. The du Pont process for the manufacture of the powder includes the "dipper" process for the nitration of the prepared cotton, which necessitates the use of cotton of very short fibre, much shorter than the fibre of the bulk of the raw materials used in the preparation of cotton waste for the manufacture of nitrocotton for cordite R.D.B.

Representatives of the du Pont Company emphasised the importance of the nitrocellulose possessing certain physical and chemical properties, among which were:—(1) A low viscosity in acetone or ether-alcohol with, for cannon soluble nitrocellulose, the highest possible solubility in the latter solvent, to give an easy-flowing colloid and consequently low solvent consumption in manufacture. (2) A satisfactory stability, especially for high-grade nitrocellulose used for rifle nitrocellulose powder and containing 13.1–13.3% of nitrogen.

With regard to (1) the du Pont Company used as their raw material a mixture of cotton hull shavings and cotton linters of very short fibre, both of which qualities were highly contaminated with lignocellulose. Their representatives emphasised also the necessity of a drastic soda boil such as they had adopted, namely, 80 lb. steam pressure for 5 hours, to give a satisfactory and uniform product. Du Pont's determined the quality by making large scale trials of representative samples of new lots of purified material.

With regard to (2), the stability of the nitrocellulose powder partly depends on the purity of the cotton used, i.e., on its freedom from lignocellulose etc. The quality of the raw materials used by the du Pont Company necessitated a treatment which would break down these compound celluloses. This it was found was effected by a drastic soda boil, and thus the requirements under this head and under (1) were met by the one operation.

The manufacture of the nitrocellulose powder in this country was eventually undertaken at H.M. Factory, Irvine, erected and operated by Messrs. Nobel's Explosives Company. The Director of Propellant Supplies decided that the stock of raw linters, of a higher grade than those used by du Pont, in this country should be used for Irvine, and the purification of them to meet Irvine's requirements should be carried out at one of H.M. cotton waste mills.

When work commenced to ascertain a suitable treatment for the raw linters much of the experimental work on cotton waste for cordite R.D.B. had been done, and the results therefrom agreed with the experience of Messrs. du Pont.

Nitrocellulose powder being a new manufacture to this country, to be carried out in a newly erected factory as sections of it became available, no programme of experimental work such as that given in the foregoing report could be done. After preliminary trials, the results of which, owing to the conditions of manufacture, could not be taken as

more than indicative, series of large-scale experiments were put in hand under proper conditions at the mill and at Irvine.

Preparation and examination of purified linters.

The conditions of preparation as regards kiering treatment and the results of viscosity tests of the products are given in the following table.

TABLE XVI.

Note.—Ratio of solution to cotton was 4:1 in all cases.

Experimental No.	Kiering treatment.			Viscosities—2% sol'n.		
	T hrs.	P lb.	S %	Max.	Min.	Mean.
du Pont sample	5	80	—	—	—	14.7
A1	6	50	3.0	461	156	342
A2	8	"	"	202	93	149
A3	10	"	"	334	76	184
B1	6	60	"	406	113	233
B2	8	"	"	378	101	192
B3	10	"	"	86	17	40
C1	6	"	4.0	332	84	163
C2	8	"	"	195	103	143
C3	10	"	"	83	38	69
D1	6	70	1.0	577	218	418
D2	8	"	"	189	126	149
D3	10	"	"	226	139	182
E1	6	"	1.5	181	152	165
E2	8	"	"	84	29	58
E3	10	"	"	103	41	63
F1	6	"	2.0	193	69	116
F2	8	"	"	60	38	49
F3	10	"	"	39	17	28
G1	6	"	3.0	18.5	17.0	14.1
G2	8	"	"	7.9	6.7	7.7
G3	10	"	"	13.8	13.6	10.2

T=time of boil. P=pressure. S=strength of caustic soda solution.

The viscosities, except for the products from the 70-lb. treatment with 2% and 3% caustic soda, show great irregularity and are very high compared with the results obtained under the R.D.B. experimental work in which different raw materials were used. Taking the average values generally, it would appear that increase in temperature of boiling, increase in time of boiling, and increase in strength of the kiering solution each reduces viscosity. This agrees with the results of previous work. The irregularities are partly due to the irregularities in the raw linters, the difficulty of reducing their viscosity, and the difficulty of obtaining a satisfactory cuprammonium solution of material having a high viscosity. As is well known, the cuticular substance of the cotton fibre can be separated from the inter-cuticular by solution in Schweitzer's reagent, and it would seem that linters contain a relatively high proportion of cuticular cellulose which, being much more resistant to conditions influencing physical change, may be expected to cause a high viscosity. Unless completely broken down, the presence of this material may cause irregularities in the nitrocotton and incomplete gelatinisation in the powder. Further information which points to the same conclusion as regard the cuticular cellulose is given below from actual manufacturing evidence.

The products from raw linters boiled at 70 lb. pressure with 3% caustic soda solution give regular results comparable with those given by the du Pont sample of cotton and lower than those obtained by the standard treatment for R.D.B. materials. The cuprammonium solutions were also quite satisfactory and lignocellulose from the seed husk etc. was practically decomposed, which was not the case with the material from the 70 lb. 2% solution boils.

From these results, and as it was not possible with the plant available to increase the pressure beyond 70 lb., this pressure was ultimately adopted for manufacture. Throughout manufacture viscosity determinations were carried out for each batch of the material produced, the total production amounting to some 2000 tons. Of the part of this which was given the standard treatment finally adopted, a proportion was boiled for ten hours and the remainder for six hours. A summary of vis-

the cellulose in it was decomposed. The effect of this decomposition on the yield is indicated in Table XVIII. The difference in yield shown between March-May period and the October-November figures was due to greater efficiency in the purification process. This efficiency was also increasing throughout the June-September period. The effect of the use of partly disintegrated material on the viscosity of the product is also shown in Table XVII. From this table and foregoing remarks it

TABLE XVII.
Viscosities of 2% solution of cottons.

Place of test.	Time of k'ering.	All tests				Disintegrated linters used.				Sound linters used.			
		No. of tests.	Maximum.	Minimum.	Mean.	No. of tests.	Maximum.	Minimum.	Mean.	Tests over 25		Tests under 25.	
										No.	Mean.	No.	Mean.
Woolwich ..	Hours.												
Mill ..	10	80	77.7	1.7	17.4	10	72.1	10.1	26.8	8	44.8	62	12.3
Woolwich ..	10	84	81.5	1.7	20.5	10	81.5	13.9	44.0	12	41.4	62	12.7
Mill ..	6	110	37.0	1.7	45.7	14	126	13.0	67.0	32	98.3	64	14.7
Mill ..	6	124	416	1.7	56.9	14	250	10.5	76.6	56	92	56	16.9

TABLE XVIII.

Period 1918.	Raw material used.	Yield.	Raw cotton per ton product (invoiced wts.).
March } April } May }	No disintegrated material ..	68.5	1.46
June ..	Partly disintegrated material	64.5	1.55
July ..	"	63.8	1.57
August ..	"	63.4	1.58
September ..	"	58.5	1.71
October ..	No disintegrated material ..	75.8	1.32
November ..	"	85.0	1.18

would seem probable that the decomposition of the cuticular cellulose takes place much more slowly.

Preparation and examination of the nitrocottons.

The nitrocottons prepared from which results are available were pyronitrocelluloses for cannon powders. They were completely soluble in ether-alcohol and contained 12.50—12.60% nitrogen. For stabilisation the boiling treatment given was one 24-hour and four 2-hour boils with water. Of the experimental batches of cotton prepared only A3, B2, B3, G1, G2, and G3 (see Table XVI.) were fully examined. The results, together with a few others from manufacturing records, are given in the following table:—

TABLE XIX.

Cotton.		Nitrocotton.									
Sample.	Treatment.		Viscosity, 2% solution.	N %	Viscosity at 25° C. in optimum solvent.						
	P. lb.	T. hrs.			Before boiling.	After boil 1.	After boil 2.	After boil 3.	After boil 4.	After boil 5.	Finished. 4% 5%
Du Pont pyronitrocellulose.											
Service 1 ..	50	10	157, 471, 329	12.51 12.52	— Too viscous	78.0	66.3	62.4	49.6	41.5	4.1 (13) — 40.1
" 2 ..	50	10	177, 274, 162	12.52	219	50.4	—	47.8	42.0	36.9	— 31.8
" 3 ..	50	10	48, 8.0, 8.2, 6.5	12.51	81.5	23.4	17.9	15.1	15.5	15.5	— 11.9
Experimental A3 ..	50	10	47, 63, 118, 76	12.52	—	—	—	—	—	—	10.2 (34)
" B2 ..	60	8	118, 93, 281, 300	12.55	—	—	—	—	—	—	8.4 (29)
" B3 ..	60	10	8.8, 2.2, 170, 33.6	12.49	—	—	—	—	—	—	7.9 (25)
" G1 ..	70	6	10, 16.7	12.54	—	—	—	—	—	—	— 12.9
Service 4 ..	70	6	133, 109, 148	12.55	709	97	64	48.6	41.4	32.6	— 32.8
" 5 ..	70	6	37, 40, 44	12.62	273	34.1	47.5	41.2	38.6	34.7	— 19.4
" 6 ..	70	6	15	12.51	—	—	—	—	—	—	—
Special lot											
Experimental G2 ..	70	8	8, 7.4	12.54	—	—	—	—	—	—	— 16.6
" G3 ..	70	10	5, 14.4	12.60	—	—	—	—	—	—	— 14.8
R.D.B. cotton ..	40	10	2.9, 3.0	12.52	58.3	25.7	21.5	20.5	16.3	15.8	— 14.1

Cotton treatment.—P=Pressure and T=Time of soda boil.

cosity tests is given in Table XVII. During the production of this quantity a stock of raw materials which had been lying exposed to the weather had to be taken into manufacture as quickly as possible, as, owing to bacterial action, a large proportion of

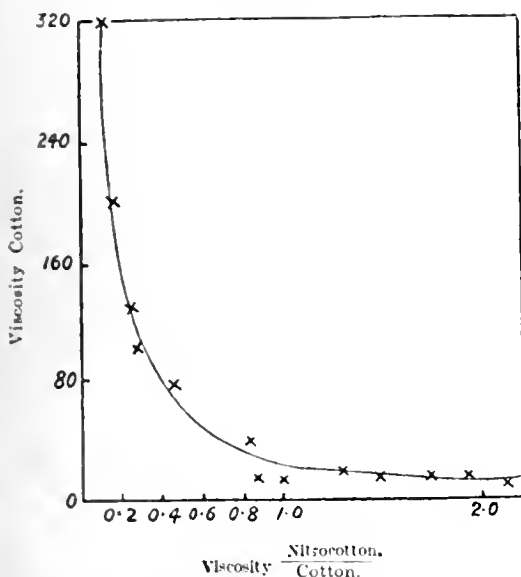
The conclusions drawn from Graph III, are confirmed by these additional results. The effect of the first boil of 24 hours on the viscosity of nitrocotton from linters is very similar to that of the two 12-hour boils on nitrocotton from R.D.B.

cotton, though in the former case there is no intermediate stage to confirm that the fall in viscosity is regular. The changes in viscosity of cellulose and nitrocellulose are essentially dependent on temperature conditions whether in the kieren of the cotton or in the nitration and stabilising processes of the nitrocotton. From this arises a point which requires further investigation and may be the cause of the slight irregularity in the results now given, namely, the effect of the hot water in contact with nitrocotton during the period of raising the contents of a boiling vat to 100° C. and during the period of running off the hot water.

Conclusion 2 from Graph III. is also confirmed by these further results, and, as will be seen later, this is of even greater importance in the manufacture of nitrocellulose powder than in the manufacture of cordite R.D.B.

Taking the mean values for the viscosity of cotton and plotting these against the ratio, viscosity of finished nitrocotton ÷ viscosity of cotton, the curve in Graph V., below, is obtained.

GRAPH V.



This curve is again similar to those given in Graph IV., but owing to the cottons in this series of experiments being boiled at different temperatures and also owing to the irregularity of their viscosities, the points lie less regularly on the curve. The latter may, however, be taken as sufficiently regular to confirm the previous conclusions, that there is a definite relationship between the viscosity of the cotton and the ultimate viscosity of the nitrocotton prepared from it. The curves generally appear to be of the form:

$$\eta_c = K.10^{(1 - \eta_n/c \div \eta_c)}$$

where η_c is the viscosity of cotton and η_n/c that of finished nitrocotton, and K a constant for each curve. Substituting the values for η_c and for η_n/c the values for $\log K$ obtained from this equation for the R.D.B. nitrocottons under Series I. and III. and for the N.C.T. nitrocottons are given in the following table.

The differences between the values of $\log K$ for Series III. and those for Series I. and linters are due to the difference in the concentration of the solution for determining η_n/c .

TABLE XX.

Series I.			Series 3.			Linters.		
Expt. No.	P.	Log K.	Expt. No.	P.	Log K.	Expt. No.	P.	Log K.
9	Atm.	1.68	1	80 lb.	0.84	81	50 lb.	1.63
10	"	1.57	2	"	0.87	82	70 "	1.37
11	"	1.71	3	"	0.87	82	50 "	1.16
12	"	1.65	4	"	0.91	102	60 "	1.30
						85	70 "	1.47
13	20 lb.	1.54	5	40 lb.	0.92	A3	50 "	1.33
14	"	1.34				133	60 "	2.03
15	"	1.42				86	70 "	1.48
16	"	1.49				G2	70 "	1.98
17	"	1.55				G3	70 "	1.48
18	"	1.38				G1	70 "	1.11
						S3	50 "	1.80
2	40 lb.	2.24						
4	"	3.06						
6	"	1.12						
8	"	1.09						
19	40 lb.	1.28						
20	"	2.25						
21	"	1.41						
22	"	1.88						
23	"	1.17						
24	"	1.54						
25	"	1.31						
26	"	1.38						

Obviously the conditions under which these experiments are carried out and the materials used cannot be expected to give accurate values for K , but even so there would seem sufficient indication that K would be found to be constant if carefully controlled experiments were done in the laboratory.

Manufacture of nitrocellulose powder.

Manufacture of "cannon nitrocellulose powder" at Irvine was for some time too beset with mechanical difficulties to allow any conclusions to be safely drawn as to the effect of the viscosity of the nitrocotton used upon the solvent required for pressing the nitrocotton-ether-alcohol dough. These difficulties were overcome, but when manufacture stopped after the Armistice it had not progressed sufficiently to enable final conclusions to be drawn. The results which were obtained from the carefully controlled manufacture of nitrocellulose powder from the experimental lots of cotton are given in the following table:—

Messrs. Nobel's comments on the manufacture of these lots were that: (1) Service batch 1, even with the larger quantity of solvent still required higher pressure and was unsatisfactory. (2) A3, B2, and B3 with the additional solvent pressed normally, but the trials were less satisfactory than was desired. (3) In dehydration the nitrocotton from G1, G2, and G3 showed considerable superiority over any previous lots. The dough from G1 when pressed gave particularly good cords of size 16 cannon powder at an average gauge pressure of 2700 lb. G2 and G3 were not quite so satisfactory as G1. Results with the special lot were similar to G1, using 106.5% of solvent. A gauge pressure of 2400–3000 lb. was used in pressing, and clean cords with extremely smooth surfaces were produced. The quality of the powder was in all respects equal to that produced at Irvine from du Pont nitrocotton.

From these results, as in the case of cordite R.D.B. manufacture, the temperature at which the cotton is kieren affects the viscosity of the purified cotton, the viscosity of the unstabilised nitrocotton and the ease of reduction in this viscosity during the boiling treatment, the viscosity of the finished nitrocotton, and most directly the pressing of the powder and the quantity of solvent required.

It is obviously desirable that the cotton should be boiled at least 70 lb. pressure with 3.0% caustic soda solution. If this be done the viscosity may

generally be reduced to below 25 C.G.S. units. In the foregoing tables instances are given where this has not been the case, and viscosities of over 100 even with the boiling pressure of 70 lb. have been obtained. No powder manufacturing trials have been done with such material, but it may reasonably be expected from all the evidence given that they would not be as satisfactory as those from cotton boiled at a high pressure and having a viscosity lower than 25 C.G.S. units.

The results from this work are therefore generally in agreement with and confirm those obtained from the cordite R.D.B. investigations.

literature to guide the work, taken comprehensively as a whole, and the necessity of devising methods of testing which have still to be perfected, render quantitative detailed conclusions impossible. On points of minor importance the deductions from the results, if not already drawn in the report itself, may readily be made:—

(a) The cellulose contained in different raw cotton cellulose materials has very different viscosities, and even in the same fibre the cellulose is probably in different states of molecular aggregation which may vary with the age or growth of the cotton.

TABLE XXI.

No.	Cotton.			N %	Nitrocotton.			Powder.
	Soda boil		Viscosity.		Viscos. ty.			
	P.	T.			In acetone 2% solution	In optimum E/A.		
						4% soln.	5% solo.	
Du Pont N C	—	—	—	12.51	27.4	4.1	(13)	100
Service batch	50	10	70 to 638	12.56	190	—	—	120
A3	50	10	47 to 118	12.52	191	10.2	(34)	108.5
B2	8	60	93 to 300	12.55	134.5	8.4	(24)	108.5
B3	10	60	2.2 to 33.6	12.49	67.3	7.9	(25)	108.5
G1	6	70	10, 16.7	12.54	101	—	12.9	106.5
G2	8	70	8, 7.4	12.54	70	—	16.6	106.5
G3	10	70	15, 14.4	12.60	75	—	14.8	106.5
Special lot	6	70	15	12.51	88.7	—	19.4	106.5

Conclusion.

Up to the present, the viscosity of cellulose, and more particularly that of its derivatives for their subsequent application, has in those industries in which it has played any part chiefly been dealt with as a specific property, certain values for which have to be obtained, rather than as a specific property depending basically on the constitution of cellulose itself. This is seen in the numerous patents which continue to be taken out, *e.g.*, in the manufacture of suitable varnishes, lacquers, and dopes, depending on empirical efforts to achieve the desired object, and thus helping, but very inconsiderably, the scientific study of the constitution of cellulose, its properties as a typical colloid substance, and the reason therefore of such properties and their changes under different conditions.

This has been particularly the case in the nitrocellulose industry. W. Will (Z. angew. Chem., 1919, 32, 133; *cf.* J. J., 1919, 479 A) has recently mentioned the same difficulties in the determination of viscosities as were encountered in this work, but could trace finally no relationship between the viscosity of the cellulose and the properties of the nitrocotton prepared from it. Further, it was concluded that conditions in the manufacture of propellant powders were more dependent on other properties than on viscosity. Attention may also be called to references in the publications of the Department of Scientific and Industrial Research on Colloid Chemistry for 1917 and 1918.

It has already been stated in Cross and Bevan in *Researches on Cellulose*, 1900–1905, that the colloidal properties of cellulose are retained and determine the properties of its derivatives obtained by chemical treatment, the example given being viscose. The importance of this to the viscose artificial silk manufacture is pointed out, and in artificial silk manufacture by the different processes, the viscosity question, chiefly of the derivatives of cellulose, has had the most important attention from the industrial point of view.

The conclusions drawn from the work under report must be essentially ones of general principle, as the lack of any earlier experimental evidence in

(b) In the purification treatment given to the cotton cellulose, the essential treatment effecting change in the cellulose is the boiling with caustic soda. The reduction in the molecular aggregate of the cellulose can be effected by heat alone, but it is accelerated by the presence of water, and more so by the presence in the water of sodium hydroxide. The changes in different raw celluloses are effected at different rates, but ultimately, if the treatment be sufficiently drastic (the temperature to which the cotton is subjected being the essential factor), the product from all sources of cotton is essentially the same, beyond which further changes may possibly be only effected by chemical changes. The products consist of normal resistant cellulose retaining its properties as a colloid.

(c) The cellulose aggregate retains its properties as a colloid throughout nitration, and governs the colloidal properties of the ester. The introduction of nitro groups into the cellulose aggregate may or may not change the aggregate, according to the conditions of nitration, but they render it capable of further modification, depending on the cellulose aggregate, and again brought about by heating the nitrocotton at different temperatures, the change being accelerated by the presence of water. Ultimately, as in the case of cellulose, the product undergoes no further change unless accompanied also by chemical change.

From the graphs showing the changes of nitrocelluloses during the boiling process the product reached in each case would seem to be an ultimate nitrocellulose.

The relation between the viscosity of the ultimate nitrocellulose from each cotton and the viscosity of the cellulose from which it was prepared is definite, so that for nitrocotton of desired viscosity the nature of the raw cellulose and the treatments to be given to it and to the nitrocotton can be determined.

(d) The properties of the “gel” formed from nitrocellulose, with or without a gelatiniser, *e.g.*, nitroglycerin, by means of solvents of the nitrocellulose, depend essentially on the physical properties of the nitrocellulose. The properties of the “gel”

determine methods and conditions of manufacture and the properties of the finished product. A qualitative agreement only has been established so far between the viscosity of the nitrocellulose and the solvent required for its satisfactory conversion into propellant powders. This qualitative relationship is of practical value, and under more accurate conditions of determination (than obtains actually in propellant powder manufacture) of the solvent required the relationship might be shown to be quantitative. The concentration of the nitrocellulose solvent "solution" during pressing of the dough is approximately 60%. For solutions of lower concentration such as obtain in other manufactures, the relation between the viscosity of a 5% solution of nitrocellulose in ether-alcohol and the quantity of solvent required in manufacture, other conditions being equal, should be more readily obtainable. It is to be understood that the solvent required in manufacture is of the optimum composition.

(e) The results of the stability trials and of the examination of the degree of gelatinisation of the powders are not yet available and, beyond general observations in manufacture, no direct tests were carried out to trace the relationship through viscosity to the other physical properties of the finished products.

(f) Other conditions being equal, i.e., the intermediate stages in manufacture, the operations in the conversion of the nitrated derivatives of cellulose into finished product, which depend on the colloidal nature of the derivative, are directly influenced by the properties of the cellulose as a colloid from which the derivatives are prepared. This influence may be traced through viscosity measurements, but a more direct relationship is apparent between the operations and the temperature to which the cellulose has been subjected in the purification treatment of the raw material. From a practical point of view, to produce the finished product as economically as possible and having the desired properties, the nature of the raw cellulosic material and the treatment given to it in purification are the fundamental factors. Subsequent operations may then be simplified and, combined with the treatment of the raw cellulose, any required properties in the derivative may be obtained.

(g) It is now perhaps permissible to infer generally that in the production of any article having cellulose as its essential base and depending on colloidal properties the above conclusions apply to a more or less greater extent.

In these conclusions some references have been made to the nature of the changes in the cellulose aggregates which lead to further considerations of the constitution of cellulose. The cellulose aggregates in nature vary widely, but they may be reduced by the action of heat, ultimate degrees of dissociation being reached according to the temperature, without the introduction, however, of any chemical change modifying the configuration in regard to esterification. Nitration may cause association, but it is clear that after nitration the aggregate may be further reduced to an ultimate degree, similarly without causing any chemical change in the nitrocellulose. It may therefore be concluded that the definite configuration of the reacting unit remains constant throughout the various dissociations. It is clear that in further work on these lines to obtain the ultimate cellulose reacting unit, either by comparison through similar groups of esters or by the comparison through different esters, the cellulose used as the starting point should be the same or in the same degree of dissociation, preferably in the lowest state, as deter-

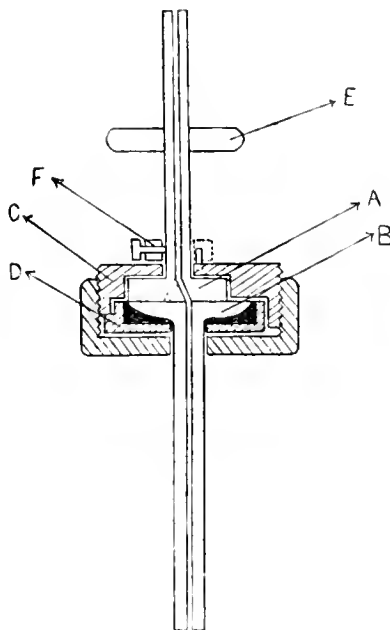
mined by its viscosity in a suitable concentration of cuprammonium solution.

In conclusion the author wishes to express his thanks to Sir F. L. Nathan, Mr. William Macnab, Messrs. Nobel's Explosives Co., Ltd., Dr. W. H. Gibson, Mr. P. Tainsh, and Mr. J. W. Black for the assistance which they have given him in carrying out the work, in compiling the report, and in its publication.

A METAL-GLASS TAP.

BY WILLIAM EDWARD GARNER.

McKelvey and Taylor have recently (*J. Amer. Chem. Soc.*, 1920, 42, 1364) given details of glass-to-metal joints used by the Bureau of Standards, U.S.A. A glass-metal tap of a different type from those described in the above paper has been constructed by the author and used with a mercury-in-glass manometer for the measurement of pressures between 2 and 10 atm. The diagram shows this tap in section. The joint is made between a metal disc, A, and a larger glass disc, B; A is attached



to a steel capillary tube, and B is fused to a glass capillary tube. A hole is drilled centrally through the glass disc and a similar hole drilled obliquely through the metal disc to the interior of the capillary tube. Both discs are placed in brass cells, C and D, which are keyed into one another when in position. The glass disc is fastened centrally into its cell by means of a cement composed of glycerin, litharge, and sodium silicate, and the exposed glass surface is carefully ground. The smaller metal disc can be revolved freely in its cell about an axis parallel to, but not in the same line as the axis of the glass capillary tube. The distance between the two axes is determined by the obliquity of the hole in the metal disc. The glass and steel surfaces are held together by means of a cap, which screws on to C. The metal and glass surfaces are carefully ground in position with fine carborundum, and in order to make a gas-tight joint, rubber grease is used as a lubricant. By revolving the metal surface on the fixed glass surface, the two holes can be

made to coincide (as in the diagram) and connexion established between the gas in the two capillary tubes. This only occurs in one position, and further movement in either direction closes the tap. The tap is opened and closed by means of a strip of metal, E, which is soldered to the steel tube, and the open and closed positions are controlled by a collar and pin, F.

The glass disc may be made by opening out the end of a capillary tube into a funnel and pressing down the hot glass on a strip of asbestos. This method has the advantage of avoiding the necessity for drilling glass which may be in a state of strain.

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THE DISTRIBUTION OF ENZYMES AND PROTEINS IN THE ENDOSPERM OF THE WHEAT BERRY.

(ADDENDUM.)

BY F. J. MARTIN.

(See J., 1920, 327 T.)

The author has supplied the following note to explain more fully how the various samples of flour were obtained:—

"The wheat was conditioned to the hardness of good average English, and the Humphries process

of spraying stocks was employed. The extraction of flour was about 70—72%.

"In the mill in which the wheat was ground a "Four-break" system is employed, the general principle of which is as follows:—The wheat is passed through the First break and the flour and semolinas (pieces of endosperm) produced are separated out; the remainder, which consists of the broken husk with a large amount of endosperm attached, goes to the Second break where a similar operation is performed. The material that then goes to the Third break has less endosperm attached to the husk, and subsequently the Fourth break removes the last scrapings of endosperm from the bran. The reductions are lettered alphabetically from A . . . N, A, B . . . denoting the highest reductions (*i.e.*, from the beginning of the mill) and . . . M, N the lowest reductions. The semolinas produced from the First break go to the highest and those from the Fourth break to the lowest reductions: the semolinas from the intermediate breaks go to the intermediate reductions.

"Examination of the stocks going to the various reductions shows that the higher reductions are comparatively free from husk, and, therefore, of particles near the cortex, while the lower reductions have a larger proportion of particles with pieces of husk attached, showing that they are derived from near the outside of the berry."

I.—GENERAL; PLANT; MACHINERY.

Mixing calculations. A. B. Helbig. Chem.-Zeit., 1919, 43, 786.

THE author gives formulae for calculating the amounts of raw materials, each containing up to four components, required to give a product of definite composition.

PATENTS.

Separating a salt from its solution by evaporation; Process and apparatus for —. P. Piccard, Geneva, Switzerland. Eng. Pat. 132,771, 15.5.19. (Appl. 12,292/19.) Int. Conv., 14.9.18.

IN the method of evaporation where a liquid is heated under pressure without evaporation in reheaters and then evaporated by reduction of pressure in the evaporator proper, the jet of heated liquid entering the evaporator is caused to impinge upon a solid obstruction upon which crystals will be deposited that will be coarser than those formed in a body of comparatively still liquor. The obstruction may preferably take the form of a screen which rotates against a scraper or is fixed and scraped by a rotating scraper.—B. M. V.

Evaporating liquids; Method of and apparatus for —. O. Carr, Asheville, N.C. U.S. Pat. 1,321,362, 11.11.19. Appl., 24.2.15.

A CHAMBER is provided with a horizontal pipe extending longitudinally along the bottom and provided with openings of variable area along its upper side. The openings increase in area towards the end remote from the inlet. Liquid to be evaporated is sprayed horizontally into the chamber above the perforated pipe, and hot air is delivered through the pipe to meet the liquid spray at right angles. The hot-air blast is sufficiently strong to carry the solids and vapour upwards to an outlet pipe at the top.—W. F. F.

Furnaces. H. Wade, London. From Fours et Procédés Mathy Soc. Anon., Liège, Belgium. Eng. Pat. 134,169, 3.5.19. (Appl. 11,113/19.)

A FURNACE is provided with alternative means of firing. A gas producer placed at one end of the furnace allows the use of producer gas, which is mixed with air at the top of the producer before it enters the furnace chamber. An arch built part way across the producer near the top forms a combustion space into which liquid fuel may be injected through the wall of the producer. A flameless combustion burner forms a third alternative, and is arranged by filling a conduit between the producer and furnace with porous refractory material and injecting a mixture of gas and air. Each of these arrangements may be used independently or to assist one another, and heated air may be used for combustion by conveying it from the regenerator to the point of combustion.

—C. A. K.

Furnaces and like apparatus for producing hot air for drying and other purposes. H. Wade, London. From Schlatter, Frorath & Co., Zurich, Switzerland. Eng. Pat. 135,332, 6.12.18. (Appl. 20,298/18.)

SEE U.S. Pat. 1,301,324 of 1919; this J., 1919, 490 A. The filter consists of several superposed layers of broken refractory brick or other fireproof, non-sintering material having catalytic properties, the pieces forming the layers being smaller towards the top. The material is heated to a high temperature, whereby there are effected not only mechanical retention of dust and chemical combustion of sulphur, but also combustion of soot and smoke.

Heating-furnace. B. D. Gray and E. W. Ruben-came, jun., Philadelphia, Pa., Assignors to The Hess-Bright Manufacturing Co. U.S. Pat. 1,321,098, 11.11.19. Appl., 6.3.17.

THE heating chamber is formed longitudinally in the furnace, and is closed at the top and ends. The articles to be heated are inserted transversely into the heating chamber through openings in the wall, and the heating gases are admitted at the rear end of the chamber through a number of vertical pipes terminating near the top of the chamber. The hot gas passes over the articles, and then out through flues leading downward and towards the rear of the chamber.—W. F. F.

Muffle furnace. W. G. Zetsche, Chicago, Ill. U.S. Pat. 1,322,214, 18.11.19. Appl., 28.2.19.

THE furnace casing is provided with fuel-feed inlets at one end, and an outlet for combustion products at the other end. A number of muffles are mounted so as to span the furnace space transversely, with one end projecting through the furnace wall. The muffles are spaced apart and are heated successively by the hot gases. The open end of each muffle is provided with a closing plug, and valve-controlled openings are provided in the furnace walls to admit air for regulating the temperature.—W. F. F.

Shaft kiln with automatic emptying device. G. Polysius, Dessau. Ger. Pat. 314,308, 20.6.16.

A SHAFT kiln is provided with an automatic emptying device and dampers arranged in succession in the discharge shoot so as to form two chambers, the lower one being connected with a fan which blows the dust into a dust chamber or supplies the kiln with air under pressure. The variations in the air pressure in other shaft kilns are avoided, as the outward passage of air under pressure is impossible, and the exhaustor does not draw air directly from the kiln. The use of several dampers also reduces the loss of heat from the kiln, as the heat in the cooling material may be used to warm the ingoing air.—A. B. S.

Air or gas; Apparatus for treating and cooling —. Heenan and Froude, Ltd., and L. Cruyt, Manchester. Eng. Pat. 134,256, 27.8.18. (Appl. 13,905/18.)

METALLIC wool (in which term is included wire, turnings, or any metallic material that will produce a spongy or porous mass) supported between plates of expanded metal, wire gauze, or the like, is built up into screens or drums which are kept wet by means of liquid sprays or by rotating them in a bath of liquid. The blades or vanes of the apparatus described in Eng. Pat. 109,648 (this J., 1917, 1171) may be made of metallic wool held in place, for example, by perforated sheets.—B. M. V.

Air or gases; Apparatus for filtering or otherwise treating —. Heenan and Froude, Ltd., and L. Cruyt, Manchester. Eng. Pat. 134,873, 8.10.18. (Appl. 16,318/18.)

AIR or gas is passed over non-perforated battles placed at an angle to the direction of flow, and formed of or covered with hygroscopic material such as felt, wool, flannel, or the like.—W. F. F.

Solids from gases; Apparatus for removing and reclaiming —. E. J. Sweetland, Montclair, N.J., Assignor to United Filters Corporation. U.S. Pat. 1,321,490, 11.11.19. Appl., 4.8.15.

GAS under pressure containing solids in suspension is passed outward through a tubular filtering member which intercepts the solids. An annular nozzle slides longitudinally on the tubular filter, and a current of gas may be forced from the nozzle

inwards through the filter at successive zones to detach the solid material.—W. F. F.

Drying or heating apparatus of continuous type. Dressler Tunnel Ovens, Ltd., and J. Williamson, Stoke-on-Trent. Eng. Pat. 134,332, 1.11.18. (Appl. 17,895/18.)

THE goods to be heated or dried are moved slowly forward on a carrier through a chamber filled with air heated by a heater situated in another chamber below the first. In the main or middle part of the furnace the heater is preferably heated by the combustion of gas or other fuel inside it, and the main movement of the air which conveys the heat to the goods is upward from the heater, among the goods, and down through side passages to the bottom of the heater, though a small quantity may be drawn off by a chimney or other draught to produce a slow longitudinal movement as well. In the tail end of the furnace the lower chamber may be omitted and air from outside allowed to pass through the goods to cool them, and the air thus preheated used for combustion in and/or circulation round the main part of the furnace. When used as a dryer, an extension at the feed end of the chamber is provided, so that most of the moisture can be removed from the goods at a low temperature. The heater for this part is preferably a nest of tubes carrying the products of combustion from the main heater, while the air which transfers the heat does not circulate, but after rising among the goods passes out of the furnace.—B. M. V.

Dryer. W. E. Prindle, London, Ohio, Assignor to The Buckeye Dryer Co., Columbus, Ohio. U.S. Pat. 1,321,332, 11.11.19. Appl., 30.4.17.

A ROTARY horizontal shell is provided with sets of longitudinal compartments spaced apart lengthwise of the shell. Each compartment has lifting blades, the number of blades increasing towards the discharge end of the shell.—W. F. F.

Cooling liquids; Apparatus for —. J. I. Thornycroft and Co., Ltd., and T. Thornycroft, London. Eng. Pat. 134,271, 21.10.18. (Appl. 17,163/18.)

A COILED double tube (the two tubes having a common axis) is placed in a tank and the liquid to be cooled passes first through the inner tube and then into the tank, which thus can serve as a reservoir for cooled liquid as well as providing additional cooling surface. The cooling liquid, though supplied from and exhausted to the exterior of the tank, is circulated through the outer tube by means of a centrifugal pump contained within the tank. The pump may be driven by external power or by a small turbine wheel which is rotated by the liquid which is being cooled as this passes from the inner tube to the outer tank.—B. M. V.

Refrigerating apparatus. H. Southon, London. Eng. Pat. 134,348, 7.11.18. (Appl. 18,237/18.)

A U-TUBE of fused silica, glass, or similar material is partially filled with dilute sulphuric acid (rather more than half acid by weight), the air is then exhausted, and the tube hermetically sealed. Most of the acid is tilted into one limb of the tube, which is then held substantially horizontal, and the limb containing the acid is gently heated, while the other limb is kept cool. Surface evaporation from the acid takes place (ebullition must not be permitted) causing concentration of the acid in one limb and condensation of water in the other. When the acid is sufficiently concentrated it is cooled, and the consequent absorption of water vapour causes evaporation in the other limb, which will now serve as a refrigerator. In order to prevent formation of ice in this limb, a small quantity of acid is purposely left there.—B. M. V.

Artificial-refrigeration method and apparatus. C. W. Miles, Anderson Township, Ohio. U.S. Pat. 1,321,230, 11.11.19. Appl., 11.11.15.

GAS is withdrawn from the refrigerator at low pressure and then liquefied in a condenser at a higher pressure. The liquefied gas is separated, the lower layer of it is pre-cooled by means of the cold, low-pressure gas from the refrigerator, and is separated from the unliquefied gas by a relatively warmer layer. The pre-cooled liquefied gas is then passed into the refrigerator.—W. F. F.

Retorts and stills. J. Prentice, East Riggs. Eng. Pat. 134,406, 9.12.18. (Appl. 20,447/18.)

A RETORT is made with a detachable bottom which can be removed upwards through the retort without removal of any of the brickwork setting. The lower end of the main body of the retort has an inwardly-directed flange which forms a seat for a corresponding outwardly-directed flange on the bottom of the still. If the bottom is provided with an outlet spout, only a few bricks have to be removed round the spout sufficient to allow the bottom to be tilted before being lifted.—B. M. V.

Distilling apparatus. O. S. Sleeper, Assignor to Buffalo Foundry and Machine Co., Buffalo, N.Y. U.S. Pat. 1,321,133, 11.11.19. Appl., 28.1.18.

THE apparatus comprises a retort, condenser, receiver, and separator, connected in series. The separator comprises a vertical chamber having its inlet and outlet on opposite sides at the top, while a baffle extends vertically downwards from the top between the inlet and outlet, terminating at a point about one-third of the height from the bottom. An outlet is provided at the bottom of the separator and is connected with an exhausting device.—W. F. F.

Filter-press plates. W. O. Mason, Leeds. Eng. Pat. 134,415, 9.12.18. (Appl. 21,231/18.)

A FILTER-PRESS plate consists of a wooden frame fitted with a fluted panel of reinforced glass.—B. M. V.

Mixing liquid, semi-liquid, or viscid materials; Machine for —. A. Sonsthagen, London. Eng. Pat. 134,451, 8.2.19. (Appl. 3117/19.)

IN a mixing machine consisting of a horizontal cylinder with two sets of opposing helical blades, one set of which gives a rather greater forward impulse than the other, the outlet is arranged in such a way that it can be swung in any direction, so that, if desired, the material may be delivered at a higher level than the mixing cylinder, thus causing the material to fill the apparatus completely and exclude all air. When admission of air is desirable the outlet may be swung downwards, air then entering through a slit or series of holes in the top of the cylinder, which may be covered with a perforated or non-perforated cover according to whether air is to be admitted or not.—B. M. V.

Grinding mills; Disc and like —. J. R. Torrance, Bitton. Eng. Pat. 134,618, 5.11.18. (Appl. 18,127/18.)

IN order to prevent scoring and uneven wear of flat discs in grinding mills, the shaft of the rotating disc is journaled in a sleeve, the outer perimeter of which is concentric with the axis of the mill, whilst the inner perimeter is slightly eccentric, so that in addition to the rotation an orbital motion is imparted to the disc. The eccentric sleeve is driven by a worm, which in turn is driven by a pair or train of pinions from the same main shaft that gives the rotary motion to the disc shaft by means of bevel wheels. Since the bevel wheel driving the shaft must be concentric with the mill and eccentric with the shaft, it is provided with internal

slots that engage with external projections on the shaft. When grinding liquid or pasty materials a scraper may be arranged to scrape the edge of the rotating disc by following its lateral but not its rotating movement, or it may be firmly attached to the rotating eccentric sleeve, so as slowly to sweep the annular collecting chamber in the fixed outer casing. In some cases it may be desirable to give a lateral movement to the companion disc, which is usually fixed. The fineness of grinding is regulated by a screw-adjusted lever pressing through a ball thrust bearing on to the end of the disc shaft.

—B. M. V.

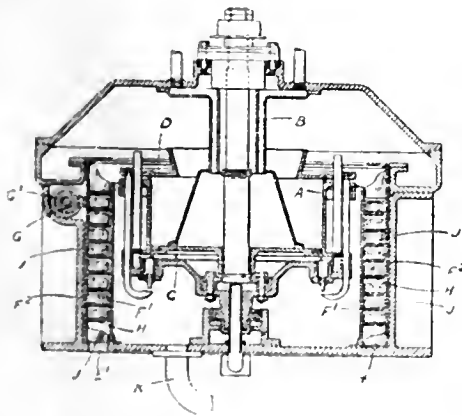
Centrifugal extractors. Safety control devices for centrifugal dryers. Centrifugal drying apparatus. L. W. Gould, Birmingham. From American Laundry Machinery Co., Cincinnati, U.S.A. Eng. Pats. (a) 134,654, (n) 134,799, and (c) 134,800, 27.11.18. (Appls. 19,532/18, 18,782/19, and 18,783/19.)

A CENTRIFUGAL extractor is provided with a double basket; the inner basket can be lifted vertically for the easier discharge of the dried goods. A belt driven from an extension of the loose pulley of the machine provides power for lifting and lowering the inner basket by means of suitable gearing. A large non-rotating cover which rises with the inner basket, and a device which can be placed close up to the raised basket and used as table for the dried goods are also provided. To ensure safe operation mechanical interlocks are provided, so that:—(a) The inner basket cannot be raised or lowered when the machine is rotating; (a) the raising and lowering mechanism cannot be operated nor the cover raised unless the brake is on and the driving belt on the loose pulley; the driving belt cannot be put on the fast pulley (for rotation) when either the basket or cover is raised, nor can the inner basket be lifted or lowered when the cover is raised; and the inner basket cannot be lowered without first moving the movable table out of the way; (c) the raising and lowering of the inner basket is restricted within definite limits.

—B. M. V.

Separators; Centrifugal —. R. A. Sturgeon, Southsea. Eng. Pat. 134,966, 22.11.18. (Appl. 19,233/18.)

LIQUID to be treated is supplied through the annular passage, B, to the centrifuge, A, which is provided with a piston, C, adapted to be raised by



the pressure produced by centrifugal force in a liquid admitted below the piston as in Eng. Pat. 24,038/13 (this J., 1915, 263). The cover, D, is normally retained by centrifugal weights, but is lifted when the solid material thrown against the wall, A, exerts sufficient pressure against it. The solid

material is broken up and forced through the annular chamber, F, the inner wall of which, F¹, is stationary, whilst the outer wall, F², is slowly rotated by worm gear, G, G¹. A series of horizontal perforated platforms, H, are attached to the wall, F¹, and a series of radial fins or scrapers, J, are attached to the wall, F². Hot air or gas is introduced through the pipe, K, and is transferred by centrifugal force into the annular chamber, F, to dry the solid material. The material is then broken up and forced through the perforated platforms in succession by the rotating scrapers, J, and the dried material is finally discharged through the apertures, E¹. In a modification the platforms, H, are alternately inclined in opposite directions and attached to both walls, F¹ and F². Scrapers are also attached to both walls, and are made with inclined edges to fit the spaces between the platforms.—W. F. F.

Separator; Centrifugal —. Automatic control for centrifugal separators. G. M. Bacon, Salt Lake City, Utah. U.S. Pats. 1,321,353 and 1,321,351, 11.11.19. Appl., 19.2 and 6.1.17.

THE rotary separating drum is provided with valves in its periphery to control the discharge of material. The valves are operated by means within the drum which are adapted to be more or less retarded in movement by the density of the material in the drum.—W. F. F.

Centrifugal concentrator. C. Schifferle, Marysville, Cal. U.S. Pat. 1,322,139, 18.11.19. Appl., 14.3.18.

THE basket of a centrifugal machine is divided into three annular compartments, the material under treatment being fed to the middle and largest one, the dressed products overflowing at the top and bottom edges. Liquid is supplied to the innermost compartment (which may be the hollow shaft), and passes through passages in the bottom of the basket to the outermost compartment. The outermost compartment communicates by means of numerous passages with the layer of material under treatment, so that liquid can be supplied to the material without there being any considerable relative circumferential motion between the two. The passages are formed in the shape of elbows, so that as long as there is an inward current of liquid none of the material under treatment will pass outwards.—B. M. V.

Gases; Process of using liquefied — for driving Motors etc. A. Willhelmi, Berlin. Ger. Pat. 312,879, 15.8.16.

CARTRIDGES containing absorbent material are saturated with liquefied gas and placed in closed pressure-containers. By the evaporation of the liquid a regulated supply of gas is rendered available for driving motors etc., or for chemical, metallurgical, or pyrotechnical purposes.—C. A. M.

Gaseous mixtures used in the liquefaction and separation of gases; Process of preventing the contamination of — with lubricants. R. Mewes, Berlin. Ger. Pat. 313,800, 6.2.14.

THE gaseous mixtures used in the liquefaction and separation of gases are liable to become contaminated with lubricants derived from the compressors, expansion vessels, and vacuum pumps of the machine. This is prevented by using a solid lubricant such as paraffin wax, stearine, glue, etc., in admixture with fine graphite.—C. A. M.

Absorbed gases; Separation of — from water. C. Hülsmeier, Düsseldorf-Gratenburg. Ger. Pat. 313,612, 4.8.17. Addition to Ger. Pat. 288,158 of 1913 (this J., 1916, 435).

THE waste from the manufacture of wired (reinforced) glass is ground in order to recover the

glass, the iron wire being removed by magnetic separators. The wire is fine and has a rough surface, and is a very efficient material for liberating absorbed gases from water. It is usually contaminated with powdered glass, but this is not a disadvantage.—W. P.

Boots for use by furnacemen, molten metal workers, and the like; Construction of —. H. E. Gammon, Swansea. Eng. Pat. 134,742, 18.3.19. (Appl. 6736/19.)

THE ordinary fixed vamp of the boot is protected by a detachable vamp, with a ventilating space between the two. A fireproof sole is fixed to the clump sole by hob-nails.

Separating liquids from vapours [in evaporators and stills]; Devices for —. N. A. Holmer, New York. Eng. Pat. 125,939, 28.2.19. (Appl. 5044/19.) Int. Conv., 25.4.18.

SEE U.S. Pat. 1,304,681 of 1919; this J., 1919, 562 A.

Distilling apparatus. U.S. Pat. 1,321,210. See VII.

Anhydrous ammonia. U.S. Pat. 1,322,251. See VII.

Condensing and purifying vapours. Ger. Pat. 314,293. See VII.

Electrical treatment of gases. U.S. Pat. 1,319,706. See XI.

Grinding machines. Eng. Pat. 134,268. See XIV.

Granulating pasty materials. Eng. Pat. 134,764. See XIXA.

Drying. U.S. Pat. 1,318,931. See XIXA.

Pyrometer. Eng. Pat. 134,498. See XXIII.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Water-gas. J. S. Mc Nicol, Scottish Junior Gas Assoc., Nov. 22, 1919. Gas J., 1919, 148, 493—495.

A YEAR'S work is described with an installation of three complete sets of Humphreys and Glasgow's carburetted water-gas plant at the Temple works of the Glasgow Corporation. The plant is capable of producing 3 million cub. ft. of purified 16-candle power gas per day, using about $2\frac{3}{4}$ galls. of oil per 1000 cub. ft. Trouble was experienced owing to the clinker accumulating on the generator walls gradually diminishing the day's make. Its formation was reduced by using a short cycle of 5 mins., $1\frac{1}{2}$ min. blow and $3\frac{1}{2}$ min. run, with blast at $17\frac{1}{2}$ in. and steam at 28 lb. It was found most economical to shut off a generator after the make dropped to 700,000 cub. ft., and to cut out the clinker by hand. The trouble was probably due to the high ash content of the coke. The average composition of the gas was: CO, 40.2%; H₂, 46.2%; CH₄, 2.1%; CO₂, 4.5%; N₂, 7%. The calorific value was about 300 B.Th.U. gross. The yield of gas was 44,800 cub. ft. per ton of coke, the steam used being about 30 lb. per 1000 cub. ft.—W. P.

Inerts; Effect of — upon the thermal efficiency in the use of gas. J. W. Wood, Midland Junior Gas Assoc., Dec. 4, 1919. Gas World, 1919, 71, 451—453, 476—477.

THE thermal efficiency of a gas must be referred to some particular operation and to some particular form of appliance or burner, though the best results are not obtained with the same appliance with all gases. A gas with a lower maximum efficiency may prove more useful in practice if it has a greater

working range over which this efficiency is maintained. The addition of "inerts" results in an increase in specific gravity of the gas, and a greater volume of gas must be passed to obtain the same heat value. At the same time less air is required for complete combustion, and aeration is improved. The decrease in flame temperature is small in comparison with the volume of inert gas added, on account of the large percentage of nitrogen already present in the air in which the gas has to burn, and the small change in volume of the gas-air mixture for complete combustion (this J., 1919, 707 A). Provided the border line between permissible practice and prohibitive qualities is not reached, for all practical purposes carbon dioxide may be treated as nitrogen. It is possible to add 25% of "inerts" to coal gas without depreciating the performance of the gas so badly as to make it commercially impracticable. The addition of air to coal gas is similar in effect to the addition of nitrogen, but in case of inefficient mixing local irregularities may occur in the gas mixture, parts of which may reach the explosion danger limit.—W. P.

Motor benzol; Production of — in medium and small gasworks. Steding. J. Gasbeleucht., 1919, 62, 635—636.

THE author advises the installation of benzol-washing processes in small gasworks. The calorific value of the gas is thereby reduced (about 6% for the removal of 30 grms. of benzol per cub. m. of gas), but the deposition of carbon by the burning gas is diminished. The costs of plant, materials, etc., are examined, together with the prices obtainable for the products, the clear profit on a year's working being greater than the cost of plant. A yield of 60,000 kilos. of benzol is estimated per year's production of 3 million cub. m. gas (20 grms. per cub. m.).—W. P.

Acetylene, ethylene, and benzene; Gas-analytical separation of —. W. D. Treadwell and F. A. Tauber., Helv. Chim. Acta, 1919, 2, 601—607.

THE quantitative absorption of acetylene from mixtures containing it, together with ethylene and benzene vapour, is conveniently effected with a solution of mercuric cyanide (20 grms.) in 2N sodium hydroxide solution (100 c.c.); the gas is shaken with the mixture (5 c.c.) for 1-2 minutes, at the end of which the acetylene is completely absorbed, whilst ethylene and benzene vapour are unaffected. A solution of mercuric nitrate (20 grms.) in 2N nitric acid (100 c.c.) which has been saturated with sodium nitrate is recommended for the absorption of ethylene in the presence of benzene vapour; the gas is agitated with 5-10 c.c. of the solution for 2-3 minutes. Propylene appears to behave similarly to ethylene, since it is indifferent to alkaline mercuric cyanide solution whilst readily absorbed by mercuric nitrate solution.—H. W.

Petroleum; Genesis of — as revealed by its nitrogen constituents. C. F. Mabery. J. Amer. Chem. Soc., 1919, 41, 1690—1697.

It has already been shown that the heavier varieties of petroleum from the California, Texas, Ohio, Canadian, Russian, and similar fields are complex mixtures of the denser hydrocarbons with oxygen, sulphur, and nitrogen derivatives; they all differ widely in composition from the lighter varieties in the eastern territory of the United States. An examination of twenty-one specimens of the latter, however, discloses in every case the presence of nitrogen in amount ranging from 0.01 to 0.48%, so that the conclusion is justified that nitrogen is contained in the petroleum of all the principal oilfields in form of combination which could have had their origin only in the remains of vegetable or animal bodies. Presumptive evidence has been given that

the associated hydrocarbons in petroleum had the same origin. The nitrogen was estimated by Kjeldahl's process on the one hand, and by a process which is a combination of the Dumas procedure for nitrogen and the oxygen combustion for carbon on the other hand. The results given by the two methods show very fair agreement. (See also J. Chem. Soc., Jan., 1920.)—H. W.

Viscosity of hydrocarbon oil mixtures; Method for calculating —. W. F. Espy. Petroleum (Chicago), 1919, 8, 27–28.

VISCOSITY numbers are not additive, and therefore no factor can be applied to the calculation of the viscosity of a mixture which will take into account the mutual effect on viscosity of each component. The author constructs an empirically-derived chart, made up of the observed viscosities of mixtures of a light and heavy oil respectively and plotted in terms of a percentage mixture of oils of higher and lower viscosities. These percentages are additive, and from such a chart there may be read off directly the volume of the two components which will give a resulting mixture of the desired viscosity.

—A. E. D.

Paraffin; Thermal expansion of and solutions of —. W. von Piotrowski. Z. physik. Chim., 1919, 93, 596–606.

THE coefficient of expansion of solid paraffin from petroleum lies between 0.000812 and 0.005108. Lignite tar paraffin has about the same value, but when molten the value is about double. The dependence of the specific gravity on the temperature in the case of solutions of paraffin in solvents of high specific gravity may be expressed by the mixture rule, but in the case of solvents of low specific gravity irregularities are found in the region of the melting point. The specific gravity of solutions, at temperatures above the melting point of the paraffin, may be approximately calculated by the mixture rule. (See also J. Chem. Soc., 1920, i., 1.)

—J. F. S.

PATENTS.

Peat fuel; Manufacture of — from damp peat as dug from the bog strata. S. C. Davidson, Belfast. Eng. Pat. 131,326, 31.10.18. (Appl. 17,483/18.)

RAW peat as dug from the bog is thoroughly mixed with slaked lime until the mixture attains a homogeneous putty-like consistence. The water is then expelled by subjecting the mixture to mechanical pressure. Briquettes formed in this manner are claimed to be free burning, and to constitute a suitable fuel for household and industrial purposes.

—W. P.

Briquettes from (a) flue dust or (b) sawdust and the products resulting therefrom; Manufacture of —. C. Michel and V. Raskin, Paris. Eng. Pats. (a) 131,886 and (b) 131,887, 2.11.18. (Appls. 17,945 and 17,946/18.)

A MIXTURE is first prepared containing (a) flue dust or culm or (b) sawdust and coal dust with pitch and a mineral addition consisting of lime and/or cement and/or a little dry clay. The pulverised ingredients are mixed whilst cold and then introduced into a closed agitator where the temperature is raised to 150°–180° C. The pitch softens, moisture is given off from the wood, and the cement and lime commence to set. The paste is compressed under a pressure of 200–300 kilos. per sq. cm. and allowed to cool in moulds, forming a compact block.—W. P.

Washing coal or other minerals or materials. C. Burnett, Trowbridge. Eng. Pat. 131,596. (Appls. 17,873, 1.11.18, and 19,112, 26.11.18.)

THE apparatus is particularly applicable to the washing of slack or fine coal. The coal is fed on to an endless belt, on to which play jets of water, which carry the coal along in the opposite direction to which the belt is travelling. The coal is delivered into a second washing chamber in which water is forced through the coal by an intermittent or pulsating process. The belt inclines downwards into the second tank. The water from the second washing is conveyed to a settling tank, from which the slurry is again conveyed for treatment to the second washing tank.—W. P.

Coke; Apparatus for quenching —. S. N. Wellington, London. Eng. Pat. 131,867, 19.8.18. (Appl. 13,452/18.)

A RECEPTACLE, adapted to run on rails, receives the whole charge of coke from the oven upon a conveyor at the bottom of the receptacle, which is provided with a water jacket from which hot water may be supplied under the pressure of the steam in the water jacket for quenching the charge.

—W. P.

Coke ovens; Heating walls of — and blocks for the construction of such walls. A. Roberts, Chicago, U.S.A. Eng. Pat. 135,091, 7.3.19. (Appl. 5718/19.)

THE walls are so designed that the desired proportion of heat from the heating gases will travel towards the interior of the carbonising chamber, leaving only a portion of the heat for the recuperating structure. Blocks in the heating wall are recessed to provide a number of gas passages in the body of the wall, so that a greater amount of heat is delivered into one face of the wall than into the opposite face. Blocks particularly adapted for the construction of such a heating wall are described.

—W. P.

Coke [from pitch]; Manufacture of —. J. Becker, Assignor to Koppers Co., Pittsburgh, Pa. U.S. Pat. 1,320,371, 4.11.19. Appl., 15.3.18.

A COKE-PITCH mixture is coked, the residue is again coked after addition of more pitch, and the process is repeated until a substantially ash-free coke is obtained, derived in the main from pitch.—A. E. D.

Coke-oven iron work. L. Wilputte, Assignor to A. A. Wilputte, New Rochelle, N.Y. U.S. Pat. 1,320,432, 4.11.19. Appl., 27.5.18.

AT the opposite ends of horizontal coke ovens are placed flash plates and metal connecting members. The oven masonry is recessed to hold these metal parts.—W. P.

Chamber oven. H. Bröcker, Harburg. Ger. Pat. 314,118, 30.11.17.

PRODUCERS are built between the gasifying chambers. The upper layers of coke in the producers are heated by the hot gases from the neighbouring chamber. Any chamber can be cut out when necessary for repairs without the working of the rest being discontinued.—W. P.

Safety device against explosions for gas mains [of coke ovens]. R. Wilhelm, Altenessen. Ger. Pat. 313,649, 8.3.16. Addition to Ger. Pat. 308,073.

A MEMBRANE attached to a gas main operates two valves passing water into a water seal and indifferent gas respectively when the pressure diminishes, so that the current of inert gas can only pass in one direction, i.e., through that portion of the gas main beyond the membrane.—D. W.

Water-gas producers. Comp. pour la Fabrication des Compteurs et Matériel d'Usines à Gaz. Paris. Eng. Pat. 112,268, 17.12.17. (Appl. 18,757/17.) Int. Conv., 19.12.16.

THE producer is fitted with a false grate to support the coke and effect the sharp separation of the clinker without withdrawing the coke. The producer grate is rectangular in shape.—W. P.

Water-gas plants; Operation of —. H. E. Smith, London. Eng. Pat. 134,573, 3.8.18. (Appl. 12,694/18.)

AN improved arrangement of the valves and gearing for operating water-gas plants of the twin generator type (Eng. Pat. 24,893 of 1908; this J., 1909, 876) is described, gas making being intermittent, and the direction of travel of the steam during the gas-making period being reversed after each of the blowing periods. Dry-faced centre valves for the direction of the air and blow gases are coupled to work in conjunction with the steam inlet connection and gas outlet by operating one central shaft. The pressure of the blowing plant may be utilised for the operation, using a floating bell suitably connected to rotate the centre valves. The bell is released at certain intervals of time according to the adjustment of the air-regulating valves.—W. P.

Hydrogen or water-gas; Apparatus for the production of — from exhaust steam. F. Lang, Frankenthal. Ger. Pat. 313,917, 24.9.16.

IN apparatus for the production of hydrogen or water-gas from exhaust steam, an attachment to the producer automatically releases the back-pressure of the steam during the periods in which the steam is not led into the producer. The steam is superheated before entering the producer, either by the waste heat of the producer or of the gases generated, or, if the supply of exhaust steam is insufficient, by the addition of highly-superheated steam from other sources. In the latter case sufficient superheated steam to maintain the required pressure is automatically led into the exhaust steam supply by means of a reducing valve.

—L. A. C.

Producer-gas; Generation of —. H. W. Bamber and E. Goldschmid-Abrahams, London. Eng. Pat. 134,113, 23.12.18. (Appl. 21,539/18.)

MEANS are described for automatically controlling the water supply to a gas producer with change of temperature, making use of the expansion and contraction of the fire-box of the producer or of some part in contact with the latter. The temperature of the furnace may be recorded by means of an indicator in connection with the device, which is intended primarily for use in producers for automobile vehicles.—W. P.

Gas from solid fuel for automobile internal combustion engines and the like; Construction of generators for —. J. W. Parker, Harrow, Middlesex, and E. Goldschmid-Abrahams, London. Eng. Pat. 134,300, 29.10.18. (Appl. 17,647/18.)

THE producer consists of an inner casing made up of superimposed sections of cast iron enclosed by an outer sheet-metal shell, forming an annular space round the furnace through which passes the air for combustion. The lower sections are provided with radiating fins which divide the annular space into vertical conduits.—W. P.

Combustible gas; Apparatus for producing —. W. Pickard, Rochdale, and D. R. Dobson, Bolton. Eng. Pat. 134,907, 11.11.18. (Appl. 18,430/18.)

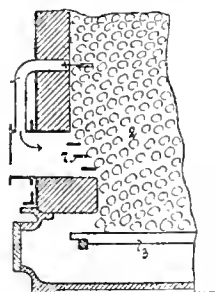
COMBUSTIBLE gas is prepared from liquid hydro-

carbon by passing the vapours mixed with steam through a red-hot mass of charcoal and iron filings (Eng. Pat. 122,079; this J., 1919, 128A). In an improved form of apparatus a burner is placed at the bottom of a central aperture, in which is situated a casing, which is in communication with chambers, the first containing iron filings and the second a mixture of charcoal and iron filings.

—W. P.

Gas generators. E. Svensson and M. A. Crank, Copenhagen. Eng. Pat. 134,454, 11.2.19. (Appl. 3358/19.)

THE producer is downward burning and is intended for low-grade fuels. Horizontally above the main



fire, resting on the grate, 3, are arranged one or more cavities in a circle, the number and size of these cavities being so chosen that they maintain a circular layer of burning fuel filling the whole circumference of the producer. These cavities contain stepped grates, 7, for supporting the fuel. When the producer is working, air from the interior of the producer, and also steam if the fuel used is moist, pass through the pipes, 8, and the grates, 7, into the burning fuel.—W. P.

Gas producers; Apparatus for the automatic cleaning or clinkering of —. J. Moussiaux, Huy, Belgium. Eng. Pat. 135,058, 1.2.19. (Appl. 2526/19.)

IN water-sealed producers the layer of combustible is supported by a somewhat thick layer of clinker, which itself rests on a revolving bed provided with chimneys or shoots for the discharge of the clinker into an external circular channel. The bottom of each chimney is formed of a fixed circular track above which the chimney travels. In each chimney the lower part of the back wall is cut away in such a manner that the clinker can spread over the track on the rotation of the chimneys, being afterwards removed by suitable scrapers or rakes formed by the front wall of the following chimney.—W. P.

Coal- and water-gas; Apparatus for the manufacture of combined —. W. D. Wilcox, Chicago, Ill. U.S. Pat. 1,321,963, 18.11.19. Appl., 17.1.16.

WASTE gases from a producer chamber pass upwards around a vertical gas retort, then into a flue containing a steam superheater. The retort is provided with a number of gas outlets at different levels through which the gases given off at different temperatures may be withdrawn separately and continuously.—W. P.

Peat and the like; Manufacture of ammonia and combustible gas or coke from —. F. Sauer, Potsdam. Ger. Pat. 314,015, 18.2.13.

DRIED peat or the like is heated in a shaft furnace so arranged that decomposition is effected in three separate zones. A mixture of air and steam is led in both at the top and bottom of the upper zone, which contains the fresh peat, and the ammonia generated is led away from the middle of the zone; the material passes slowly down through the middle neutral zone into the lower zone, and the combustible gas produced in the lower zone is led away through a separate outlet. The pressure of the mixture of air and steam led into the upper zone is regulated by the pressure of gas escaping from the lower zone.—L. A. C.

Ammonia and tar recovery process [for producer-gas]. J. van Ackeren, Assignor to Koppers Co., Pittsburgh, Pa. U.S. Pat. 1,320,369, 4.11.19. Appl., 26.5.19.

PRODUCER-GAS, superheated with respect to its tar vapour and water content, is freed from dust by electrical precipitation, and is then cooled to a point at which tar vapours are condensed whilst the water is still in the state of vapour. The tar mist is then electrically precipitated.—A. E. D.

Gas washer. Simon-Carves, Ltd., and J. H. Brown, Manchester. Eng. Pat. 133,987, 19.9.18. (Appl. 15,208/18.)

THE gas is made to pass in a horizontal direction alternately through spaces having positive means for production of spray, and through latticed slats kept wet by the spray. A spray catcher consisting of vertical and horizontal baffles is placed before the outlet of the washer.—W. P.

Gas washer. A. A. Goubert, Englewood, N.J., Assignor to H. Bentz, Montclair, N.J. U.S. Pat. 1,320,852, 4.11.19. Appl., 1.5.18.

A GAS washer is provided with a number of baffles consisting of closely arranged plates over which water is kept flowing. The plates in successive baffles increase in number, and decrease in cross-sectional area, and also in respect to the horizontal and vertical spaces separating them.—W. P.

Gas liquor; Process for removing acid constituents from —. Gewerkschaft des Steinkohlenbergwerks Lothringen, Gerthe. Ger. Pat. 313,918, 16.7.18.

IN A continuous process for removing the acid constituents from gas liquor by distillation, the gases produced are freed from ammonia by washing, the solution being returned to the still. The residual gases pass through a second wash column, which is arranged in two sections, to effect the separation of water rich in hydrogen sulphide from the upper portion, and water rich in carbon dioxide from the lower portion.—W. P.

Hydrocarbon; Rectification of —. De Bataafsche Petroleum-Maatschappij, and J. H. C. de Brey, The Hague. Eng. Pat. 123,522, 30.10.18. (Appl. 17,741/18.) Int. Conv., 21.2.18.

TO separate a mixture of two volatile components, e.g., natural gas containing gasoline, and recover the gasoline, a process of rectification under pressure is claimed. A preliminary treatment consists in applying sufficient pressure to form a two-phase system, which is then submitted to rectification. A pressure of 20 atmospheres or more may be employed.—A. E. D.

Peat, waxes, oils, or fats; Production of — from peat, peat-straw, mosses, lichens, algae, grass, straw, pine needles, and the like. K. H. V. von Porat, Stockholm. Eng. Pat. 133,989, 21.9.18. (Appl. 15,357/18.)

THE vegetable matter is extracted by means of terpenes or their derivatives, having a boiling point higher than 100° C. (e.g., oil of turpentine), preferably under pressure.—A. E. D.

Oil-converting apparatus. J. H. Adams, Brooklyn, N.Y., Assignor to Texas Co., Houston, Tex. U.S. Pat. 1,320,351, 28.10.19. Appl., 1.12.09.

OIL is supplied continuously, under pressure, to an elongated retort of restricted cross-section, to which a high temperature is applied locally. The vapours are maintained in contact with the oil surface during the cracking operation, and are subsequently condensed.—A. E. D.

Oils; Process and apparatus for the conversion of liquids, fluids, and —. J. H. Adams, Brooklyn, N.Y., Assignor to Texas Co., Houston, Tex. U.S. Pat., (a) 1,320,726 and (b) 1,320,727, 4.11.19. Appl., 31.12.14 and 29.3.15.

(A) HIGH-boiling oil is subjected to heat under its own vapour pressure. Superheated steam is introduced into the oil and the volatile products are separated from the heavy residues, which are re-treated. (b) In an apparatus for carrying out the process described under (a), automatic pressure-control valves are provided between the still and the condenser and also at the end of the condensing worm. A thin horizontal cracking still containing a metal of melting point lower than the cracking point of the oil may be used. It communicates with an expansion dome, from which the converted vapours may be withdrawn at the top and the unconverted oil at the bottom.—A. E. D.

Hydrocarbon oils; Treatment of —. R. H. Brownlee, Pittsburgh, Pa. U.S. Pat. 1,320,376, 4.11.19. Appl., 11.7.17.

A CURRENT of gas is introduced near the lower end of a treating system, which is heated and maintained under pressure, and oil is introduced at a point below the upper end of the system. The oil and vapour flow upwards through the system, passing at intervals through areas in which the speed is reduced in order to cause separation of the heavier components. The separated heavy oils flow downwards in counter current to the stream of gas. Part of the separated heavy oil is trapped in the zones where the speed is reduced, and the trapped oil is from time to time drawn off into the lower part of the apparatus.—A. E. D.

Petroleum products; Apparatus for treating —. U. S. Jenkins, Chicago, Ill. U.S. Pat. 1,321,749, 11.11.19. Appl., 27.9.16.

FOR the production of light oils, heavy hydrocarbons are treated under pressure in a still fitted with means for removing vapours of "greater average density than that of the lowest produced in the still from selective points below the top of the still," and for mechanically circulating the liquid in the still.—L. A. C.

Briquette machine. F. H. Rogers, London. From St. Louis Briquette Machine Co., St. Louis, Mo., U.S.A. Eng. Pat. 135,061, 5.2.19. (Appl. 2839/19.)

Gas generators or the like; Apparatus for feeding fuel to —. G. H. Bentley and E. G. Appleby, London. Eng. Pat. 135,272, 18.11.18. (Appl. 18,888/18.)

Coal and the like; Method of cleaning —. H. R. Conklin, Joplin, Mo., U.S.A. Eng. Pat. 130,972, 7.7.19. (Appl. 16,976/19.) Int. Conv., 5.8.18.

SEE U.S. Pat. 1,290,516 of 1919; this J., 1919, 166 A.

Petroleum and the like; Method and apparatus for distilling —. E. C. R. Marks, London. From Cleveland Trust Co., Cleveland, Ohio, U.S.A. Eng. Pat. 134,567, 21.4.18. (Appl. 6921/18.)

SEE U.S. Pat. 1,260,584 of 1918; this J., 1918, 293 A.

Fuel; Liquid —. E. C. R. Marks, London. From United States Industrial Alcohol Co., New York. Eng. Pat. 131,766, 30.1.19. (Appl. 10,712/19.)

SEE U.S. Pat. 1,296,902 of 1919; this J., 1919, 353 A.

Petroleum pitch. Eng. Pat. 114,617. See III.

Carbon monoxide in hydrogen. Eng. Pat. 131,243. See VII.

Hydrogen generators. Eng. Pats. 131,901-2. See VII.

Sulphuric acid from coal gas etc. Ger. Pat. 300,036. See VII.

Gas-testing machine. U.S. Pat. 1,320,584. See XXIII.

[Combustible] gases. U.S. Pats. 1,321,063-4. See XXIII.

Inflammable gases. Ger. Pat. 313,858. See XXIII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

PATENTS.

Petroleum pitch; Destructive distillation of —. K. Nomi, Kitatoshimagnn, Japan. Eng. Pat. 114,617, 11.2.18. (Appl. 2432/18.) Int. Conv., 4.4.17.

PETROLEUM pitch is distilled at about 500°—600° C., oil and gas being produced, and a pure coke left. The gas is mainly hydrogen.—A. E. D.

Destructive distillation of carbonaceous materials. F. M. Perkin, and Nitrogen Products and Carbide Co., Ltd., London. Eng. Pat. 134,236, 19.2.18. (Appl. 2920/18.)

INCLINED or vertical retorts of refractory material for low temperature distillation of coal etc. are provided with passages or channels in the walls for the preliminary passage of superheated inert gas or steam, in order to increase the yield of tar, tar oils, and paraffin hydrocarbons. The retorts are elliptical in cross-section, and are connected together in pairs.—W. P.

Carbons for electric arc lamps. W. Heape, London, and H. B. Grylls, Weybridge. Eng. Pat. 133,376, 2.8.18. (Appl. 12,662/18.)

THE carbons are enclosed whilst dry in a chamber or receptacle which is then evacuated. The chamber or carbons may be heated during the evacuation. After thus removing occluded gases, the carbons are immersed in an impregnating liquid. (See Eng. Pats. 126,673 and 128,057; this J., 1919, 455 A. 568 A.)—B. N.

Mercury arc lamp. P. G. Nutting, Wilksburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,320,087, 28.10.19. Appl., 13.3.18.

A MERCURY arc lamp comprises anode and cathode legs, and a connecting arc tube formed of hard glass softening only at a high temperature. Tungsten leading-in wires and terminals are sealed into the ends of the legs.—B. N.

Incandescence cathode for discharge tubes. W. Germershausen, Leipzig. Ger. Pat. 303,274, 16.3.17.

THE cathode is composed of a resistant material of high melting point, e.g., iridium, upon which a thin layer of a metal such as tungsten, affording copious emission of electrons when heated, is wound spirally, in direct electrical contact.

—J. S. G. T.

Distillation of waste liquors. Eng. Pat. 120,721. See V.

Distillation of waste cellulose lyes. Ger. Pat. 313,607. See V.

III.—TAR AND TAR PRODUCTS.

Naphthalene; Manufacture of pure — from coal tar. J. A. Davy. Coke Oven Managers' Assoc., Nov. 29, 1919. Gas J., 1919, 148, 489—490.

THE naphthalene is present in the carbelic and light creosote oils, which represent about 20—25% of the tar distilled. These oils are washed for carbelic and cresylic acids before further treatment, as the latter have the effect of keeping the naphthalene in suspension. After cooling for a period of 48 hours the excess oil is drained off from the precipitated naphthalene, which is then distilled in a 10-ton gas-fired still. Large flaky crystals are obtained which still contain 25—30% of oil. The main quantity of this is removed by hydraulic pressure (35 cwt. to 2 tons per sq. in.) in a steam-jacketed press. The "pressed naphthalene," containing about 1% of oil, is then melted and washed, first with sulphuric acid and then with caustic soda solution, these being followed by sodium sulphate solution, on which the naphthalene floats. After separation it is subjected to a final distillation. The yield of finished product from crude "salts" is 40—45%.—W. P.

Side-chain oxidations with potassium permanganate. [Oxidation of nitrotoluenes to nitrobenzoic acids.] L. A. Bigelow. J. Amer. Chem. Soc., 1919, 41, 1559—1581.

THE action of potassium permanganate in alkaline solution on the three nitrotoluenes has been investigated with a view to determining the optimum conditions for the technical preparation of the nitrobenzoic acids. The experiments were usually performed by gradually adding the solid permanganate to a boiling suspension of the nitrotoluene in aqueous sodium hydroxide solution. Changes in the conditions of oxidation, in general, merely cause a different proportion of the nitrotoluene to be attacked by the oxidising agent, the ratio of nitrobenzoic acid produced to unchanged nitrotoluene remaining essentially constant. It is concluded, therefore, that the quantity of nitrotoluene or nitrobenzoic acid entirely destroyed by the oxidising agent is practically constant and almost independent of the oxidation procedure, and that at least two entirely independent actions take place, namely, the oxidation of the organic compound and the decomposition of the permanganate into manganese oxides and free oxygen. Increase in the concentration of the alkali in the oxidising mixture up to a certain point favours the oxidation of *o*-nitrotoluene, beyond which point a further increase produces essentially no effect, all other conditions being kept constant. By this increase in alkali concentration the oxidation of the *meta*-isomeride is hindered, such oxidation being most effective in an essentially neutral medium; with *p*-nitrotoluene, oxidation is favoured up to a certain point, beyond which the quality of the product becomes very poor. Increasing dilution of the reaction mixture favours the oxidation of all the nitrotoluenes, probably owing to retardation of the decomposition of the permanganate into manganese oxides and oxygen. Under all circumstances *p*-nitrotoluene is oxidised most readily, the *ortho*-compound next, and the *meta*-isomeride least readily. Nearly the same results are obtained when the oxidation is effected in copper or enamel-lined vessels, but distinctly lower yields and products of poorer quality result when iron vessels are used both with and without alkali. The addition of salts of calcium and magnesium produces no noticeable effect in the oxidation of *p*-nitrotoluene in neutral solution, and a like result is obtained when pyridine is added to an alkaline solution, although the latter acts as a powerful negative catalyst in certain permanganate reactions. (See also J. Chem. Soc., 1920, i., 20.)—H. W.

Motor benzol. Steding. See IX.

Tetralin. Vollmann. See XIII.

PATENTS.

Carbazole; Production of high percentage —. Soc. d'Eclairage, Chauffage, et Force Motrice, Paris. Eng. Pat. 121,455, 7.10.18. (Appl. 16,291 18.) Int. Conv., 4.12.17.

CRUDE carbazole is dissolved in phenols, such as commercial carbolic acid, cresols, or xylenols at about 70° C., using 5–10 parts of solvent according to the richness of the carbazole; the solution is filtered hot, and the crystals which separate on cooling are washed with a small amount of the cresol mixture. The product from the first crystallisation yields an 80% carbazole, which may be purified to 90% by a second crystallisation from phenol or its homologues. The crude carbazole used as the initial material may consist of the residue obtained by distilling the liquor from the purification of anthracene by means of pyridine. Crude carbazole may also be obtained by the action of water on potassium-carbazole, produced by treating crude or concentrated anthracene with caustic potash.

—J. F. B.

Benzyl chloride and benzal chloride; Production of — and of certain homologues and substitution products of these compounds. Levinstein, Ltd., H. Levinstein, and W. Bader, Manchester. Eng. Pat. 131,250, 27.7.18. (Appl. 12,227/18.)

PRODUCTS chloro-substituted in the side chain are obtained by treating hydrocarbons at temperatures below 0° C. with hypochlorous acid in aqueous solution. Thus, for example, toluene (3 mols.) is emulsified with a hypochlorite solution containing 1 mol. of active chlorine, the mixture is cooled to –5° C., and dilute sulphuric or other acid ($\frac{1}{2}$ mol.) is added gradually. The product contains unchanged toluene, 60–70% of the theoretical quantity of benzyl chloride, and small quantities of benzal chloride and other chlorinated products, and is purified by fractional distillation. Similarly a second atom of chlorine can be introduced, it preferably occupying a position in the methyl group already substituted if more than one side chain is present. The method is not applicable to nitro-substitution products, sulpho-chlorides, or cresol esters, whilst sulphonic acids under these conditions are substituted in the nucleus.

—G. F. M.

Quinone; Preparation of —. J. M. Weiss, New York, and C. R. Downs, Cliffside, N.J., Assignors to The Barrett Co. U.S. Pat. 1,318,631, 14.10.19. Appl., 27.5.18.

BENZOQUINONE is produced by subjecting benzene in the vapour phase to the action of a gas containing oxygen at a suitable temperature and in the presence of a suitable catalyst.—G. F. M.

Benzene; Catalytic oxidation of —. J. M. Weiss, New York, and C. R. Downs, Cliffside, N.J., Assignors to The Barrett Co. U.S. Pat. 1,318,633, 14.10.19. Appl., 8.4.19.

BENZENE is oxidised to compounds containing hydrogen, oxygen, and less than six carbon atoms by the action of a gas containing oxygen at a temperature of 300°–500° C. in the presence of a suitable catalyst.—G. F. M.

Phenols and their substitution products; Preparation of aromatic —. E. H. Zollinger and H. Roehling, Berlin. U.S. Pat. 1,321,271, 11.11.19. Appl., 16.11.15.

AN aromatic halogen compound, in which the halogen can be substituted by hydroxyl, is heated

under pressure to the temperature of saponification with a solution of an alkali hydroxide and a lead salt which forms an insoluble compound with the phenol. The reaction mixture is then quickly cooled to cause the crystallisation of the metallic compound of the phenol, and this is separated and decomposed to liberate the free phenol.—G. F. M.

Benzaldehyde; Production of —. J. M. Weiss, New York, N.Y., and C. R. Downs, Cliffside, N.J., Assignors to The Barrett Co. U.S. Pat. 1,321,959, 18.11.19. Appl., 25.6.19.

BENZALDEHYDE is produced by passing a mixture of toluol vapour and a gas containing oxygen over an oxide of molybdenum heated above 500° C.

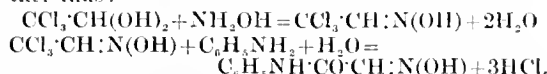
—L. A. C.

Anthranilic acid; Process of producing —. R. S. Potter, Grantwood, N.J., Assignor to The Barrett Co. U.S. Pat. 1,322,052, 18.11.19. Appl., 17.6.19.

As a step in the manufacture of anthranilic acid, carbon dioxide is passed into a solution containing an alkali anthranilate and an alkali carbonate to convert the carbonate to the bicarbonate; the precipitated bicarbonate is separated by filtration, and an excess of sulphur dioxide is passed into the filtered liquor.—L. A. C.

Isonitrosoacetanilide and its substitution products; Production of —. J. R. Geigy A.-G., Basle. Ger. Pat. 313,725, 11.5.18.

CHLORAL hydrate in presence of hydroxylamine or hydroxylamine sulphonic acids is allowed to act at an appropriate temperature on aniline or its ring-substituted halogen, alkyl, alkyloxy, or carboxyl derivatives, or their N-monoalkyl or monoaryl derivatives. Chloraloxime is formed and reacts further thus:—



Hydroxylaminesulphonic acid is prepared by slowly adding 50% sulphuric acid to a mixture of sodium nitrite solution, ice, and bisulphite until the liquid turns Congo red paper distinctly blue. The separation of the sodium hydroxylaminedisulphonate first formed into hydroxylaminesulphonic acid and sodium sulphate proceeds rapidly but is only completed after standing for two days. On boiling under a reflux condenser, hydroxylamine sulphate is produced, and this gives, when boiled with aniline and chloral hydrate, isonitrosoacetanilide, bright yellowish plates, m. pt. 175° C. with decomposition, slightly soluble in cold water, benzene, fairly soluble in ether and alcohol, precipitated from solutions in dilute alkalis by acids. On warming with strong sulphuric acid it yields isatin- β -imine, which separates into isatin and ammonium sulphate when its dark red solution in sulphuric acid is diluted with water. *p*-Chloroaniline gives with hydroxylamine sulphate and chloral hydrate isonitroso-acet-*p*-chloroanilide, white needles, m. pt. 165° C., slightly soluble in cold water and ether, soluble in alcohol.—H. J. H.

Phenolic bodies; Manufacture of sulphonates of aromatic hydrocarbons for use in making —. F. Cohellis, Charleston, W.Va., U.S.A. Eng. Pat. 131,715, 6.2.19. (Appl. 2946/19.)

SEE U.S. Pat. 1,301,909 of 1919; this J., 1919, 569A.

Coke [from pitch]. U.S. Pat. 1,320,371. See IX.

Sulphonic acids. Ger. Pat. 312,867. See VI.

Malvic acid. U.S. Pat. 1,318,632. See XX.

IV.—COLOURING MATTERS AND DYES.

Indigo industry; Future prospects of the natural —. Effect of superphosphate manuring on the yield and quality of the indigo plant. W. A. Davis. Agric. Res. Inst., Pusa. Indigo Publ. No. 4, 1918. 19 pages.

The changes imposed on indigo cultivation in Bihar by the appearance of synthetic indigo have resulted in the rapid impoverishment of the indigo soils in recent years and a consequent serious reduction in the yield of dye. Superphosphate manuring has now been tried on several estates, and very favourable results have been obtained, confirming the view previously advanced (Indigo Publ. No. 1, this J., 1918, 194 K; 1919, 248 A), that the depletion of available phosphate in the soil was the principal cause of the failure of indigo crops. Not only was the actual yield of green plant per acre far higher in the case of superphosphate-treated land, but the quality, or richness in indican, was superior, so that the yield of eake indigo amounted to as much as 32 seers (64 lb.) per acre for a single cutting, more than was ever obtained from the Java plant in its best days.—G. F. M.

Indigo; Loss of — caused by bad settling and means of obviating this. Use of dhak gum, its effect on yield and quality. W. A. Davis. Agric. Res. Inst., Pusa. Indigo Publ. No. 3, 1918. 16 pages.

The use of dhak gum, a ruby-coloured exudation from the dhak tree (*Butea frondosa*) is recommended to improve the settling of indigo after heating. An increase in yield of 3–6 seers (6–12 lb.) of indigo per 100 maunds (8000 lb.) of plant has been obtained, representing an increase of about 70% with Sumatran plants and 35% with Java plants, and no appreciable deterioration of quality ensues. Dhak gum is most useful in factories where so-called "green vats" are obtained, but even under very favourable conditions of fermentation perfect settling is rare, and use of the gum as part of the ordinary routine is advisable, as even if the seed-water contains only 1 part of indigo in 10,000 it represents a loss of 30% with Sumatran working. The gum should be used in the proportion of 1 seer (2 lb.) to 100 maunds (8000 lb.) of plant, a strained solution being added to the beating-vat about 5 mins. before beating is ended.—G. F. M.

Indican from indigo-yielding plants; Improved method of preparing —. B. M. Amin. Agric. Res. Inst., Pusa. Indigo Publ., No. 5, 1919, 9 pages.

An improved and simplified method of isolating indican, applicable to all *Indigofera* sp. and other indigo-yielding plants, and admitting of the separation as indican hydrate of at least 80% of the glucoside present in the aqueous extract of the leaves, is carried out as follows:—The leaves are twice extracted with boiling water, and the extract is treated with slaked lime and filtered to remove gums, tannins, etc. The filtrate is evaporated in an open dish, treated with carbon dioxide to precipitate the lime still in solution, and after refiltration the clear solution is evaporated to a thick syrup, which is extracted three or four times with acetone. The acetone is distilled off from the extract, and the aqueous residue, when cooled in ice, sets to a crystalline mass of crude indican hydrate, $C_{14}H_{17}O_5N \cdot 3H_2O$. The crystals are separated and dried at the ordinary temperature. Pure white anhydrous indican, m. pt. 177°–178° C., is obtained by precipitation with benzene from a hot alcoholic solution of the hydrate.—G. F. M.

PATENTS.

Quinone dyestuffs; Production of —. M. Becke, Weidling, W. and H. Suida, Vienna. Ger. Pat. 300,707, 12.6.15.

NAPHTHAQUINONES or their derivatives are combined

with aminobenzenesulphonic acids which contain a further free or substituted amino or hydroxyl group or with dyestuffs containing amino or sulphonic groups in the proportion 1 mol. quinone to 1 reactive amino group. The reaction is effected in aqueous solution or suspension or by grinding together the reacting substances. Thus aminoazo dyestuffs are combined with naphthaquinone, so that each molecule of the latter or its derivative reacts with one amino group, by the aid of an oxidising agent, air, or even another molecule of quinone. When the naphthaquinone contains separable negative substituents the combination is effected by such condensation agents as carbonates, bicarbonates, or acetates. Here, also, when a sulphonic group is split off, the addition of an oxidising agent capable of giving up one atom of oxygen is advantageous. While the colour is often completely formed in the cold in aqueous solution or suspension, the application of heat is more usually necessary, especially with nitrosonaphthols. The resulting dyestuffs are soluble in water and contain a quinone nucleus which promotes adhesion to the fibre; the fastness of the shades produced is increased by after-treatment with metallic salts.

—H. J. H.

Solutions of organic substances. Ger. Pat. 313,726. See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Wool; Absorption and retention of soap by —. A. Woodmansey. J. Soc. Dyers and Col., 1919, 35, 169–171.

CROSS-BRED serge, spun silk yarn, cotton wool, and cotton cambric were found to absorb from a 1% solution of Castile soap 3.6, 1.8, 1.4, and 0.4% respectively of fatty acid calculated on the weight of material treated. The author describes a number of experiments which indicate that preferential absorption of the basic component of the soap occurs and show that, within limits, absorption increases with the duration of immersion and the concentration of the soap bath, and that the amount of soap remaining in cloth on scouring depends on the nature and thoroughness of the scouring process.

—S. S. A.

Wool and silk; Colouring of — with strong acids. P. Waentig. Text. Forsch., 1919, 1, 59–63. Chem. Zentr., 1919, 90, IV., 492.

If boiling 50% sulphuric acid or fuming hydrochloric acid is poured on to wool in presence of a few crystals of sugar, a red colour develops in a short time. Without the sugar pure wool gives very little colour, at most only a faint rose tint, but in presence of considerable quantities of cotton, or artificial silk, or of other cellulosic substances or of substances, such as pentosan, yielding furfural, a decided red colour is produced. The presence of fat in the wool leads to a brown colour. Silk behaves in a similar manner to wool.—B. V. S.

Cotton fibres and yarns; Effect of certain industrial processes on the strength of —. R. S. Greenwood. J. Textile Inst., 1919, 10, 274–278.

THE material used for the investigation was a sample of Sakelarides' Egyptian cotton; its grade was estimated as "fully good, fine staple," and the average length was 1½ in. to 1¾ in. The results appear to show that a yarn realises more of the available strength of the individual fibres than has been previously assumed. Previous observers give 20–25% as the strength realised, as compared with 67% found by the author. By mereerising yarn without tension the strength is increased 23%, the strength of the individual fibres being little

affected. It is concluded that when the fibre is allowed to shrink to its full extent no strengthening occurs. The fibres when removed from the yarn, however, are curly, and this suggests that the yarn has shrunk rather than the fibre. By mercerising yarn with tension there is also an increase in strength, but no increase in the fibre strength. In each case bleaching gives rise to an increased strength. The increase varies according to the previous treatments of the yarn. It cannot be concluded, however, that bleaching will always strengthen a yarn, but only that bleaching, properly carried out, will not weaken a two-fold yarn. The fibre strength appears to be considerably reduced—on an average 20%. This, together with the increased yarn strength, points to a greatly increased cohesion of the fibres in the yarn, possibly due to the removal of the natural wax lubricant.

—L. L. L.

Bast fibres; Retting of —. III. P. Kraiss. Z. angew. Chem., 1919, 32, 326–327. (See this J., 1919, 169A, 530A.)

FURTHER experiments on the bicarbonate process of retting carried out on nettles made it clear that nettles sterilised by treatment with chloroform could not be retted, but that on inoculation with a single stem of unsterilised nettle, or with a pure culture of bacteria obtained from the retting liquor of nettles, fermentation set in and retting was complete in 3 days. The retting of nettles takes place rather more rapidly than that of flax, but they are more bulky and require a bath equal to 20 times their weight, whereas flax can be retted with a bath equal to 10 times. Trials were made to substitute chalk for the sodium bicarbonate, but in the case of flax the action of chalk alone was not sufficiently rapid. It was found, however, that the use of chalk permitted a considerable economy in the use of sodium bicarbonate, so that instead of a 0.5% solution of the latter substance a liquor containing 0.5% of chalk and 0.1% of sodium bicarbonate could be used with equal effect. Thus it should be possible on the large scale to perform the retting of 1000 kilos. of flax straw with a bath of 10 cub. m. of water containing 50 kilos. of chalk and 10 kilos. of sodium bicarbonate, the action being completed in 3 days at 35°–37° C., as compared with 5 days required for the ordinary warm water retting process. Moreover, the new alkaline process of retting is practically odourless, whereas the acid retting waters have a most offensive odour.—J. F. B.

Fibres; Substitute — and their manipulation. "Cottonising." E. O. Rasser. Monatsch. Textilind., 1919, 34, 41–43. Chem. Zentr., 1919, 90, IV., 423–421.

"COTTONISING" may be applied to any fibres having a woolly feel, such as jute, hemp, flax, typha, and the like, but chiefly to flax and hemp tows and spinning wastes, as well as to fibres derived from the pulling of rags, twine and cloth wastes, recovered flax and hemp fibres, jute wastes and, lastly, flax and hemp grown for seed. A distinction is made between technical and purely chemical cottonising. In technical cottonising the wastes or fibres are passed through specially constructed tearing willows, and then submitted to a crimping process. A real resolution into ultimate fibres does not occur, and only coarse yarns can be spun which, owing to single projecting hairs, are not so smooth as the chemically isolated ultimate fibres, and therefore cohere more effectively both to each other and to other fibres spun along with them. In the chemical process of cottonising caustic soda and chlorine are employed, also urine, sodium carbonate, and Turkey red oil soaps, further, oxygen or

substances which develop gases. According to a process by Niesytka-Norna, flax straw is decorticated dry by machines and the fibre is then washed in such a manner that the washing fluid is pressed several times with considerable force through the material. Hemp is more easily cottonised than flax. In order to make the cottonised fibre more suitable for spinning, a treatment with a strong cold caustic soda lye is recommended, followed by hot water.

—J. F. B.

Marine fibre [*Posidonia Australis*]. J. Read and H. G. Smith. Commonwealth of Australia Inst. Sci. and Ind., Bull. No. 14, 1919, pp. 60.

MARINE fibre is pale brown in colour, lighter but coarser than coir fibre, to which it bears a general resemblance. The filaments are short in staple (50–200 mm.), and the length of the ultimate fibres is about 1 mm. The mean tensile strength of the filaments is 20.15 kilos. per sq. mm. section, and the elongation 5.29%, but the results are somewhat variable. The filaments are, however, extremely brittle. Chemically, the marine fibre is a highly pronounced lignocellulose, apparently intermediate between jute and wood, but it is characterised by an exceptionally high resistance to the action of dilute alkalis and solvents of cellulose, and by its unusual affinity for dyes, especially basic dyestuffs. *Posidonia* cellulose is obtained with a yield of 55% when the raw fibre is subjected to the chlorination process. The cellulose shows a resemblance to the cellulose of esparto and straw, both as regards length of fibre and its high furfural value. The raw material yields 7.5% of furfural and the isolated cellulose 9.2%. *Posidonia* cellulose, however, does not give colorations with aromatic amines like the cellulose from grasses. Treatment of the cellulose with 17.5% sodium hydroxide solution gave 79.2% of resistant α -cellulose, yielding only 2.9% of furfural. In this respect also it resembles esparto cellulose very closely. It is estimated that 4,600,000 tons of the material is available in the S. Australian deposits, and that the fibre, washed and purified by acid, could be landed at a European port at a cost of £19 per ton. Its shortness, coarseness, and irregularity of staple, the lack of flexibility and cohesion, and the structural characteristics of the filament are all opposed to its employment in the manufacture of fine textiles. For lower grade purposes it might be used as a diluent in admixture with wool, its dyeing properties and low heat conductivity being favourable points. For coarse textiles *Posidonia* fibre is inferior to jute in mechanical properties, but it could be used, and its resistance to chemical and bacterial agencies is advantageous for outdoor work. In so far as the textile inferiority of the material is dependent on the weakness and brittleness of the filaments, a remarkable improvement may be effected by suitable pre-treatment with dilute acids. The flexibility and, to some extent, the tensile strength are increased by steeping the fibre for several hours in 2% nitric acid; other acids have a similar, though weaker, influence. The authors suggest that previous unfavourable reports might be reconsidered with this in view, particularly since the large loss in the carding operation is to be attributed to the lack of flexibility of the filaments in the untreated condition. The authors' investigations have not shown favourable results for the *Posidonia* fibre as a starting point for the manufacture of explosives (compare Smart and Pecover, this J., 1918, 300T). On the other hand, provided a suitable method could be found for the isolation of the ultimate cellulose fibres, the utilisation of *Posidonia* in the paper industry would appear to offer the most promising outlet. The close similarity of *Posidonia* cellulose to esparto cellulose is a strong indication of its suitability as a material for printing papers and the lower grades of writing paper.—J. F. B.

Cellulose and nitrocellulose: absorbent power for gases, and constitution. B. Oddo. Gazz. Chim. Ital., 1919, 49, ii., 127—139.

CELLULOSE absorbs various gases to extents increasing with the solubilities of the gases in water, and absorption of a second gas is accompanied by partial displacement of a gas already absorbed. Both decan- and endeca-nitrated celluloses absorb dry hydrogen chloride and sulphur dioxide. (See also J. Chem. Soc., 1920, i., 16.)—T. H. P.

Nitroacetylcellulose. B. Oddo. Gazz. Chim. Ital., 1919, 49, ii., 140—145.

The preparation and the properties of a mixed acetyl-nitro derivative of cellulose, probably the tetracetyloctonitrate, are described. (See also J. Chem. Soc., 1920, i., 16.)—T. H. P.

Paper pulp; The colouring of — for textile purposes. E. Püschel. Papier-Zeit., 1919, 44, 1742—1743, 1744—1745. Chem. Zentr., 1919, 90, IV., 507—508.

The order of addition to the pulp is first the resin size, then the dyestuff solution, and lastly, after mixing for half-an-hour, aluminium sulphate. If it is desired that the stuff should become warm through prolonged beating, the alum is added shortly before emptying. Mineral colours, and basic and acid dyestuffs, added in the above manner, require no addition of mordant for fixation. In order to avoid uneven dyeing with basic dyestuffs it is advisable to add a little alum to the pulp before the dye. Acid dyestuffs develop their strongest colouring power with heavily sized papers containing 5% of size; their use is not economical in conjunction with weakly sized papers for spinning, and they have to be avoided altogether for unsized cellulose and cellulon yarns. In dyeing with substantive colours sufficient sodium carbonate should be added after the size to give the pulp a neutral or slightly alkaline reaction; the dyestuff solution is then added and 5—10% of Glauber's salt or common salt, calculated on the weight of the fibre, in order to fix the colour; the aluminium sulphate is added last. The quantity of salt used depends on the depth of shade; the water squeezed out of the pulp should not be coloured. Substantive colours are absorbed more readily if the stuff be heated to 50° C. The dyeing of finished paper yarns in dyeing machines has not given satisfactory results; even by prolonged action and the use of penetrating agents the colour does not go through. The best penetration of the dye is obtained by the foam-dyeing treatment.—J. F. B.

Paper pulp; Influence of temperature on the rate of draining of —. S. Smith. Papierfabr., 1919, 17, 1121—1123.

The author has tabulated the values of γ/η representing the ratio of the viscosity to the specific gravity of water at increasing temperatures. He then determined the rate of flow of water at different temperatures through two columns of gravel, fine and coarse. The results showed that the ratio of time of flow to γ/η was a constant for each material at all temperatures, depending only on the size of the particles. On the other hand, when the viscosity law so established was applied to some observed data published by Skark (Papierfabr., Festheft, 1912, 94, 767, and 802) for the rate of draining of variously beaten paper pulps at different temperatures, it was found that the above ratio was not constant, but decreased with increasing temperature. Thus the increased rate of draining of paper pulp on the wire at increased temperatures does not depend only on the change in the viscosity of the water, but is assisted by another factor which, provisionally, may be assumed to be

the shrinkage of the fibres by dehydration and consequently the increase in the size of the capillary passages between them.—J. F. B.

Paper; Quantitative estimation of mixed pulp in newsprint —. Pulp and Paper Mag., 1919, 17, 948—950.

CHEMICAL methods appear to be of little value for the estimation of mechanical and sulphite wood pulps in newspaper, and it is necessary to resort to microscopical methods. Using an accurate sample of the sheet, a careful estimation is made consisting of an average of 10 fields on each of three separate slides. The proportion of sulphite pulp thus estimated is considerably in excess of the percentage by weight actually present, because the cellulose, wood fibre loses 40% of its weight without corresponds to its weight relatively to the ligno-cellulose fibre. In the process of conversion into cellulose, wood fibre loses 40% of its weight without alteration of its dimensions. The difficulty of estimation varies according to the state of preparation of the fibres, the papers of the United States being considerably more beaten, and hence less easily analysed than Canadian papers. As the percentage of sulphite pulp increases the tendency to overestimate it by microscopical inspection is decreased. The following rules have been established as the result of the examination of a large number of mixtures. Scandinavian pulps: In mixtures estimated to contain 50% of sulphite pulp deduct three-tenths of the estimated percentage of sulphite and add this to the mechanical pulp. In mixtures estimated at 65% sulphite deduct one-fourth of this and add it to the mechanical. In mixtures estimated at 80% sulphite deduct one-tenth and add it to the mechanical. Canadian pulps: In mixtures estimated to contain about 42% of sulphite deduct seven-eighths of this and add it to the mechanical pulp. The factor seven-eighths holds good down to 35% of sulphite pulp, but below 35% the factor must be increased as the estimated percentage of sulphite falls. It is considered that an accuracy of 1% in either direction may be attained.—J. F. B.

PATENTS.

Wool, cotton, or like fibres; Machines for drying or carbonising —. J. Charlesworth, Huddersfield. Eng. Pat. 134,100, 11.12.18. (Appl. 20,560/18.)

In a drying or carbonising machine in which heated air is introduced at the bottom and rises through the material, which is fed in at the top and is conveyed through the machine by a series of creeper conveyors, a canopy is fixed over the delivery end in which the hot exhaust gases are collected and then conveyed by a pipe or conduit through the lower part of the machine to the feed orifice of the fan. The hot exhaust gases, mixed with sufficient cold air, are then re-heated and again introduced into the drying chamber.—J. F. B.

[Wool] piece or like scouring processes; Treatment of effluent from —. Distillates, Ltd., and G. G. Jarman, Kirkheaton, Yorks. Eng. Pat. 134,410, 11.12.18. (Appl. 20,559/18.)

THE effluent from woollen piece goods scouring machines is heated to boiling and allowed to cool; the soap from the sodium carbonate contained in the effluent either separates out naturally or is separated by centrifuging or filtering the cold liquor. If necessary the effluent is evaporated to a concentration sufficient to ensure the separation of the soap on cooling. The separated soda liquor is suitable for re-use in scouring.—J. F. B.

Wool and other fibrous substances; Machines used in the washing and scouring of — and for performing similar operations. W. Taylor, Huddersfield. Eng. Pat. 131,714, 5.2.19. (Appl. 2781/19.)

A DEVICE used for the immersion and carrying forward of the fibrous material consists of smooth glazed prongs of earthenware, porcelain, or other vitreous substance, resistant to acids and alkalis, fitted into a slotted bearing and secured by a plate bolted on to the bearing, or fixed by other means so as to be easily replaceable.—S. S. A.

Wool; Process for increasing the durability of —. Farb. vorm. Meister, Lucius, and Brünig, Höchst. Ger. Pat. 299,772, 2.10.15.

WOOL is treated at the ordinary temperature with salts of chromium or aluminium, particularly with the substances which are used in chrome tanning, such as chrome alum, chromium chloride and their basic salts. Alum or aluminium sulphate in the form of neutral or basic salts may also be employed. The treatment of the wool may take place by prolonged steeping or by padding or impregnating with more concentrated solutions, and may be carried out either on the loose wool or on piece goods. The treated wool possesses increased resistance to the action of air and light. (See also this J., 1919, 496A.)—J. F. B.

Silk; Process for degumming — by treatment with water under pressure. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 301,255, 16.1.16.

SMALL quantities of substances with an alkaline reaction are used along with the water. The degumming is satisfactorily performed without the use of soap, and the treated silk possesses the same lustre and feel as that degummed by the usual methods. Suitable alkaline substances are ammonia, sodium carbonate, caustic soda, sodium phosphate, and borax.—J. F. B.

Fabrics for aircraft. F. Sage and Co., Ltd., London, and N. A. T. N. Feary, Peterborough. Eng. Pat. 131,611, 4.11.18. (Appl. 18,064/18.)

PAPER or light textile material, coated on one side with a solution of casein, is applied to each side of an interlaced or woven wire fabric. The whole is compressed so that the paper or textile fabric on one side of the wire is depressed into the interstices between the wire to meet and adhere to that on the other side of the wire. The resulting material may be impregnated with oil or oil varnish or the like under pressure.—S. S. A.

Fireproofing for aircraft. P. R. Bradloy, East Orange, Assignor to Aircraft Fireproofing Corporation, Nutley, N.J. U.S. Pat. 1,321,708, 11.11.19. Appl., 22.3.18.

SEVERAL layers of protective substance, one of which contains alginic acid, are applied to the cloth or wood to be fireproofed.—S. S. A.

Porous substances such as textile fabrics, wood, etc.; Process for preserving —. Grubenholz-impregnierung Ges. m. b. H., Berlin. Ger. Pat. 299,761, 24.10.16.

THE material is treated with fluorosulphonic acid, either as such or as salts of metallic or organic bases, or in mixture with diluting substances, such as sodium sulphate or chloride. In the latter case the two dry salts may be mixed and compressed into tablets. Zinc fluorosulphonate gives a clear solution in water and does not form basic salts. Organic liquids such as creosote may be used as solvents for the fluorosulphonic acid or its salts.—B. V. S.

Non-inflammable fabrics; Process of impregnation for the production of —. W. Naher, Pforzheim. Ger. Pat. 299,773, 27.1.16.

THE fibre before spinning and weaving is sprayed with a solution of sodium stannate, applied in such a way that it dries at the moment of deposition. The impregnated material may then be treated with an aqueous solution of carbonic acid, washed, and dried. The operations may be repeated if necessary. Material so treated retains its normal elasticity, and does not lose its fire-resisting properties by washing.—B. V. S.

Hop-bines; Process for obtaining fibres capable of being spun and a tannin-like material from —. E. Reis, Heidelberg. Ger. Pat. 301,387, 15.9.16.

HOP-BINES are extracted with hot water and then treated with a dilute solution of sodium or potassium hydroxide, sulphite, or bisulphite or magnesium bisulphite, which removes certain pectin substances, leaving others which, by their glutinous properties, serve to strengthen the fibres. From the first aqueous extract, after fermenting and distilling off the alcohol, a tannin-like substance is recovered, which may be used in dyeing and tanning.—B. V. S.

Smooth, stiff vegetable fibres; Process for making — suitable for carding and spinning. M. Bohm und Sohn, Vienna. Ger. Pat. 305,577, 25.1.18.

STIFF, smooth fibres such as kapok, asclepias, and typha fibres are made soft and pliable by treatment with a dilute solution of pyridine bases, washing, and drying.—B. V. S.

Flax and hemp straws; Process for treating —. P. Püschel, Hilvetshof. Ger. Pat. 305,682, 1.11.17.

THE straw, after retting in water, is rinsed with warm water, preferably at 30° C., before being artificially dried, in order to remove as far as possible the viscous retting fluid adhering to the individual stems when they are subsequently pressed between squeezing rolls. In this manner, in spite of the artificial drying, a textile fibre and tow are obtained which are superior in colour and flexibility to the product obtained by natural drying. A higher temperature than 35° C. weakens the fibre under the squeezing operation, while water below 20° C. is not effective in removing the viscous fluid.—J. F. B.

Fibrous plants; Process for loosening the bast of —. Nessel-Anbau-Ges., Berlin. Ger. Pat. 307,144, 2.2.18.

PLANTS such as nettle, ramie, broom, hops, rushes, flax and the like, or portions of such plants comprising the bast, are treated with solutions of sugars under ordinary, reduced, or excess pressure. The use of sucrose, dextrose, maltose, and molasses is specified.—J. F. B.

Fibrous plants, particularly nettles; Process for obtaining the fibre of —. Nessel-Anbau-Ges. Ger. Pat. 312,381, 13.1.16.

THE plants are subjected in thin layers in a closed vessel to the action of superheated steam and then suddenly and strongly cooled. Superheated steam at 200° C. and 0.5 atm. pressure is used at first and is raised subsequently to 300° C. and 3 atm. pressure. The treatment with superheated steam and sudden cooling bring about a rapid and complete separation of the fibres without any deleterious effect on their textile quality.—J. F. B.

Wool substitute from cellulose and similar solutions; Manufacture of —. Glanzfäden A.-G., Petersdorf. Ger. Pat. 312,301, 15.7.19.

ARTIFICIAL filaments from plastic solutions, whilst being freed from the solvents and precipitating

bath chemicals which they contain, are kept under a tension which is induced by the kinetic energy of shower baths directed upon them. For example, spun threads in the form of hanks are carried through a series of chemical and washing shower baths in such a way that the removal of all chemicals takes place in a single passage. The gentle tension thus obtained is sufficient to prevent the thread from giving an opaque and brittle product, and the yarn is strong, soft, bulky, and possesses a slight lustre like wool.—J. F. B.

Cellulose or material containing cellulose; Manufacture of solutions of — A. G. Bloxam, London. From Zellstoff-fabrik Waldhof, Mannheim-Waldhof, Germany. Eng. Pat. 132,815. 12.10.17. (Appl. 14,806/17.)

In making solutions of cellulose by treating the material with a mixture of hydrochloric and sulphuric acids containing less than 39% HCl (Ger. Pat. 306,818 of 1917; this J., 1919, 131 a) a more rapid and complete solution is obtained by previously treating the cellulose with an alkali. For instance, 1 part of cellulose is kneaded with 5 parts of caustic soda solution of 17% strength for half-an-hour; water is then added, the alkaline liquor is separated, the cellulose is washed, scoured, washed again, and dried. The purified cellulose is then dissolved in a mixture containing 35% HCl and 10–12% H₂SO₄.—J. F. B.

Cellulose acetate; Manufacture of compositions, preparations, or articles having a basis of — H. Dreyfus, London. Eng. Pat. 133,353, 29.4.18. (Appl. 7193/18.)

Pure toluene-*o*-monomethylsulphonamide and pure toluene-*p*-monomethylsulphonamide are solid at the ordinary temperature, and are unsuitable as plastic-inducing solvents. Mixtures of toluene-*o*-monomethylsulphonamide or toluene-*o*-monoethylsulphonamide with some of the corresponding para-compounds are, however, suitable for this purpose. They may be used with or without triphenyl or tri-cresyl phosphate and monomethylurea, etc., for the production of plastic cellulose acetate products. —L. L. L.

Cellulose [acetate] compound; Non-inflammable — W. G. Lindsay, Newark, N.J., Assignor to The Celluloid Co. U.S. Pat. 1,319,229, 21.10.19. Appl., 9.3.18.

ACETYLCELLULOSE is mixed with phenyl salicylate and a solvent common to both.—J. F. B.

Celluloid, vulcanite, or the like; Substitutes for — W. T. Robinson-Bindley, A. W. Weller, and E. Dulcken, London. Eng. Pat. (a) 134,564 and (b) 134,565, 27.2.18. (Appls. 3502 and 3503/18.)

(a) An infusible celluloid or vulcanite substitute, insoluble in acids, spirits, oils, or petrol, is obtained by condensing *o*-, *m*-, or *p*-cresol with formaldehyde or an equivalent quantity of a substance adapted to yield formaldehyde, in the presence of a neutral sulphate at 60°–90° C. according to whether a soft or hard product be desired. The reagents (7 parts by weight of cresol and 5 parts by weight of formaldehyde) may be brought together in the gaseous form, preferably under a pressure of 10–20 lb. per sq. in., the cresol being volatilised by a current of steam, and sufficient sodium sulphite being packed into the reaction tube to ensure contact with the reacting gases. Alternatively, formaldehyde, 50 parts, or volatilised paraformaldehyde may be blown into *o*- or *m*-cresol, 50 parts, maintained at 60° C. in the presence of 5–10 parts of the catalyst dissolved in a small quantity of the formaldehyde solution until a treacly mass is formed. The material may be hardened by heating in an autoclave at

140°–160° C. at a pressure of 200 lb. per sq. in., or on the water-bath at 60°–80° C. without pressure. Fillers may be incorporated with the material whilst it is still in the semi-molten condition prior to its being placed in the hardening moulds. (b) Substitutes for celluloid, vulcanite, or the like are obtained by incorporating camphene and nitrocellulose or cellulose acetate or mixtures of these or of camphene and celluloid with the material obtained by any known process for the condensation of phenol or cresols (without separation of the individual cresols) with formaldehyde or by the processes described under (a) or in Eng. Pat. 134,563 (page 35 a).—A. de W.

Pyroxylin composition and process of making same. W. F. Doerflinger, Staten Island, N.Y. U.S. Pat. 1,320,458, 4.11.19. Appl., 6.12.18.

A COATING composition is made with a non-drying oil and pyroxylin dissolved in a volatile solvent containing diacetone-alcohol, *n*-butyl alcohol, a volatile ketone, and benzene.—J. F. B.

Pyroxylin solvent and composition containing the same. E. M. Flaherty, Parlin, N.J., Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,321,611, 11.11.19. Appl., 16.3.18. Renewed 24.6.19.

A NITROCELLULOSE composition is treated with a solvent consisting of a mixture of ethyl acetate, butyl alcohol, and benzene.—S. S. A.

Pyroxylin bodies; Process of producing — Solvent for pyroxylin bodies. M. V. Hitt, Parlin, N.J., Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pats. 1,321,633 and 1,321,634, 11.11.19. Appl., 10.12.17. Renewed 24.6.19.

PYROXYLIN is dissolved in a mixture of ethyl acetate, benzene, and ethyl alcohol, and the solvent mixture is evaporated.—S. S. A.

Millboard or the like; Process for impregnating articles of — to render them waterproof. Apparatus for impregnating articles of pulp- or straw-board. E. Slight and A. D. Lacy, London. Eng. Pats. 134,010 and 134,176, 21.10.18. (Appls. 17,165/18 and 12,880/19.)

THE articles are first partially impregnated with a solution of casein, to which formaldehyde may be added if desired, in order to stiffen them and economise the consumption of the waterproofing composition, subsequently applied in the form of a mixture of wax and resin. In some cases the casein may be combined in the same bath with the wax and resin. The articles after immersion in the bath are carried by a conveyor from one end to the other of a heated impregnating chamber, wherein the superfluous solution is collected and returned to the bath. The chamber is heated by means of a furnace, the products of combustion from which pass through a flue in the inclined hollow bottom of the impregnating chamber, throughout its entire length, and thence through uptakes into the chamber itself. The gas-heated impregnating bath is situated at the lower end of the inclined chamber, and the products of combustion from this are likewise delivered into the chamber. The endless conveyor is preferably constructed of metal slats, the edges of which are turned over and joined together by wire links to form an endless band.—J. F. B.

Paper; Apparatus for making — J. T. Murphy and E. J. Raney, Assignors to J. L. Carey, Chicago, Ill. U.S. Pat. 1,318,900, 14.10.19. Appl., 30.1.19.

Two heating engines are provided at different levels, the lower one being operated intermittently

and the higher one continuously. A stuff-chest, having a capacity greater than that of the intermittent bending engine, is situated at an intermediate level, and means are provided for conveying stock from the intermittent engine to the stuff-chest and from the stuff-chest to the continuous bending engine.—J. F. B.

Paper; Process of removing mineral matter from — V. Nakamura, Assignor to Nippon Sen-I Kogyo Kabushiki Kaisha, Tokyo. U.S. Pat. 1,318,902, 14.10.19. Appl. 9.7.18.

PAPER is subjected to the action of a solvent consisting of *aqua regia* containing an excess quantity of nitric acid and hydrofluoric acid for the removal of mineral matter.—J. F. B.

Paper pulp; Process and apparatus for treating — G. C. Howard, Tacoma, Wash. U.S. Pat. 1,322,489, 18.11.19. Appl. 8.5.17.

A dilute mixture of pulp and water is caused to impinge upon the lower part of one side of a pair of rapidly rotating rolls disposed within a vat of semi-circular cross-section, whereby the pulp is drawn between the rolls and a homogeneous mixture is discharged at the opposite side of the vat.

—W. E. F. P.

Paper-yarn; Process for strengthening — O. Ruff, Breslau. Ger. Pat. 302,551, 6.7.17.

THE paper, before spinning, is soaked in a previously heated solution of crude cresol, formaldehyde and some alkali, either on the spinning machine or in a separate process. A larger proportion of alkali is required for a sulphite-pulp than for a sulphate-pulp paper. After spinning, the yarn or the material woven therefrom is heated for some time at 80°–110° C.—B. V. S.

Paper, fabrics, and the like; Process for the sizing, waterproofing, dressing, etc. of — W. Schmidt, Elberfeld, and E. Heuser, Darmstadt. Ger. Pat. 305,525, 12.7.17.

THE paper pulp or the fabric is treated with tar from lignite or bituminous coal either in a fine state of division or in the form of a tar soap from which the tar is precipitated on the fibres by the addition of acid, formaldehyde or the like. The adhesion of the tar to the fibre can be increased by the addition of resin, casein, or animal size. The tar can be resinated by the action of formaldehyde etc., and the coloration of the paper pulp can be modified by bleaching agents.—B. V. S.

Paper; Process for sizing — by the aid of montan wax. P. Klemm, Gautzsch. Ger. Pats. (a) 305,678, 12.1.17, and (b) 310,076, 8.5.17.

(a) A STABLE emulsion of montan wax, suitable for sizing paper, is obtained by heating it with a small quantity of resin soap or naphthenic acid soap in alkaline solution. (b) Liquid resin (*Tallol*) obtained from soda-cellulose waste lyes (this J., 1916, 1163) is saponified and used as the emulsifying agent, somewhat less than 25% of the quantity of montan wax being required.—B. V. S.

Paper and the like; Process for sizing — Feldmühle Papier und Zellstoffwerke A.-G., Berlin. Ger. Pat. 307,087, 30.11.17.

FIBROUS materials or finished products prepared from them are treated with solutions of the organic matter (lignin) from waste sulphite liquors rendered precipitable by treatment with alkalis, and the size is precipitated on the fibre by the addition of acids or suitable salts. The lignin size may be used in admixture with other known paper-sizing agents. By heating with alkalis the sulphonic group is almost completely eliminated from the lignin-sulphonic acid and a dark brown alkaline solution

of lignic acid is obtained, which gives a voluminous precipitate with acids or salts. About 10–20% of caustic soda, calculated on the dry substance of the sulphite waste liquor, is required. Alternatively, the alkaline liquor may be neutralised with acid, precipitated by alum or kieserite, and used for sizing in the proportion of 2 —J. F. B.

Paper; Manufacture of hard-sized — O. Ruff, Breslau. Ger. Pat. 313,112, 25.1.18.

PAPER prepared with the addition of vegetable mucilages or with substances capable of yielding mucilages, or paper made from pulp which has previously been treated with acids or acid salts (see Ger. Pats. 309,999, 311,772, and 312,179; this J., 1919, 281A, 625A, 896A) is subjected to a heating treatment. Example: About 10–30% of powdered peat calculated on the dry substance is added to the pulp in the hollander, and after the paper has been made it is heated at 120° C. for about 2 hours.

—J. F. B.

Cleaning or polishing material [*"Putzwolle"*]. Reis and Co., Friedrichsfeld. Ger. Pat. 303,302, 26.5.17.

WASTE from paper industries, more particularly from paper spinning and weaving, is softened and made absorbent by treatment with alkalis, inorganic or organic acids, sulphurous acid, or chlorine compounds, and then bleached and worked up into "cleaning waste" in the usual way.—B. V. S.

Coating or impregnating material; Method and apparatus for continuously — O. Minton, Short Hills, N.J. U.S. Pat. 1,322,327, 18.11.19. Appl., 22.7.19.

MATERIAL in the form of a strip is passed over rollers into and through a liquid seal which has no action on the material. The liquid seal is in the form of a U-tube and forms the inlet to a vacuum chamber through which the material then passes over guide rollers. The material passes out of the vacuum chamber through another U-tube containing the coating or impregnating material.

—W. F. F.

Waste liquors from cellulose manufacture or other similar waste liquors; Dry distillation of — with strong bases in the presence of steam. E. L. Rinman, Djursholm, Sweden. Eng. Pat. 120,721, 30.10.18. (Appl. 17,718/18.) Int. Conv., 8.11.17.

WASTE liquors from the manufacture of cellulose from wood, straw, or other materials by the soda process, waste liquors produced by boiling vegetable matters with caustic soda until the cellulose is dissolved, or waste liquors from sulphite pulp manufacture, after boiling with lime, are mixed with suitable proportions of caustic soda and lime, the mixture is concentrated by evaporation until it contains about 25% of water and then subjected to destructive distillation, preferably by means of hot gases in the presence of superheated steam. The temperature is maintained below 200° C. until the water has been substantially driven off, then between 200° and 300° C. until ammonia, wax, and methyl alcohol have distilled over, and, lastly, between 300° and 500° C. during the formation of acetone and, later, of light oils and heavy oils. The proportions of bases to be added per 100 parts of organic matter range between 10 and 65 parts Na₂O and between 30 and 40 parts CaO.—J. F. B.

Lyes from soda- or sulphite-cellulose process; Dry distillation of waste — E. L. Rinman, Stockholm. Ger. Pat. 313,607, 1.9.15.

THE use of milk of lime instead of finely-powdered slaked or quicklime results in a 30% increase in the yield of acetone. Waste lye from the soda-

cellulose process, for example, is concentrated to 30° B. (sp. gr. 1.26) and mixed with a quantity of milk of lime corresponding to 400 kilos. CaO per 1000 kilos. wood. This mixture is concentrated to 55° B. (sp. gr. 1.62) and then distilled with superheated steam, the temperature being slowly raised to 500° C. The yield is about 40 kilos. of acetone and 50 kilos. of oil per 1000 kilos. of wood.—W. P.

Lignin substances insoluble in acids and suitable for sizing paper; Process for obtaining — from the ligninsulphonic acids of sulphite-cellulose waste lyes. Feldmühle Papier und Zellstoffwerke A.-G., Berlin. Ger. Pat. 307,663, 27.1.18. Addition to Ger. Pat. 307,087 (see preceding abstract).

THE liquor containing the ligninsulphonic acids is poured into a hot concentrated caustic alkali solution and the mixture heated until the interaction is complete. The reaction is quicker, more certain and more convenient in operation in this way than if the alkali solution is added to the sulphite liquor. The lignin substances are separated from the resulting solution by treatment with acid, and are redissolved in alkali for use in paper-sizing.

—B. V. S.

Paper-pulp; Machines for straining —. Watford Engineering Works, Ltd., and J. Paramor, Watford. Eng. Pat. 134,923, 13.11.18. (Appl. 18,587/18.)

Paper-making machines and the like [; Driving gear for —]. W. A. Aitken, Gravesend. Eng. Pat. 135,259, 15.11.18. (Appl. 18,728/18.)

Cleaning and polishing powder [from cellulose hydrate] applicable for use in dentifrices, metal-polishes, and the like. L. V. Slight, Assignee of F. W. Spies, New York, and H. D. Pease, Richmond Hill, N.Y., U.S.A. Eng. Pat. 121,592, 12.11.18. (Appl. 18,510/18.) Int. Conv., 20.12.17.

SEE U.S. Pat. 1,275,779 of 1918; this J., 1918, 651 A.

Sea-tang; Method of treating — [for paper manufacture etc.]. V. A. Frydensberg, Tisvildeleje, Denmark. U.S. Pat. 1,322,237, 18.11.19. Appl., 9.4.18.

SEE Eng. Pat. 120,761 of 1917; this J., 1919, 39 A.

Rubber from fabrics etc. U.S. Pats. 1,321,200—1. See XIV.

Fermentation of cellulose. Eng. Pat. 134,265. See XVIII.

Ethylidene esters. Ger. Pat. 313,696. See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Vegetable fibres; Elimination of nitrogen from — in the bleaching process. S. H. Higgins. J. Soc. Dyers and Col., 1919, 35, 164—169.

By repeatedly distilling 30 grms. of air-dried yarn in a liquor containing 4% of caustic soda on the weight of material and titrating the ammonia evolved with N/10 sulphuric acid, American and Egyptian cotton yarn and linen yarn were found to contain 0.180, 0.275, and 0.420% of nitrogen respectively. Comparing the protein values calculated from these results with the figures given by Osborne for typical proteins, the author infers that the nitrogen in cotton is not all present as protein and that the elimination of proteins from textile fibres during bleaching cannot be measured by simple nitrogen determinations. Lime hydrolyses

the proteins, giving results similar to those obtained with caustic soda. American yarn was subjected to treatments imitative of large-scale bleaching operations, then well washed and distilled with sodium hydroxide. It was found that the yarn was freed almost completely from protein nitrogen by boiling with caustic soda or soda ash, but that of the residual 8.3% of nitrogen practically none was removed by a second alkali boil. The products of lime hydrolysis are only partly soluble and resist removal by washing with water. In the case of linen the proteins are removed effectively by caustic soda or scouring (i.e., lime boil, sour, and soda ash boil), but only partially by soda ash. The author therefore maintains that, contrary to the statement of Cross, Bevan, and Briggs (this J., 1908, 262), cotton or linen when scoured with caustic soda or with lime boil, sour and soda ash boil, are incapable of forming chloramines on treatment with bleaching-powder solution because they no longer contain appreciable quantities of proteins.—S. S. A.

Permanganate; Bleaching with —. W. Kind. Z. ges. Textilind., 1919, 22, 246—247, 255—256. Chem. Zentr., 1919, 90, IV., 412.

IN bleaching with permanganate it is necessary to work in acid solution, using sulphuric acid and on no account hydrochloric acid, which develops free chlorine and attacks the cellulose very severely. The alkalinity may be reduced by magnesium sulphate, but its action is not quite satisfactory, and in some cases the strength of the bleached goods is impaired; moreover, the oxygen is not so completely utilised as in an acidified bath. Strong standing baths of permanganate are dangerous, since local attacks on the fibre may be induced by residues of alkali.—J. F. B.

Active oxygen in bleaching and laundry preparations; Efficiency of —. A. Grün and J. Jungmann. Seifenfab., 1919 39, 69—73. Chem. Zentr., 1919, IV., 90, 757—758.

THE authors have worked out a method for the determination of the loss of active oxygen in laundry processes, and the efficiency of the methods of treatment can thus be established. The active oxygen remaining in the liquid after use can be determined by permanganate, and this added to the amount of gaseous oxygen generated by the treatment shows the total consumption as compared with the quantity of active oxygen originally present. The method of estimation consists in treating a fabric in a special apparatus (this J., 1918, 729A) with the solution of the bleaching agent and collecting and measuring the gaseous oxygen evolved. In one set of trials a preparation consisting of a mixture of soap, soda, sodium silicate and perborate was used, the solution containing 0.005% of active oxygen. It was important to establish the influence of the hardness of the water. For this purpose an unbleached, raw cotton fabric was used and trials were carried out with waters ranging from 0° to 14° (German) hardness. The direct losses of active oxygen were found to be inversely proportional to the hardness of the water. With almost completely softened water the loss amounts to one-half or more; with waters of 3°—6° of hardness it is from one-fourth to one-third, and with harder waters it becomes negligible. If, however, any traces of heavy metals or other foreign matters are present, the losses are considerably larger. The consumption of active oxygen by the fabric is comparatively independent of the hardness of the water. Experiments with dirty fabrics also showed that the loss of active oxygen was considerably greater when using soft water than with hard waters. In the case of soft water the active oxygen was always practically completely discharged,

whether clean or dirty cloth or no cloth at all was used. The constituents of hard waters stabilised the perborate, so that in blank experiments the major portion of the active oxygen remained in the bath, and with clean or fairly clean fabrics about one-third remained; only in the case of dirty goods was practically the whole of the oxygen discharged. Of this total quantity two-thirds was utilised, and one-third was lost. The liberation of part of the active oxygen in the form of gas is regarded as an unavoidable side-reaction which runs parallel with the bleaching action. From the difference between the oxygen consumption of dirty and clean fabric, the true efficiency of the oxygen consumption by the dirt was found to be about 20%.

—J. F. B.

Chromed dyeings; Fastness to fulling of —. A. Reyl. *Färber-Zeit.*, 1919, 30, 237—238.

COMPLAINTS are sometimes made in connection with dyestuffs intended to show good resistance to fulling when dyed on wool after-treated with chromium, and it is found that the colour bleeds out during the fulling operation. The cause of the trouble generally lies in the material to be dyed. For instance, re-manufactured wool is very frequently present in large quantities, and if this material has previously been dyed with a colour which is not fast, it will not be fast to fulling even when topped with the mordant dyestuff. The material should be tested by vigorously washing a piece with sodium carbonate or soap, and if any of the colour comes out of the shoddy, it must be treated before dyeing with the chrome dyestuff. Such treatment may consist in working the goods in a lukewarm bath of sodium carbonate until no more colour bleeds out in the test. Preferably, however, since the alkali damages the fibre, the colour of the shoddy should be taken out by a stripping agent. The goods must then be thoroughly rinsed before dyeing. The use of a stripping agent instead of soda presents the advantage that it leaves the goods much lighter in colour and capable of taking clearer and paler shades. If the fault cannot be traced to the presence of shoddy, the defective fastness of the chrome-mordanted wool to fulling may be due to the presence of grease, owing to the goods having been insufficiently scoured. The grease obstructs the penetration of the dye solution into the fibre and causes the colour to be deposited only superficially; such unfixed dyestuff will readily come out during fulling.—J. F. B.

Strength of cotton. Greenwood. See V.

Colouring paper pulp. Püschel. See V.

PATENTS.

Bleaching; Composition for and process of —. J. F. King, Assignor to H. B. Haines, Philadelphia, Pa. U.S. Pats. 1,321,643 and 1,321,644, 11.11.19. Appl., 28.6.19.

The goods to be bleached are introduced into a solution of sodium chloride, potassium hydroxide, sodium carbonate, and hydrogen peroxide in water, the temperature is raised, and the bleached material is subsequently rinsed.—S. S. A.

Silk; Method of loading —. Deutsche Gasglühlicht A.-G. (Auerger), Berlin. Eng. Pat. 116,102, 22.5.18. (Appl. 8517/18.) Int. Conv., 22.5.17.

The fibre is dried at a low temperature, after saturating with tin chloride and centrifuging, and treated with alkaline gases, such as ammonia or methylamine. In order to avoid damage to the silk the gases may be diluted with air or indifferent gases. The loading thus obtained is two and a half times as much as that usually obtained. The rinsing baths are clear and without precipitate. The loaded fibre may be further treated in known

manner with phosphates and silicates, and the handle, lustre, and firmness of the silk are the same as are obtained by the usual process. Tin salts may be replaced by other salts, for example, chromium salts.—L. L. L.

Dyeing certain colours on vegetable fibres, yarns, and fabrics. V. H. Gatty, Preston. Eng. Pat. 133,620, 16.4.19. (Appl. 9708/19.)

The material is impregnated with solutions of salts of copper and manganese, dried by hydro-extraction, squeezing rollers, or the like, and then passed through a hot alkaline bath. The alkaline bath may consist of hydroxides, silicates, or carbonates of sodium and potassium, either singly or in combination. The shades range from pale olives, yellow browns and fawns, deep olives, tobacco browns, to full dark chocolate shades.—L. L. L.

Sulphonic acids of xylene and solvent naphtha; Utilisation of — [for production of foam baths for dyeing]. Meister, Lucius, u. Brüning, Höchst. Ger. Pat. 312,867, 4.5.17.

The sulphonic acids of higher aromatic hydrocarbons or the resinous substances obtained by the action of sulphuric acid and formaldehyde on these hydrocarbons may be used to promote the production of foam baths for dyeing. The viscous, tough or hard resinous masses are easily sulphonated in the cold with weak oleum. The sodium sulphates are soluble in water, and the solutions form an abundant and tenacious froth. This occurs not only with neutral and alkaline solutions, but also with acid solutions. To produce a good foaming dye bath, about 5 grms. per 1 litre of liquor is necessary.—H. J. H.

Hop-bines. Ger. Pat. 304,387. See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Chlorine cells [Electrolytic] —. F. G. Wheeler. Chem. and Met. Eng., 1919, 21, 436—438.

As the cheapest and most adaptable cell the author adopted a type of the form used by Hargreaves in 1894, cylindrical in shape and built so that the entire exterior surface is the cathode. To minimise plugging and capillary action of the diaphragm, a thin sheet diaphragm was devised which gave an efficiency approximating to unity over long periods of time. This cell can be shut down without damage, because, on re-starting, as the cell becomes warmer the viscosity of the brine decreases and it flows readily through the diaphragm, removing the ferric hydroxide precipitated in the pores of the diaphragm by the stoppage. Steam blown into the cathode compartment keeps the cell hot, thus promoting the flow of brine through the diaphragm, reducing the concentration of the hydroxyl ions, and thereby increasing the efficiency of the cell, but its use is not economical if power is cheap, on account of the cost of re-concentrating the solution, which becomes diluted by condensed water.—S. S. A.

Bucher process [for the fixation of nitrogen]; Equilibrium conditions in the —. J. B. Ferguson and P. D. V. Mannig. J. Ind. Eng. Chem., 1919, 11, 946—950.

The reaction which takes place on heating a mixture of sodium carbonate, carbon, and iron in an atmosphere of nitrogen in Bucher's process (this J., 1917, 451) is represented by the equation— $\text{Na}_2\text{CO}_3 + 4\text{C} + \text{N}_2 \rightleftharpoons 2\text{NaCN} + 3\text{CO}$. Since it is probable that the equilibrium constants in the reaction cannot be experimentally determined, an attempt has been made to determine certain

empirical relations which may be regarded as a measure of the actual conditions of equilibrium. For this purpose determinations were made of the variations in the amounts of alkali in a charge which at the equilibrium point was converted into cyanide, with known variations in the stream of gas passing over the charge. Experiments at 950°–1000° C. with concentrations of carbon monoxide up to 80% did not cause any change in an iron boat in which the charge was placed, which confirmed the statement of Hilpert and Dieckmann (this J., 1915, 964) that iron is stable at these temperatures in the presence of equilibrium mixtures of carbon monoxide and dioxide. The percentages by weight of sodium carbonate converted into cyanide in the series of experiments were plotted against the carbon monoxide content of the original gas, it being assumed that no great error is introduced by unknown reactions of the carbon monoxide at very high or very low concentrations. From the curves thus obtained the following deductions with regard to commercial cyanide nitrogen-fixation processes were drawn: The temperature has relatively little influence upon the possible yield of cyanide in the case of low concentrations of carbon monoxide, but would have considerable influence if producer-gas containing e.g. 30% CO were used. Using producer-gas, a conversion up to 63% can be obtained at 1000° C., and this yield would probably be increased by raising the temperature. The presence of 15% carbon monoxide in the initial gas mixture reduces the amount of conversion by 30% compared with that given by pure nitrogen, whilst 60% reduces it by 50%. If the initial gas contains relatively large amounts (25–30%) of carbon monoxide slight variations in the proportions of that constituent have little effect upon the yield. It is probable that the controlling reaction in the Bucher process is the primary reduction of the sodium carbonate by carbon to form sodium, and the effect of the carbon at this stage may be merely to ensure that the carbon dioxide pressure is maintained well below the dissociation pressure of the carbonate.

—C. A. M.

Ammonium sulphate and potassium nitrate; Production of — from potassium salts. H. Hampel. Chem.-Zeit, 1919, 43, 617–619, 634–636.

The author outlines two continuous processes for the manufacture of ammonium sulphate by utilisation of the combined sulphuric acid present in the vast quantities of German potassium salts which are available. In the first process, in which ammonia and synthetic nitric acid are utilised, the reactions are explained by the equations: (1) $K_2SO_4 + Ca(NO_3)_2 = 2KNO_3 + CaSO_4$; (2) $CaSO_4 + 2NH_3 + CO_2 + H_2O = CaCO_3 + (NH_4)_2SO_4$; (3) $CaCO_3 + 2HNO_3 = Ca(NO_3)_2 + CO_2 + H_2O$. The by-products, such as potassium nitrate, obtained in this process are more valuable than the initial materials. In the second process ammonium sulphate and magnesium oxide are produced and the following reactions occur: (1) $MgSO_4 + CaCl_2 = MgCl_2 + CaSO_4$; (2) $CaSO_4 + 2NH_3 + CO_2 + H_2O = CaCO_3 + (NH_4)_2SO_4$; (3) $CaCO_3 + 2HCl = CaCl_2 + H_2O + CO_2$; (4) $MgCl_2 + H_2O = MgO + 2HCl$. A description of suitable plant is given. Comparisons of estimated costs of these processes with the cost of the ordinary process using sulphuric acid are quoted.

—W. J. W.

Calcium carbide; Action of bromine on —. E. Barnes. Chem. News, 1919, 119, 260–261.

4.5 GRMS. of finely-powdered calcium carbide left in contact with 45 grms. of dry bromine in a 50 c.c. sealed bottle for five months gave a product which, after removal of excess of bromine, weighed

32.4 grms. and contained 22 grms. of hexabromoethane, 8.8 grms. of calcium bromide, and 0.2 gm. of unchanged carbide. Calcium carbide and bromine heated in sealed tubes to 100° C. until no further action was apparent produced carbon and calcium bromide. Dry chlorine was found to have no action on calcium carbide exposed to it at the ordinary temperature for two months.

—S. S. A.

Sodamide; Autoxidation of —. H. Schrader. Z. anorg. Chem., 1919, 108, 44–48.

WHEN exposed to air the amides of the alkali metals undergo autoxidation with formation of nitrite, hydroxide, and ammonia. When finely-divided sodamide is exposed to air in presence of a little water a yellowish-red product is formed, which has now been shown to be a peroxide, probably of the formula $NaNH_2O_2$. After 58 days a sample was found to contain 0.44 mol. % of peroxide and 6.9 mols. % of nitrite calculated on the sodamide taken. The analysis was carried out by adding the product to a saturated solution of barium chloride and filtering off the precipitated barium peroxide, the nitrite being determined in the filtrate. In dry air autoxidation does not take place at the ordinary temperature, but at 100°–110° C. it proceeds slowly, 9.5 mols. % of peroxide being formed in the course of 170 hours. The peroxide is stable in dry air, but in moist air is changed into a white substance, the aqueous solution of which gives the peroxide reaction.

—E. H. R.

Barium nitrate; Reduction of — by the alternating current. P. Wenger and A. Lubomirski. Ann. Chim. Analyt., 1919, 1, 339–342.

THE reduction of barium nitrate in aqueous solution increases with the amperage of the alternating current, except when mercury electrodes are used, in which case a maximum yield of nitrate is obtained with 0.6 amp. Increase of temperature increases the yield with lead electrodes, decreases it with aluminium and zinc electrodes, and has but little effect in the case of copper, cadmium, or magnesium electrodes. Using mercury electrodes and a current of 0.4 amp., 71% of the nitrate is reduced in 5 hrs. The oxygen liberated from the nitrate combines with the electrodes forming suboxides (in the case of copper, mercury, cadmium, or tin electrodes), hydroxides (with lead, zinc, aluminium, or magnesium electrodes), or oxides (with nickel or silver electrodes). Modification of the surface of the electrodes affects the yield of nitrite. The metallic electrodes do not in themselves reduce the barium nitrate.—W. P. S.

Ferrocyanides; Electrometric method for the determination of — depending on a change in oxidation potential. G. L. Kelley and R. T. Bohn. J. Amer. Chem. Soc., 1919, 41, 1776–1783.

SOLUTIONS of ferrocyanides may be estimated by titration with potassium permanganate in the presence of sulphuric acid, using the sudden change in EMF observed with a bright platinum electrode to mark the end point. About 1 gm. of potassium ferrocyanide in 250 c.c. of water is treated with 2.5–5.0 c.c. of sulphuric acid (sp. gr. 1.58) and titrated with 0.05N potassium permanganate, using an electrometric apparatus which will indicate changes of potential of one millivolt. A small precipitate may form during the titration, but this will re-dissolve. The titration should be performed slowly with mechanical stirring. As the end point is approached the permanganate should be added drop by drop and time allowed for the completion of the reaction. The end point is taken as the point of greatest change in potential for equal additions of permanganate. Should too much perman-

ganate be added, the end point may be approached from the other side by adding a standard solution of ferrocyanide. The presence of ferricyanides does not interfere with the titration, nor do amounts of chloride up to 1 gm. of sodium chloride. Any salts which under the conditions of experiment produce a precipitate with either ferrocyanide or ferricyanide seriously interfere with the method.

—J. F. S.

Aluminium phosphate; Soluble —. O. Svanberg. Z. anorg. Chem., 1919, 108, 70—72.

WHEN a few drops of a 1—2% solution of potassium dihydrogen phosphate are added to a neutral solution of aluminium chloride, both being previously coloured yellow by the addition of a drop of methyl orange solution, the colour of the mixture immediately becomes red without the formation of any precipitate or turbidity. On warming aluminium phosphate is precipitated, but there is no further colour change. It is shown that the hydrogen ion concentration for mixtures of the two salts reaches a maximum with about 40 mols. % KH_2PO_4 , this accounting for the colour change. Salts of calcium, magnesium, zinc, or cadmium do not give the reaction. Beryllium sulphate behaves like aluminium chloride, but is more sensitive to excess of the phosphate.—E. H. R.

Permutit; Basic exchange in —. V. Rothmund and G. Kornfeld. Z. anorg. Chem., 1919, 108, 215—225.

THE previous work (this J., 1918, 559 A) has been extended to include cases of exchange between divalent and trivalent ions in permutit. For the case of divalent ions the exchange was studied between copper-permutit and the nitrates of magnesium, calcium, strontium, and barium. The equilibrium is independent of dilution, and the tendency to permutit formation increases with the atomic weight of the metal. When the exchange is between ions of different valency, as between silver-permutit and barium nitrate or lanthanum nitrate, dilution has considerable influence on the equilibrium. (See also J. Chem. Soc., Jan., 1920.)

—E. H. R.

Kaolinite and other silicates; Constitutional formulae of —. R. Wegscheider. Z. Elektrochem., 1919, 25, 352.

THE constitution of polysilicates is such that the silicon atoms are never directly combined, but always combined through oxygen. (See also J. Chem. Soc., Jan., 1920.)—J. F. S.

Lead oxides; Determination of volatile matter in —. O. Andersen. J. Amer. Ceram. Soc., 1919, 2, 782—783.

IF the oxide contains no appreciable amount of metallic impurities a weighed amount is mixed with ground quartz which has previously been calcined at 100°C ., in the proportion of 3 parts of PbO to 1 part of silica, and placed in a narrow platinum crucible. A thin layer of silica is placed on top and the crucible with its contents is weighed, ignited at 800°C . for 26 mins. and then at 1000°C . for 15 mins. The eutectic formed melts nearly 100°C . below the melting point of pure lead monoxide (880°C .), and does not attack platinum. The crucible is covered, allowed to cool, and is then re-weighed, the loss being the volatile matter present in the lead oxide. The amount of pure PbO is found by difference.—A. B. S.

Arsenic trichloride; Action of acetylene on —. O. A. Dafert. Monatsh. Chem., 1919, 40, 313—323.

ACETYLENE does not react to any considerable extent with arsenic chloride at the ordinary temperature

or at the boiling point of the latter; in the presence of anhydrous aluminium chloride at the ordinary temperature, however, diacetylene arsenic chloride, $\text{AsCl}_3 \cdot 2\text{C}_2\text{H}_2$, is readily formed as a heavy yellow oil, sp. gr. 1.6919 at 15°C ., b.p.t. 250°C . The vapours are strongly irritating but not markedly poisonous, though exhibiting strong bactericidal power. When compared with the corresponding aluminium and antimony compounds, the arsenic derivative exhibits a remarkable stability, since it can be distilled and is not decomposed by water. It evolves acetylene when heated with potassium hydroxide solution. At a higher temperature arsenic trichloride and acetylene in the presence of aluminium chloride yield a black, organo-arsenic compound of high molecular weight, which is very stable towards reagents but sensitive to light, and which, in its properties, resembles the aluminium derivative prepared by Baud in a similar manner.

—H. W.

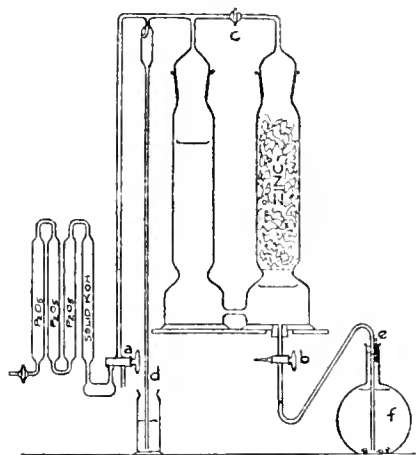
Bromine in mineral waters and brines; Determination of —. W. F. Baughman and W. W. Skinner. J. Ind. Eng. Chem., 1919, 11, 951—959.

THE water or brine, rendered alkaline if necessary with sodium carbonate, is evaporated to dryness, and the residue transferred to a reaction vessel which is connected to two absorption vessels, the first containing 20 c.c. and the second 5 c.c. of a solution of 1 gm. of sodium sulphate and 0.2 gm. of sodium carbonate in 25 c.c., in each case diluted to 200 c.c. The reaction vessel contains glass beads and the residue from the mineral water is distributed over these. After the introduction of a solution of 15 grms. of chromic anhydride in 10 to 12 c.c. of water, the evaporation basin is washed out with water, and the washings (about 25 c.c.) also introduced. Air is then aspirated through the apparatus until the saline material in the reaction vessel is in solution, when the apparatus is closed, and allowed to stand overnight. A current of air (about 0.25 to 0.5 litre per min.) is then drawn through it for 3 hours, while four successive portions of 2 c.c. each of 3% hydrogen peroxide are added at intervals of 30 mins. (to accelerate the evolution of bromine). The contents of the two absorption vessels are evaporated nearly to dryness, and the residue returned together with 25 c.c. of water to the reaction vessel, which has meanwhile been cleaned and recharged with glass beads and 15 grms. of chromic anhydride. The two absorption vessels are charged with 10 grms. and 3—4 grms. respectively of potassium iodide in 200 c.c. of water, a slow current of air drawn through the apparatus for about 1 hour, and the liberated iodine titrated. In the first aspiration the bromine becomes concentrated, whilst in the second treatment with chromic anhydride so little chlorine is present that pure bromine is evolved. (See also J. Chem. Soc., Jan., 1920.)—C. A. M.

Hydrogen of high purity; Preparation and testing of —. J. D. Edwards. J. Ind. Eng. Chem., 1919, 11, 961—963.

THE apparatus shown in the diagram obviates the drawbacks of the ordinary Kipp generator. Pure hydrogen is at once obtained and fresh acid can be introduced without admitting air. The apparatus is exhausted through *a*, whilst the tube, *d*, which dips into mercury, serves as an indicator of the residual pressure and as a safety valve. The acid flask, *f*, is nearly filled with sulphuric acid (1.8) and a few fragments of zinc, so that the hydrogen evolved will sweep out all air. The vent *c* is then closed until the pressure of the gas is sufficient to force the acid into the tube, to free the connections from air. The tube beyond the tap, *b*, enters the generator through a rubber stopper

which is always under liquid. The rate of generation of the gas is controlled by the tap, *c*. Tests made by means of the interferometer (this J., 1915,



638) in comparison with pure hydrogen derived from the electrolysis of barium hydroxide, showed that the hydrogen generated by this apparatus contained less than 1:10,000 parts of impurities. Weaver's colorimetric test for acetylene (this J., 1916, 347) indicated not more than 1 or 2 parts per million. No arsenic or antimony was found. Tests of the hydrogen from a Kipp generator, by means of the interferometer, showed that it contained 83.86% H₂, after 17 mins., 93.59% after 22 mins., and 99.97% after 42 mins.—C. A. M.

Lead chromate. Gröger. See XIII.

Colour reactions of molybdenum. Barbieri. See XXIII.

PATENTS.

Concentrating solutions [sulphuric acid]; Method of —. H. V. Welch, Los Angeles, Cal., U.S.A. Eng. Pat. 134,593, 30.10.18. (Appl. 17,698/18.)

SEE U.S. Pat. 1,289,984 of 1918; this J., 1919, 163 A. Spray from dilute sulphuric acid is subjected to heat sufficient to evaporate it completely, and the vapour is then cooled to a temperature slightly below the maximum boiling point for sulphuric acid, to produce a mist of sulphuric acid of high concentration, which is precipitated electrically. The residual vapour is cooled to a lower temperature, at which practically the whole of the remaining sulphuric acid is condensed as a mist, which is also precipitated electrically to obtain sulphuric acid of lower concentration.

Sulphuric acid and other liquids; Apparatus for the fractional distillation of —. H. Jander, Brooklyn, N.Y. U.S. Pat. 1,321,210, 11.11.19. Appl., 8.3.19.

In a distilling apparatus, a duct for the passage of hot gases is arranged adjacent to, but out of communication with, the distilling chamber into which extend radiating devices adapted to absorb heat from the gases in the duct.—W. E. F. P.

Sulphuric acid; Process for obtaining free — from coal gas, Mond gas, etc. J. Behrens, Bremen. Ger. Pat. 300,036, 16.11.15.

THE ammonia-free gas is washed with water containing sulphurous acid. Sulphur is precipitated and thiosulphate and polythionic acids are formed. The latter are decomposed when the liquid is finally concentrated by the application of heat, yielding

sulphur dioxide, which is used again, sulphur, and sulphuric acid.—W. P.

Arsenious and selenious oxides [from pyrites]; Apparatus for the recovery of —. H. Jander, Brooklyn, N.Y. U.S. Pat. 1,321,211, 11.11.19. Appl., 3.4.19.

SEVERAL cells are arranged adjacent to a pyrites burner. Each cell is provided with cooling means and one is connected with a reheating device.

W. E. F. P.

Nitric acid of high concentration; Process for obtaining —. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 297,903, 30.5.15.

If steam and air are passed through a mixture of nitric and sulphuric acids containing nitrous gases, and the resulting nitrous vapours are dried by means of sulphuric acid, the yield of 95–96% nitric acid is almost quantitative. Any gases which pass out of the condenser are absorbed in sulphuric acid in a tower, and this acid then passes to the drying tower. Instead of pure sulphuric acid a mixture of sulphuric and nitric acids may be used as the drying agent. The amount of air and the dimensions of the condensing and drying apparatus depend on the amount of nitrous gases in the acid mixture. If there is much nitrous gas an oxidation chamber should be placed between the dryer and condenser.—A. B. S.

Hydrochloric and hydrobromic acids; Preparation of — from free chlorine or bromine. Consolidierte Alkaliwerke, Westeregeln. Ger. Pat. 313,875, 6.3.18.

CHLORINE or bromine is allowed to act upon lignite without external heating, the lignite being moved in the opposite direction to the gas stream. The temperature rises to 90°–100° C., at which the reaction proceeds. The product is extracted to remove halogenated products, and then treated to recover halogens by extraction or by distillation under reduced pressure. By the use of carbon bisulphide for extraction a by-product is obtained in the form of a halogen-containing resinous substance.—W. P.

Gases and vapours [nitric acid, hydrochloric acid, or the like]; Apparatus for condensing and purifying —. R. Rosenthal, Charlottenburg. Ger. Pat. 314,293, 21.7.18.

THE hot gases generated during the manufacture or distillation of nitric acid, hydrochloric acid, or the like, are passed upwards through a condensing tube, of which the lower portion is air-cooled and the upper portion is cooled by a stream of water. The gases are partially cooled in the lower portion and condensation is effected in the upper portion; the condensed liquid flows down the tube and meets the ascending stream of hot vapour whereby certain impurities, e.g., nitrogen peroxide in the case of nitric acid, are removed. The purified liquid passes out at the lower end of the tube, through a coil cooled by the overflow of cooling water from the upper portion of the condensing tube, and into a receiver; uncondensed gases leave the tube at the upper end and pass into an absorption apparatus.—L. A. C.

Magnesia; Process for the manufacture of — [from dolomite or the like]. J. C. Delage, Bordeaux, France. Eng. Pat. 134,626, 8.11.18. (Appl. 18,335/18.) Int. Conv., 8.11.17.

POWDERED dolomite, dolomitic limestone, or the like, is added gradually to a solution of sodium bisulphate heated to 80°–90° C. until the solution is neutral to litmus; calcium sulphate is separated from the solution by decantation, or by means of a filter press, and the precipitate is washed free from

soluble salts. Alternatively the powdered dolomite may be heated with dry sodium bisulphate at a temperature sufficient to fuse the latter, with subsequent extraction of the soluble salts by means of water. The solution of magnesium sodium sulphate obtained by either method is electrolysed by a current of $3\frac{1}{2}$ to 4 volts and 10 amps. per sq. dm. of anode surface in a vat separated into compartments by membranes impermeable by the solution but permeable by the current. The anode and cathode compartments are provided with carbon and sheet-iron electrodes respectively. The magnesite precipitated at the cathode is separated from the solution by filtration, and is washed and calcined. Free sulphuric acid is liberated at the anode, but as the precipitation of magnesite at the cathode decreases with increase of acidity, the liquor from the anode compartment overflows into a vessel in which it is neutralised by dolomite and is then returned to the apparatus.—L. A. C.

Magnesium carbonate; Process for obtaining —. A. T. Elliott, Assignor to A. L. Stewart, Los Angeles, Cal. U.S. Pat. 1,321,421, 11.11.19. Appl., 2.8.17.

MAGNESITE containing silica is leached with an acid liquor; undissolved matter and impurities are removed from the solution, and magnesium carbonate is precipitated by addition of a soluble carbonate. The precipitate is separated, and the solution is acidified and used for leaching a further quantity of magnesite.—L. A. C.

Magnesium hydroxide; Manufacture of pure —. O. F. Kaselitz, Bernburg. Ger. Pat. 313,943, 4.1.18.

MAGNESIUM chloride solution obtained in the manufacture of potassium chloride is mixed with the requisite amount of a solution of an alkaline-earth sulphide or hydrosulphide in the cold; any residue is removed, and the clear solution is boiled. If calcium sulphide is used, one-half of the magnesium is precipitated as magnesium hydroxide in accordance with the equations: $-2\text{CaS} + 2\text{MgCl}_2 + 2\text{H}_2\text{O} = \text{CaCl}_2 + \text{Ca}(\text{SH})_2 + \text{MgCl}_2 + \text{Mg}(\text{OH})_2$ or $= 2\text{CaCl}_2 + \text{Mg}(\text{SH})_2 + \text{Mg}(\text{OH})_2$, but if the calcium sulphide is converted to the hydrosulphide by passing hydrogen sulphide through the solution before addition of the magnesium chloride, or if hydrogen sulphide is passed into the cold mixed solutions, conversion of the magnesium chloride into magnesium hydroxide is practically complete.

—L. A. C.

Magnesium sulphate; Manufacture of —. E. E. and P. C. Dutt, Jubbulpore, India. Eng. Pat. 134,884, 18.10.18. (Appl. 17,076/18.)

A MIXTURE of calcium sulphide and a compound or mixture of compounds containing magnesium is subjected at a dull red heat in a reverberatory furnace to the action of air, steam, and of carbon dioxide when this is not formed during the reaction. Magnesium sulphate is obtained from the resulting mass by lixiviation.—S. S. A.

Potassium salts from blast furnace slag; Recovery of —. E. Bury, O. Ollander, T. Smith, and F. Bainbridge, Saltburn-by-the-Sea. Eng. Pat. 134,665, 3.12.18. (Appl. 20,009/18.)

CALCIUM chloride is added to a stream of molten blast-furnace slag as the latter is caused to flow down a cascade within a closed chamber. The vapour (potassium chloride) produced is drawn off and treated in scrubbers.—W. E. F. P.

Potassium sodium sulphate; Process of obtaining from saline liquors. G. B. Burnham, Borosolway, Cal. U.S. Pat. 1,321,282, 11.11.19. Appl., 16.6.19.

POTASSIUM sodium sulphate is precipitated by add-

ing sodium sulphate to liquor already containing suitable concentrations of potassium ions and sodium sulphate.—W. E. F. P.

Alkalis from silicates; Process of extracting —. R. C. Hills, Assignor to O. Newell, Denver, Colo. U.S. Pat. 1,321,632, 11.11.19. Appl., 5.12.18.

From a product containing calcium chloride and alkali chlorides obtained in the extraction of alkalis from silicates, the calcium chloride is dissolved out with alcohol.—S. S. A.

Ammonia; Removal of — from high-pressure gases. E. B. Maxted and T. A. Smith, Walsall. Eng. Pat. 134,572, 3.8.18. (Appl. 12,681/18.)

To remove ammonia from high-pressure gas mixtures, as obtained in the synthesis of ammonia from its elements, anhydrous cobaltous chloride at an elevated temperature is employed. The ammonia is absorbed when its concentration exceeds a certain partial pressure, and given up when this pressure is sufficiently diminished.—W. E. F. P.

Hydrogen-nitrogen mixtures; Process for obtaining —. Harburger Chemische Werke Schönn und Co., and W. Daitz, Harburg. Ger. Pat. 298,911, 18.5.15.

CARBON monoxide and dioxide are removed from water-gas by condensation or absorption; the carbon monoxide is employed for the quantitative combustion of oxygen in air or other mixture of oxygen and nitrogen, and the residual nitrogen is mixed with the mixture of nitrogen and hydrogen remaining from the water-gas for the preparation of nitrogen-hydrogen mixtures for the production of ammonia. The power required for the condensation or absorption under pressure is provided by the heat generated in the combustion of the oxygen.—A. B. S.

Ammonia; Manufacture of anhydrous —. A. Lowenstein, Chicago, Ill. U.S. Pat. 1,322,251, 18.11.19. Appl., 28.8.16.

A PORTION of the expanded gaseous ammonia resulting during the cyclic refrigeration process which involves the condensation, expansion, and re-absorption of ammonia obtained by heating an aqueous solution of the gas, is withdrawn and replaced by aqueous ammonia.—S. S. A.

Nitrogen fixation; Furnace for —. A. F. Crowell, jun., Saltville, Va., Assignor to Nitrogen Products Co., Providence, R.I. U.S. Pat. 1,321,892, 18.11.19. Appl., 7.3.18.

IN a vertical retort into which a charge is fed to react with free nitrogen, the walls are "offset" at the feeding point to prevent choking of the charge.—W. J. W.

Ammonia; Method of oxidising —. A. Classen, Aachen, Germany, Assignor to The Chemical Foundation, Inc. U.S. Pat. 1,322,291, 18.11.19. Appl., 24.1.16.

A MIXTURE of oxygen and ammonia is subjected to the action of catalysts formed from basic substances and substances capable of accelerating the oxidation of the ammonia, the basic substances being in excess of the active catalytic substances.—W. J. W.

Nitrogen; Process for oxidation of — in an arc with alternating current. Bergmann-Elektrizitäts-Werke A.-G., Berlin. Ger. Pat. 298,952, 7.1.16.

NITROGEN is oxidised in a chamber containing fixed electrodes, the supply of air passing through the chamber being varied synchronously with the periodicity of the alternating current used.

—A. B. S.

Cyanogen compounds; Process for the production of —. E. R. Lewis, Philadelphia, Pa. U.S. Pat. 1,321,459, 11.11.19. Appl., 15.3.19.

A CHARGE consisting of a powdered mixture of an alkali metal, carbon, iron, and an inert non-alkaline mineral is brought into contact with heated nitrogen in a heated reaction chamber. The proportions of the charge are such that there is sufficient carbon to combine with the alkali metal, sufficient iron to reduce the temperature of the reaction, and sufficient of the inert mineral to prevent the mass becoming viscid.—L. A. C.

Cyanides; Apparatus for producing — from nitrogen or air. H. B. Kipper, Muskegon, Mich. U.S. Pat. 1,322,026, 18.11.19. Appl., 19.2.18.

THE apparatus comprises a rotary reaction cylinder connected to a fuel furnace and a preheater, and regenerators each communicating at one end with the furnace and at the other end with the cylinder and the preheater.—S. S. A.

Aluminium chloride; Process for producing —. L. Burgess, New York, Assignor to Standard Oil Co. U.S. Pat. 1,321,281, 11.11.19. Appl., 3.1.19.

ANHYDROUS aluminium chloride is produced by bringing into contact, under reacting conditions, aluminium carbide and the chloride of an element below aluminium in the electrochemical scale as regards affinity for chlorine.—W. E. F. P.

Zinc oxide; Manufacture of —. Production of zinc oxide. Production of metal oxides and other metallic compounds. (A) and (D) F. G. Breyer, Palmerton, Pa., J. A. Singmaster, Bronxville, N.Y., and A. E. Hall, Palmerton, Pa.; (B) and (C) F. G. Breyer, A. E. Hall, and G. R. Waltz, Palmerton, Pa., Assignors to The New Jersey Zinc Co., New York. U.S. Pats. (A) 1,322,088, (B) 1,322,089, (C) 1,322,090, and (D) 1,322,091, 18.11.19. Appl. (A) 8.1.19., (B) and (C) 12.7.19. and (D) 1.8.19.

(A) IN the production of zinc oxide or lead-zinc oxide by the Wetherill process the bed fuel is supplied to the furnace hearth in the form of briquettes. The furnace hearth is supplied with a relatively deep layer of bed fuel in the form of briquettes, and on this ignited layer is spread a working charge of (B) zinciferous or zinciferous and plumbiferous or (C) other metalliferous material in admixture with a reducing agent. (D) IN the production of oxides or other compounds of volatilizable metals by the Wetherill process, the bed fuel is supplied to the furnace hearth in the form of briquettes.—S. S. A.

Metal oxides and other compounds of metals; Production of —. Manufacture of zinc oxide. J. A. Singmaster, New York, F. G. Breyer and E. H. Bunce, Palmerton, Pa., Assignors to The New Jersey Zinc Co., New York. U.S. Pats. (A) 1,322,143 and (B) 1,322,142, 18.11.19. Appl., 22.7.19.

(A) A BED of solid fuel is fed on to a moving perforated hearth, e.g., a chain grate, and passes under three compartments of the furnace. In the first the fuel burns with air admitted from below; in the second compartment a layer of a briquetted mixture of metal-bearing substance and reducing agent is fed upon the burning fuel, and in the third a combustion-supporting gas is supplied to the charge and the products of reaction are removed by any suitable means. (B) Zinc oxide or zinc-lead oxide is manufactured by the process described under (A).—B. M. V.

Tungstic acid; Recovery of — from ores. Imperial Trust for the Encouragement of Scientific and Industrial Research, and O. J. Stannard, London. Eng. Pat. 134,891, 6.11.18. (Appl. 18,206/18.)

FOR the recovery of tungstic acid from ores, residues, or other substances, e.g., wolframite, hübnerite, ferberite, and scheelite, the material is crushed, mixed with carbonaceous material, and heated to redness, the reduced mass being subsequently heated at about 300° C. in a current of chlorine gas, free, or almost free, from air. The volatilised or sublimed mass which results is treated with hot water, with or without the addition of nitric or hydrochloric acid. For the recovery of stannic chloride from wolfram ores the sublimate is heated with hot water and hydrochloric acid, and the resulting solution of stannic chloride is separated from the precipitated tungstic acid. The process may be made cyclic by heating the mixture of chlorides obtained in a current of dry air and using the chlorine thus recovered for the treatment of further quantities of ore.—S. S. A.

Arsenides of calcium or magnesium; Manufacture of —. J. D. Riedel A.-G., Berlin-Britz. Ger. Pat. 300,152, 18.4.16.

CALCIUM or magnesium is heated with arsenic together with some arsenide from a previous preparation as a diluent to slow down the reaction. Sand is used as the diluent in the first place. The products can be used for the preparation of other arsenic compounds.—W. P.

Alkali perchlorate; Method for the technical production of — by heating alkali chlorate. F. Meyer, Berlin. Ger. Pat. 300,713, 18.4.15.

IF the vessel containing the chlorate is heated in a metal bath so as to ensure accurate control of the temperature between 470° and 520° C. for potassium chlorate and 30° C. lower for the sodium salt, a yield of 70–75 parts of perchlorate for each 100 of chlorate is obtained. Below the temperatures mentioned the reaction is too slow; above them it is so rapid as to be dangerous. The reaction should be stopped when 80% of the chlorate has been converted, as its continuance after this point results in a lower yield.—A. B. S.

Lime mud; Process for treating — in rotary furnaces. G. Pelysius, Dessau. Ger. Pat. 313,595, 23.9.16.

WITH the lime mud is incorporated such materials as salts of the alkalis, potash mica, etc., which cause the lime to ball together and so not to pass out of the furnace in the form of dust, which would otherwise be carried forward to the chimney.

—H. J. H.

Ammonium bicarbonate; Preparation of a stable and odourless form of —. Oesterr. Verein für chemische und metallurgische Produktion, Aussig. Ger. Pat. 313,827, 22.12.17.

PRECIPITATED crystalline ammonium bicarbonate is carefully dried and then treated with moist carbon dioxide under pressure (2–3 atm.). In order to diminish its free surface the bicarbonate is briquetted before or after drying or before or after the treatment with carbon dioxide.—W. P.

Ammonium salts; Process for producing — from industrial gases containing ammonia. Chem. Fabr. Kalk Ges.m.b.H., Cöln-Kalk. Ger. Pat. 314,234, 8.5.15.

GASES containing ammonia are passed through a solution of iron. The gases must contain or have added to them sufficient carbon dioxide to ensure the complete precipitation of the iron as carbonate.

The liquid is filtered and the iron-free filtrate treated for the recovery of ammonium salts.

—A. B. S.

Hydrogen peroxide; Synthetic production of — and apparatus therefor. R. Moritz, Chatou, France. Eng. Pat. 120,045, 8.10.18. (Appl. 16,376/18.) Int. Conv., 15.5.17.

PALLADIUM or an alloy containing palladium in the form of a tube, ribbon, or wire, saturated with hydrogen is caused to act upon a solution of oxygen in water which may be under pressure. Means are provided for constantly renewing the supply of hydrogen—for instance, by moving the wire or band of palladium through an atmosphere of hydrogen in another vessel; or the hydrogen may be passed through the interior of a tube of palladium immersed in the liquid, which is maintained in the presence of oxygen under pressure. Alternatively, the process may be carried out by electrolysis in a vessel lined with ebonite, tin, rubber, or silver, and capable of resisting pressure, acidulated water saturated with oxygen being passed over the electrodes. The cathodes are composed of palladium or an alloy of palladium and the anodes of platinum, gold, graphitic carbon, or iron oxide fused in an electric furnace.—J. F. B.

Spathic iron ore; Means for treating — to render it suitable for use in the manufacture of hydrogen. W. J. and W. R. Bates, Silverdale, Staffs. Eng. Pat. 134,155, 13.3.19. (Appl. 6251/19.)

RAW ironstone is rendered highly porous by stacking it in a heap with a covering of small fuel, slowly burning the heap for a lengthy period, applying water to the hot mass when the ore is sufficiently calcined, and washing the cold material to remove fuel ash etc.—W. E. F. P.

Hydrogen generators. A. R. Griggs, London. Eng. Pats. (A) 134,901 and (u) 134,902, 9.11.18. (Appls. 18,382 and 18,384/18.)

HYDROGEN generators of the kind in which iron ore is alternately reduced and oxidised are usually made with an inner combustion chamber, a middle annular ore chamber, and an outer annular chamber surrounding the whole, the gases passing to a stack or hydrogen storage according to the operation being performed. (A) During the purging or change-over from reduction to oxidation primary steam is admitted to the base of the combustion chamber and passes through the whole apparatus, whilst secondary steam is admitted to the base of the outer chamber and sweeps that chamber and the stack outlet, so that the primary steam need only be sufficient to sweep out the combustion and ore chambers. A reduced pressure is maintained in the stack by means of steam jets, and all corners in the apparatus are rounded off as far as possible in order to facilitate removal of gases. (u) To aid the readjustment of the apparatus at the change-over, Venturi tubes with gauges are placed in the pipes for reducing gas and air supply, which indicate the pressure or volume (or both) of the gas or air passing.—B. M. V.

Carbon monoxide in hydrogen or gases containing the same; Apparatus for indicating and for recording quantities of —, applicable also for like purposes. E. K. Rideal, London, and H. S. Taylor, St. Helens. Eng. Pat. 134,243, 4.6.18. (Appl. 9205/18.)

By a process of catalytic fractional combustion in the presence of added air or oxygen, the carbon monoxide present in the gas under examination is converted into carbon dioxide, which is then brought into contact with lime water. Means are provided for recording the change in electrical con-

ductivity of the lime water and thus the amount of carbon dioxide absorbed. (See also this J., 1919, 807 A.)—W. E. F. P.

Graphites; Purification of natural —. E. Ridoni, Turin, and Soc. Talco e Grafiti Val Chisone, Pinerolo, Italy. Eng. Pat. 131,894, 8.11.18. (Appl. 18,330/18.)

PARTIAL separation of silica etc. from graphite is effected by the use of rather less than the theoretical quantity of hydrofluoric acid of lower concentration than that of the commercial concentrated acid, the advantages being that fume and waste of acid are avoided, wear of vessels is reduced, and the hydrofluoric acid may be prepared directly from sulphuric acid and a fluoride. Graphite containing 75–80 or even 80–85% C can be prepared in this way.—B. M. V.

Nitrogen; Process for effecting fixation of atmospheric — in a blast furnace. E. W. Haslup, New York. Eng. Pat. 131,885, 21.10.18. (Appl. 17,365/18.)

SEE U.S. Pat. 1,310,478 of 1919; this J., 1919, 629 A. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 4037 and 16,760 of 1893, 18,792 of 1894, and 10,305 of 1907; this J., 1894, 517, 886; 1895, 967; 1908, 1052.)

Sulphur dioxide; Treatment of gases containing —. E. V. Espenhahn, Brighton, Victoria. Eng. Pat. 131,913, 15.11.18. (Appl. 18,772/18.) Int. Conv., 15.11.17.

SEE U.S. Pat. 1,315,183 of 1919; this J., 1919, 818 A.

Barium oxide; Process of producing —. J. B. Pierce, jun., Charleston, W. Va., U.S.A. Eng. Pat. 135,285, 19.11.18. (Appl. 18,993/18.)

SEE U.S. Pat. 1,305,618 of 1919; this J., 1919, 576 A.

Pyrolusite; Method of treating —. C. V. Jørgensen, Copenhagen. Eng. Pat. 135,305, 22.11.18. (Appl. 19,266/18.)

SEE U.S. Pat. 1,303,911 of 1919; this J., 1919, 536 A.

Ammonia; Process of oxidising — and apparatus therefor. L. C. Jones, Syracuse, N.Y., and C. L. Parsons, Washington, D.C., U.S.A. U.S. Pat. 1,321,376, 11.11.19. Appl., 19.2.18.

SEE Eng. Pat. 132,551 of 1918; this J., 1919, 817 A.

Thorium; Recovery of — [as pyrophosphate]. M. A. Goltz, Winona, Minn. U.S. Pat. 1,322,185, 18.11.19. Appl., 28.2.17.

SEE Eng. Pat. 112,380 of 1917; this J., 1918, 88 A.

Tungstates; Method of obtaining pure —. J. B. Ekeley and W. B. Stoddard, Boulder, Colo. U.S. Pat. 1,322,185, 18.11.19. Appl., 21.3.19.

SEE Eng. Pat. 122,264 of 1918; this J., 1919, 131 A.

Hydrogen. Ger. Pat. 313,917. See IIa.

Gas liquor. Ger. Pat. 313,918. See IIa.

Ammonia from peat. Ger. Pat. 314,015. See IIa.

Carbonic acid. U.S. Pat. 1,319,515. See XIa.

Hypochlorite preparations. Ger. Pat. 309,106. See XIa.

VIII.—GLASS; CERAMICS.

Glass furnaces; Durability of the burners in —. H. Knoblauch, Sprechaal, 1919, 52, 407—III.

THE flames in the furnace should be as short as possible and the chimney only just tall enough to provide the requisite draught. There should be a

slight pressure in the furnace whereby gases pass outwards through any leaks in the brickwork. The distance between the bottom of the pots and the gas and air ducts should not be too great; 3 ft., though common, is excessive; 2 ft. 8 in. is ample, or the development of the greatest heat where it is not required will destroy that part of the furnace. The sides of the combustion chamber should be vertical or very slightly inclined; they should not be gathered inwards in a series of inverted steps, as is often the case, as this confines the heat too much to the combustion chamber. There should be at least two ports leading from each regenerator to the combustion chamber; too many ports are ineffective, as they cause the heat to remain in the combustion chamber. When the upper edges of the combustion chamber wear away too rapidly it is often a sign that the combustion of the gas is incomplete—possibly due to excessive draught. It may also be caused by carrying the walls of the gas and air ducts too far into the combustion chamber, and may be remedied by shortening them so that there is a distance of 16 in. between the top of these walls and that of the combustion chamber. The use of several midfeathers in the combustion chamber is undesirable; one is sometimes an advantage if placed between the two ports leading from the regenerator. To overcome the difficulties associated with a hot pocket for receiving any glass spilt in the furnace, the bottom of the combustion chamber may be inclined downwards to a door near the front of the furnace. On opening the latter any glass in the pocket may be removed without disturbing the action of the furnace. If the combustion chamber is divided by a movable midfeather and provided with two dampers—one for each half of the chamber—better control of the furnace is ensured.—A. B. S.

Lead oxide; Volatilisation of — from lead silicate [glass] melts. O. Anderson. J. Amer. Ceram. Soc., 1919, 2, 784—789.

EXPERIMENTS on the amount of PbO volatilised from the surface of lead silicate glasses at temperatures from 900° to 1400° C. showed that the rate of volatilisation from an unstirred glass heated for a short time (15 mins.) was almost identical with that from a stirred glass heated for a longer period (1 hour), but the rate of volatilisation in an unstirred glass rapidly diminished. The volatilisation of lead oxide in making optical glasses is too small to affect their refractive index if the conditions of heating are kept constant for all batches. Large losses on a commercial scale may be avoided by varying the rate of filling the pot and the use of covers.—A. B. S.

Calcined clays; Re-hydration of —. J. S. Laird and R. F. Geller. J. Amer. Ceram. Soc., 1919, 2, 828—832.

Raw clays, when heated in the presence of water in a high-pressure autoclave to 200°—250° C. for a very long time are not hydrated appreciably, but clays calcined at 600°—700° C. can be largely re-hydrated, and regain much of their former plasticity in 8—48 hours. After being worked up and dried two or three times, the re-hydrated material resembles a raw clay. Clays which have been calcined at higher temperatures are re-hydrated far more slowly and incompletely. The ease with which the re-hydration of lightly calcined clays occurs suggests that on ignition they form an anhydride which is broken down by other reactions at higher temperatures with the formation of sillimanite.—A. B. S.

Porcelain; Further studies of —. F. H. Riddle. J. Amer. Ceram. Soc., 1919, 2, 812—827.

THE effect of lime and magnesia on the micro-structure of porcelain is to reduce the temperature

at which the body matures, the finishing temperature of a body containing magnesia being lower than that of one containing lime. Solution of the quartz and the distribution and size of the sillimanite crystals vary according to the conditions of firing. The translucency of bodies containing lime and magnesia fired at cone 11 (1320° C.) is as great as that of a body containing only feldspar fired at cone 14 (1410° C.), as both lime and magnesia increase the sillimanite and the solution of the quartz, the latter being probably due to the decreased viscosity of the body. The addition of small amounts of dolomite to porcelains maturing between cones 9 and 11 (1280°—1320° C.) reduces the temperature of vitrification. It does not affect the colour. The toughness (as measured by the loss of weight of the material in a rattler test) is increased. An increase in the proportion of whiting reduces the toughness, but makes the mass more dense and vitreous. Bodies rich in dolomite are more crystalline and break with a coarse fracture; the solution of the quartz and the formation of sillimanite are well advanced. The compressive strength of porcelain containing 0—3% of whiting and dolomite averaged 51,228 lb. per sq. in., the greatest strength being obtained with whiting alone, but the difference is so small that dolomite may advantageously replace whiting, as it is cheaper and gives a greater resistance to chipping. On prolonged grinding the water of plasticity and the drying shrinkage are increased, but the working properties of the body are reduced. The effect is the same as an increase of fluxes or of ball clay. Fine grinding also causes earlier maturity and earlier overfiring.—A. B. S.

Porcelain; Relation between the composition and thermal expansivity of —. F. H. Riddle. J. Amer. Ceram. Soc., 1919, 2, 804—811.

PORCELAIN rods 2 cm. in diameter and of such a length that they were 31 cm. when fired to maturity were pressed in the stiff-plastic state in a small hydraulic press, and their coefficients of thermal expansion were determined for the temperature ranges 25°—200° C., 200°—400° C., and 25°—400° C. The first series of bodies contained 70—85% of clay, but never more than 50% of raw kaolin, the remainder being clay which had been calcined at cone 14 (1410° C.). The flint content was 0—18.5% and varied inversely with the feldspar. It was found that bodies high in clay showed low thermal expansion. The variations in the proportion of flint within the narrow limits possible in these bodies did not cause any decided reduction of expansivity. Bodies containing more than 64% of silica but less than 70% of clay showed a marked thermal expansion. In a second series in which flint was replaced by synthetic sillimanite the reduction in thermal expansion was much more marked than in bodies rich in clay, but a body with 85% of clay was not improved by the addition of sillimanite. The flint is the chief cause of thermal expansion, both on account of its inversion changes and also through the increase of the silica content of the body. The coefficient of expansion lies between the following limits:—First series: 25°—200° C., $2.9-6.1 \times 10^{-6}$; 200°—400° C., $3.0-5.3 \times 10^{-6}$; 25°—400° C., $3.1-5.5 \times 10^{-6}$. Second series: 25°—200° C., $3.1-6.2 \times 10^{-6}$; 200°—400° C., $3.5-5.1 \times 10^{-6}$; 25°—400° C., $3.3-5.5 \times 10^{-6}$. Bars of synthetic sillimanite burned to cone 26 (1580° C.): 25°—200° C., 2.7×10^{-6} ; 200°—400° C., 3.9×10^{-6} ; 25°—400° C., 3.3×10^{-6} .—A. B. S.

Silica bricks; Apparent versus true specific gravity of —. L. R. Office. J. Amer. Ceram. Soc., 1919, 2, 833—835.

A DRY chip of a brick, weighing about 10 grms., was boiled in water for 1 hour, and its sp. gr. determined by means of a Westphal balance. The

chip was afterwards ground to powder, dried at 100°C ., and the sp. gr. determined in a pyknometer. Of 27 samples tested in this manner, 22 showed a slightly higher, 2 the same, and 3 a lower true sp. gr., the mean true sp. gr. being 0.011 higher than the apparent sp. gr.—A. B. S.

Enamelling; Cause and control of blistering in sheet steel —. C. Treischel. J. Amer. Ceram. Soc., 1919, 2, 774—781.

A form of blistering in which the enamel splits off the steel sheet in an aggregation of small blisters or in parallel rows or striations is due to absorption of hydrogen liberated when the sheet is pickled in a 10% solution of hydrochloric acid. If the pickling process is replaced by treatment with a sand blast or if only a 3% solution of acid is used no blistering occurs. In the latter case the time required for pickling is not noticeably increased, but each piece should be brushed before washing so as to assist in the removal of the scale.—A. B. S.

Kaolinite. Wegscheider. See VII.

Lead oxides. Andersen. See VII.

PATENTS.

Sheet-glass; Apparatus for the production of —. T. J. McCoy, Shinglehouse, Pa., U.S.A. Eng. Pat. 134,807, 11.10.18. (Appl. 21,788/19.)

AN apparatus for making sheet glass continuously consists of a tank for the molten glass, with an upwardly inclined bottom ending in an overflow-spout; an inclined cover for the spout, a movable front wall adjacent to the front end of the cover, and means for varying the inclination of the cover so as to create an air-space between it and the front wall and thus regulate the temperature of the glass. Guiding ribs extend over the top and front of the spout and hot air is passed through the space between the lower portion of the side walls and the spout so as to maintain the edges of the glass at the same temperature as the remainder. After the glass has flowed over the spout in a stream of regulated thickness it passes vertically downward and then along an annealing lehr.—A. B. S.

Sheet glass; Manufacture of —. J. Proeger, Pittsburgh, Pa. U.S. Pat. 1,320,091, 28.10.19. Appl., 9.8.18.

IN order to equalise the heat in glass sheets during the drawing operation the sheet is drawn through a substantially enclosed receptacle provided with a passage-way through it and with openings in the top and bottom. Gases enter the receptacle through the latter opening, and means are connected to the receptacle to draw the hot gases from the sides, thereby allowing the gases to move across the sheet.—B. N.

Glass ovens or furnaces. E. C. R. Marks, London. From Soc. Anon. Fours et Procédés Mathy, Liege, Belgium. Eng. Pat. 135,115, 14.1.19. (Appl. 9496/19.)

A GLASS pot-furnace comprises several chambers each containing a pot surrounded by fragments of porous refractory material, the bottom of each chamber being formed with cavities filled with such fragments and which receive air and gas at their base. A regenerator may be provided in the base of the furnace for heating the gas and air used therein. Heating is effected by flameless surface combustion.—A. B. S.

Hard porcelain for fuses; Process for production of —. Porzellanfabrik A. Schweig Ges.m.b.H., Weisswasser. Ger. Pat. 298,480, 22.12.16.

THE body mixture is mixed with oil and pressed into the desired shape. The oil ensures the ready removal of the mass from the dies, acts as a binder, and enables a dense and homogeneous porcelain to be produced.—A. B. S.

Glass. Corning Glass Works, Assignees of H. P. Gage and W. C. Taylor, Corning, N.Y., U.S.A. Eng. Pat. 127,586, 28.5.19. (Appl. 13,508/19.) Int. Conv., 28.5.18.

SEE U.S. Pat. 1,305,793 of 1919; this J., 1919, 579 A.

Separation of absorbed gases from water. Ger. Pat. 313,612. See I.

[Refractory] cement. U.S. Pat. 1,321,085. See IX.

IX.—BUILDING MATERIALS.

Lime-mortar. Dissolution and peptisation of solids. V. Kohlshütter and G. Walther. Z. Elektrochem., 1919, 25, 159—183.

AN experimental study of the hydration of lime in all its stages was undertaken in the hope of obtaining practical data in support of the authors' theory of the colloid state and the relation between colloidal and true solutions. The account of the experiments is preceded by a long theoretical discussion on the nature of solution, the hydration of lime, and the properties of mortar. The slaking of lime is a good example of the step-by-step character of the solution process, the stages being the formation of:—(a) calcium hydroxide; (n) hydrated forms of the hydroxide; (c) lime cream, a non-settling suspension or paste; (p) milk of lime, a colloidal suspension of lime in the saturated solution; (e) lime water, a true solution. It is emphasised that the structure of the material from which the lime is made must have a profound influence on the properties of the lime, on its slaking properties, on the character of the lime cream obtained, and on the hardening properties of the mortar, exactly as is found in practice. For the experiments lime was used prepared from calcium carbonate, hydroxide, and oxalate. When slaked by exposure in a damp atmosphere the three samples differed considerably in the rates at which they absorbed water. Up to the formation of the hydroxide the rates were nearly the same, but afterwards the lime from carbonate absorbed most and that from oxalate least rapidly. The former also absorbed the greatest total quantity, about 1.5 mols, and the latter the least, about 1.25 mols. When the products were dehydrated and again slaked in the same way, the influence of the original source was still manifest, showing that the specific structure of the particles had not been destroyed. The samples also showed marked differences in their rates of sedimentation when slaked with much water, and the rates and order of settling were found to depend on the method of slaking, i.e., whether the lime was added to water or the water poured over the lime. Different electrolytes present in the slaking water had different effects on the rates of sedimentation, some retarding and some accelerating the rate of settling. No definite conclusions could be drawn from the results of the experiments beyond the general inference that in the first stage of the action of water on lime complex chemical and capillary forces come into action with the colloidal particles of the solid. —E. H. R.

Cements rich in alumina. K. Endell. Zement, 1919, 8, 391—321, 331—336, 347—350. Chem. Zentr., 1919, 90, IV., 658.

THE ternary system, $\text{CaO}-\text{SiO}_2-\text{Al}_2\text{O}_3$, has been examined to determine the limits within which compounds capable of hydraulic setting are formed. It is shown that good cements are obtained from clinkers in which the following crystalline phases, which can be identified mineralogically, are predominant:— $\text{CaO}, \text{Al}_2\text{O}_3$; the group of calcium orthosilicates, $2\text{CaO}, \text{SiO}_2$; $3\text{CaO}, 5\text{Al}_2\text{O}_3$. The following do not form cements:— $3\text{CaO}, \text{Al}_2\text{O}_3$,

and gehlenite $2\text{CaO}, \text{Al}_2\text{O}_3, \text{SiO}_2$. The strength of the products under compression is dependent on the alumina content.—H. J. H.

Thermal conductivity of solid insulators. Thornten. See XI.

PATENTS.

Slabs [from leather waste]; Production of hard flooring or insulating —. H. Gronrose, Copenhagen. Eng. Pat. 124,415, 19.12.18. (Appl. 21,264/18.) Int. Conv., 19.3.18.

HARD flooring or insulating slabs are made by mixing 100 kilos. of pulverised leather waste with 3–5 kilos. of boiled linseed oil, drying the mass, mixing it with about 200 kilos. of spirit varnish, 1 kilo. of infusorial earth, and $\frac{1}{2}$ kilo. of red lead, and pressing into slabs under a pressure of 800 kilos. per sq. cm. Alternatively, the varnish and infusorial earth may be mixed with the leather, shaped under pressure, and the product coated with boiled linseed oil.—A. B. S.

Kilns; Apparatus for automatically discharging lime, cement, and like —. C. Candlet, Paris. Eng. Pat. 131,269, 12.11.18. (Appl. 18,542/18.) Int. Conv., 10.8.18. Addition to 119,235 (this J., 1919, 750 A).

THE lower part of a shaft kiln is provided with four cones, having horizontal axes and supported on separate shafts so as to leave a clear space adjacent to their apices, each cone being formed of two conical portions having different angles, with the apex directed toward the centre of the kiln. The smaller obtuse-angled portion near the apex may be provided with ridges or ribs to allow large pieces of material to pass between the cones without being subjected to heavy torsional stresses. The omission of central supports permits the admission of air to the centre of the kiln. Above the cones, and immediately below the grate through which the air enters, is a truncated cone or collecting ring, with a vertical axis, which directs the small pieces from the periphery to the centre of the kiln and relieves the pressure on the bases of the cones. The rotation of the collecting ring causes the cones to rotate by friction. In a modification, the cones are rotated mechanically so as to crush the material discharged from the kiln.—A. B. S.

Concrete. Harland and Wolff, Ltd., and A. Marks, Belfast. Eng. Pat. 134,598, 1.11.18. (Appl. 17,901/18.)

A LIGHT-WEIGHT concrete is made by mixing 2 parts by weight of Portland cement and 1 part by weight of sawdust, and damping the mixture by the addition of a fluid containing 1 vol. of sodium silicate to 3 vols. of water. Sufficient 10–20% solution of calcium chloride is then added to form a wet mortar containing a slight excess of sodium silicate. The surface of the concrete may be made waterproof by coating it with a similar mixture containing a smaller proportion of fine sawdust. If the surface is to be exposed to the weather it is also hardened by treating it with dilute hydrofluoric acid. Alternatively, hydrochloric acid may be used instead of calcium chloride solution if the cement contains sufficient free lime, or if lime is added as a filler.—A. B. S.

Concrete; Pumice aggregate for —. A. Ham-bloch, Andernach. Ger. Pat. 310,120, 14.8.18.

PUMICE powder is freed from coarse particles by levigation and then dried and used as an aggregate with or without previously coating it with cement slurry, milk of lime, etc., in order to make it impervious externally. Such pumice only weighs 200–250 kilos. per cb.m.—A. B. S.

[Refractory] cement. H. B. Boeth, Assignor to A. Ramsay and Son Co., Montreal, Canada. U.S. Pat. 1,321,085, 11.11.19. Appl., 18.11.18.

A PROTECTIVE coating for bodies subjected to high temperatures consists of a mixture of felspar, angular grains of crushed quartz, silica sand, and a binder.—A. B. S.

Roofing, flooring, or paving materials; Manufacture of —. W. Bunte, Hannover-Linden, and W. North, Hannover. Ger. Pat. 313,565, 8.11.18.

NATURAL or artificial hollow particles are mixed with ground pitch and compressed. The product obtained by mixing pitch with ground calcined kieselsguhr has a rich copper colour. It is resistant to wet, frost, and sunshine, and particularly towards changes of temperature. It can be used either as flooring or roofing material.—H. J. H.

Asbestos cement sheets; Process for glazing —. A. Silber, Offenburg. Ger. Pats. (A) 313,594, 9.4.16, and (B) 313,939, 9.9.16.

(A) THE sheets are fired, allowed to cool to 70° – 100° C., and dipped for a short time into a soap solution at the same temperature. The soap reacts with the quicklime, forming an insoluble lime soap, which fills up the pores left by the dehydration under heat so that the material regains its original strength. (B) The articles, after coating with the glazing material, are heated under load or tension, to the temperature of incipient melting of the glaze, after which the load is removed and the melting completed. A uniform and unbroken glaze surface is produced and distortion of the body is avoided. After the burning the article is treated with a soap solution as in (A).—H. J. H.

Fireproofing. U.S. Pat. 1,321,708. See V.

Preserving wood. Ger. Pat. 299,761. See V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Open-hearth furnaces; Improvements in the technique of furnace firing, with special reference to —. W. Tafel. Stahl u. Eisen, 1919, 39, 1280–1283.

FROM the nature of the firing of most metallurgical furnaces the greater part of the heat supplied is lost in the waste gases. In order to utilise this waste heat, three courses may be adopted—preheating the material treated, preheating the gas and air for combustion, and the application of special heating installations, as, for example, steam boilers. For reclaiming the waste heat from hearth-melting furnaces the first method is not practicable and the second only partially. The installation of steam boilers in connection with open-hearth furnaces introduces some difficulties, especially the danger of explosions. This danger may be partially overcome, but not completely, by reversing the gas and air at different times. In order to make explosions impossible it is recommended that the waste gases coming from the gas and air regenerators be drawn through separate channels after passing the reversing valves.

—T. H. Bu.

Steel; Viscosity of — at high temperatures. P. Chevenard. Comptes rend., 1919, 169, 712–715.

THE increase in length of a wire maintained at constant temperature and subjected to a weight applied without shock was recorded on a photographic plate having a uniform transverse motion, so that a curve representing extension : time was plotted. Oxidation was prevented by heating the wire in an atmosphere of nitrogen, and for each metal a series of heatings was made varying the initial condition of the wire, the temperature, and the load. Using a self-hardening nickel-chromium

steel, the curve approximates to a straight line with a constant rate of extension (V) for any particular temperature, though this factor is altered if the diameter of the wire is reduced appreciably. Regarding the rate of extension (V) as a coefficient indicating the degree of viscosity, and plotting it with varying load against temperature, the curve mounts very steeply at a temperature of 500° – 600° C., indicating the elimination of internal stresses as soon as a red heat is attained. The increase in extension, rdt , in the time dt is equal to the decrease in elasticity $\left(\frac{dp}{E}\right)$, resulting from the reduction in section (dp), so that $rdt - dp/E = 0$. The solution of this equation is the time $\left(t \frac{p_1}{p_0}\right)$ of heating capable of bringing the original tension, p_0 , to a given value, p_1 , and, accepting certain assumptions, a value is obtained,

$$t \frac{p_1}{p_0} = \frac{1}{E} \int_{p_0}^{p_1} \frac{dp}{V}$$

It is deduced that for all steels with the exception of high-speed steels, the usual practice of annealing for several hours at 600° – 700° C., followed by slow cooling, is sufficient for the removal of internal stresses caused by heat treatment or mechanical working.—C. A. K.

Molybdenum in steel and iron; Qualitative and quantitative test for —. S. L. Malowan. *Z. anorg. Chem.*, 1919, 108, 73–80.

A SPECIFIC and very sensitive test for molybdenum is given by xanthic acid. The test is best carried out with freshly prepared xanthate solution in the following manner: A solution of potassium hydroxide in absolute alcohol is shaken with excess of carbon bisulphide until no more of the latter is dissolved. To the solution so obtained 30% acetic acid is added until a slight yellow turbidity is formed, and the reagent is added drop by drop to the solution to be tested. If the latter has been prepared by dissolving the test material in strong acid, most of this should be neutralised before adding the reagent. If molybdenum is present an intense red colour immediately develops, which is quite stable in water and the intensity of which is proportional to the amount of molybdenum present. The sensitiveness of the reaction is such that 0.000005 grm. Mo in 0.0007% solution can be detected with certainty in presence of other elements. The colour is readily soluble in alcohol, amyl alcohol, ether, and chloroform, less readily in petroleum spirit and benzene. It is readily extracted from its reddish-violet ethereal solution by alkalis, and is precipitated from the alkaline solution by acids. The ethereal solution decomposes on standing with formation of molybdenum sulphide. The reaction is not given by vanadium, tungsten, titanium, or uranium; chromates give a dark coloration with xanthic acid, and should be reduced, if present, before the test is made. The reaction can be used for the colorimetric estimation of molybdenum, by comparison with a standard solution, but since ethereal solutions decompose too quickly, the ether should be mixed with 35% of petroleum spirit for the extraction and with 70% of petroleum spirit for the necessary dilution.

E. H. R.

Heuser's [magnetic] alloys. O. von Auwers. *Z. anorg. Chem.*, 1919, 108, 49–69.

A THEORETICAL discussion of the ferromagnetic alloys of the aluminium-manganese bronze series. Their properties are explained on the assumption that the magnetic carriers are of the form $(AlM_3)_x$, where M_3 is an isomorphous mixture of manganese and copper. These complexes dissociate at the magnetic transition temperature, about 600° C. When the alloy is quenched from this temperature

it has very weak magnetic properties, because there is no opportunity for the complexes to re-form. By slow cooling or by the so-called ageing process, i.e., prolonged heating at temperatures between 100° and 300° C., the molecules become associated to form the magnetic particles, and the magnetic properties of the alloy are developed. The theory explains the rise of the magnetic transition temperature as the time of ageing is lengthened, and also the rise in the value of the coercive force as the ageing temperature is raised to about 260° C. and the subsequent fall in its value when higher temperatures are used. (See also *J. Chem. Soc., Jan., 1920.*)—E. H. R.

Manganese-bismuth, manganese-zinc, and manganese-silver alloys. P. Siebe. *Z. anorg. Chem.*, 1919, 108, 161–183.

MANGANESE and bismuth are not completely miscible, but show a gap between 23% and 99.5% Mn. By the cooling curve method five arrest points were found, at 259° , 442° , 597° , 1043° , and 1252° C. The first and last represent the separation of eutectics containing respectively about 0.5% Mn and 0.5% Bi, whilst the others indicate three compounds, the compositions of which have not been determined. The five phases can all be distinguished microscopically. The first compound separates from alloys containing from 0.5 to 9% Mn, the second from those with 9 to 11% , and the third from those with 11 to 20% Mn. Mixtures containing more than 23% Mn separate into two layers. The hardness increases with the manganese content. The alloys are ferromagnetic, this property being peculiar to the first of the above compounds. Zinc-manganese alloys were prepared containing up to 50% Mn. A zinc-mixed crystal eutectic was found at 416° C., containing a very small proportion of manganese. The mixed crystals may contain from 11% to 50% Mn. Alloys containing only 2% Mn are hard and brittle; the hardness increases with the manganese content and with 40% Mn the hardness is 5 on Mohs' scale. Silver-manganese alloys show a sharp change in chemical and electrical properties at about 14.5% Mn (0.25 mol.). Those containing less than this proportion of manganese are unattacked by solutions of silver and copper salts or by dilute acids, whilst those with more than the limiting amount of manganese are rapidly attacked. (See also *J. Chem. Soc., Jan., 1920.*)—E. H. R.

Flotation; A contribution to the study of —. H. L. Sulman. *Inst. Min. and Met.*, Nov. 20, 1919. [Advance copy.] 95 pages.

THE author explains the complex of facts associated with flotation on the broad lines of the molecular hypothesis. Flotation reactions arise from the unbalanced molecular attractions in the surface layers of solids and liquids which are the seats of excess energy. Selective wetting effects always occur in the direction of the greatest possible reduction of interfacial energy. High interfacial energy implies low liquid/solid adhesion and floatability, and *vice versa*. Degree of wetting is connected quantitatively with the contact angle. For pronounced floatability this should approach or, preferably, exceed 90° . For complete wetting the interfacial tension must be nil, a condition which coincides with complete deflocculation. The hysteresis of contact angles permits a wide range of equilibrium for a floating particle. The addition of reagents to pure water and crushed ore and specific procedures are necessary to intensify preferential wetting effects until they become of practical use in the separation of sulphide minerals from gangue. An immiscible oil is employed to diminish the surface energy of the sulphide, and the addition of acid or alkali to the water promotes adhesion between gangue and water. Solid surfaces are probably penetrated by the molecules of liquids, and their

adhesion thus enhanced. Solid surfaces undergo molecular modification by friction, and molecules of any liquid in contact with them are included in greater number in the modified surface. This is of importance in the wet crushing of ores. Freshly-broken solid surfaces often undergo a spontaneous molecular alteration with a diminution of surface energy. Concentration of foreign molecules at the surface of a pure or homogeneous liquid reduces the surface tension and confers the property of frothing. Frothing reagents leave a partial strain at the bubble surface; mineral adsorption stabilises the film, especially if the mineral be oiled or flocculated. The bubble system should be disseminated through the ore pulp mass. By the use of acid, alkalis, sodium silicate, and certain sols deflocculation results by removal of strain from the surfaces of suspended particles. Particles tend to agglomerate by the coalescence of their strained layers. Flocculation is increased by mechanical agitation, minute oiling of the particles, and by contact with air (bubbles). In standard mineralised froths the layers may be scores of particles in thickness. If a substance can be flocculated it can generally be floated. Electrical phenomena are of minor importance. Flotation depends on bringing about the most advantageous selective adhesions, selective adsorptions, and selective flocculations between the ore pulp particles. (See also Willows; this J., 1919, 450 r.)

—T. H. Bu.

α -Naphthol and xylydine mixture as a flotation agent. Chem. and Met. Eng., 1919, 21, 418.

ACCORDING to the Annual Report of the Consolidated Coppermines Co., by the introduction, in place of a mixture of coal tar, coal-tar creosote, and pine oil, of a mixture of α -naphthol and xylydine as flotation agent in an alkaline circuit, the concentrating ratio has been increased from 14'68:1 to 17'0:1, and the moisture content of the filter-cake reduced from 17'5 to 9'39%. The increased cost of reagents is more than compensated by a decrease of more than half in the freight and smelter charges, and by a threefold increase in the capacity of the filter-presses. From a mill-head material containing 1'4% Cu a concentrate containing 18% Cu can be produced, and the iron, sulphur, and insoluble content may be governed within such limits as to permit of smelting the concentrate with less than 20% of barren flux.

—S. S. A.

Colloidal mercury. Gutbier and Weise. See XI.

Aluminium vessels in dairies. Utz. XIXa.

Colloidal copper. Gutbier and Sauer. See XX.

PATENTS.

Iron and steel; Process for the manufacture of —. D. R. Blair and H. B. Clapp, London. Eng. Pat. 134,283, 26.10.18. (Appl. 17,509/18.)

FINELY-DIVIDED iron ore is heated to about 1350° C. with a carboniferous reducing agent, together with sodium chloride and other materials, so as to produce a fusible slag which will readily flow away from the reduced metallic iron, leaving the latter unmelted and in a spongy condition. It is claimed that ores high in phosphorus, sulphur, or titanium will yield a more pure iron by this method owing to the reduction taking place without fusion of the metal. The spongy mass may be squeezed and forged into wrought iron, or converted into steel.

—C. A. K.

Steel and steel articles; Surface hardening of —. Vickers, Ltd., London, and W. J. D. Sherwood, Sheffield. Eng. Pat. 134,599, 1.11.18. (Appl. 17,903/18.)

A MODIFICATION for special purposes of Eng. Pat.

5588 of 1910. Air-hardening steel (*e.g.*, for tools) at a temperature (650°—800° C.) below the hardening temperature is subjected to rapid heating to above the hardening temperature and subsequently cooled. The time of heating is not sufficient to allow the interior of the metal to become heated by conduction above the temperature at which hardening takes place. Heating may be effected by passing an oxyacetylene flame over the surface at a speed of from 4 in. to 10 in. per min. according to the particular steel treated.—C. A. K.

High-speed steel. J. R. Valentine, Fulton, N.Y. U.S. Pat. 1,321,163, 11.11.19. Appl., 27.2.18.

A STEEL containing 13—14% W, about 5% Cr, about 1% V, less than 1% C, and 1%—1'25% Mn.

—J. W. D.

Steel; High-speed — containing chrome, tungsten, and molybdenum with addition of cobalt. Oberschlesische Eisen-Industrie A.-G., Gleiwitz. Ger. Pat. 300,765, 19.2.16.

IN a steel with almost 7% of tungsten, cobalt and molybdenum amounting to 5—10% together are used in place of any further tungsten required. In such a steel a molybdenum content of 2—6% and cobalt content of 5—2% may be used provided that the sum of the two does not exceed 10%. A satisfactory product contains C 0'65%, W 5'5%, Mo 3'2%, Co 3'5%, Cr 4'8%, V 0'4%. If the molybdenum content exceeds 6% the steel becomes more difficult to work, sensitive to heat, and brittle.

—H. J. H.

Steel; Manufacture of —. A. H. Henderson, Baltimore, Md. U.S. Pat. 1,322,496, 18.11.19. Appl., 18.2.19.

IN the production of steel for ingots or castings, chloride of lime (15 lb.), anhydrous aluminium oxide (2 lb.), and anhydrous borax (2 lb. per ton of metal) are added during the fusion of the charge; and lead (6 lb.) and zinc (1—2 lb. per ton of metal) are added to the molten metal in the ladle.

—W. E. F. P.

Steel; Process for making basic — by a combined open-hearth and converter process. O. Thiel, Landstuhl. Ger. Pat. 290,632, 24.2.15.

A PART of the Thomas charge is refined in the open-hearth furnace, and is then added to the converter charge and the whole blown together, or after blowing the converter charge, the refined metal from the open-hearth furnace is added, and the refining completed by further blowing. The refined metal, which is poured into the mixer for raising the temperature of the pig iron, must not be made from Thomas pig iron. It may be made by melting scrap and other pig iron together. The mixer iron should contain enough phosphorus for carrying out the Thomas process. Besides other advantages the time of blowing in the converter is reduced.—T. H. Bu.

Cast iron; Production of refined — from ordinary pig iron. Vereinigte Hüttenwerke Burbach-Eich-Düdelingen, Werk Dommeldingen, Luxemburg. Ger. Pat. 306,482, 27.6.16.

THE iron is refined in a reverberatory furnace under such conditions that, as far as possible, removal of the carbon avoided. High-phosphorus iron, as required for chill cast rolls, with up to 0'4% of phosphorus, is made by arresting the process at the correct moment. A highly-basic slag is used. Silicon is removed first, and if the quantity is large the silicious slag is poured off, whereupon dephosphorisation proceeds more rapidly. Sulphur and manganese are removed with the phosphorus. When dephosphorisation has proceeded sufficiently far, the slag is poured off, lime added, and about half the charge is poured and alloyed with ferro-

silicon and ferromanganese. To the other half in the furnace a fresh charge of pig iron is added.—H. J. H.

Furnace, especially for manufacture of iron and steel. W. Corsalli, Berlin, Ger. Pat. 313,620, 19.9.13.

THE furnace is equipped with regenerators for gas, air, or both, and a chamber traversed by the waste gases in which slag can be kept molten. The hot waste gases pass downwards from the hearth and through the slag chamber and then to the regenerators. Additional burners are provided in the slag chamber whereby its temperature can be further raised if necessary. By arranging that the flame passes in one direction only, it is possible so to construct the furnace that the gas stream passes between an inner and an outer stream of air or vice versa. Thus the flame has two zones of combustion—an inner and an outer—which facilitates control of its position and oxidising or reducing character by regulation of the pressure of air and gas. To avoid excessive heating of the furnace wall opposite the burner, as a result of the unidirectional character of the flame, this wall is set back so as to leave room for a chamber where preheating of charges and crucibles can be effected. Additions can be made to the slag while in its melting chamber so as to increase its value as a fertiliser or as a paving material. The slag may also be removed from the furnace and granulated preparatory to the recovery of titanium.—H. J. H.

Heating steel ingots; Furnace and process for —. K. Koller, Buda-Pesth, Ger. Pat. 313,757, 10.1.18.

THE furnace is designed to enable the ingots while remaining stationary to be heated to various temperatures and at various speeds simultaneously. Thus the ingots for the production of Mannesmann tubes can be rapidly heated to redness, then maintained at a temperature to relieve internal strains, and subsequently heated to the temperature of rolling. The heating gas is introduced by jets at the top of the furnace, and the air through ports running along the side at the floor level. The furnace is divided into sections each capable of independent regulation of temperature, either by division walls and independently regulated gas and air ports or by long, narrow air ports in the floor capable of independent control by valves.—H. J. H.

Iron or steel; Treatment of — for prevention of oxidation or rusting. L. S. Chadwick, East Cleveland, and M. Resek, Cleveland Height, Assignors to Cleveland Metal Products Co., Cleveland, Ohio. U.S. Pat. 1,320,734, 4.11.19. Appl., 6.2.17.

THE metal is treated with a solution containing an alkali phosphate, a salt of a metal less basic than iron, and an acid.—A. E. D.

Rustless surface on iron and steel; Production of a — by boiling phosphoric acid. W. Schmidting, Cöln-Mannsfeld, Ger. Pat. 313,578, 1.5.18.

AN anti-rust solution containing phosphoric acid, calcium, and zinc is mixed with metallic oxides and water. When applied to the iron articles a layer of intimately mixed zinc-iron phosphates is produced on the surface. The calcium phosphate, in an extremely fine state of division, is distributed through the coating, making it more resistant and adhesive.—H. J. H.

Electric furnace for heating, annealing, or melting metallic materials. Stabilimenti "Biak" Ing. A. Pouchain, Turin, Italy. Eng. Pat. 124,438, 10.3.19. (Appl. 5931/19.) Int. Conv., 9.3.18.

A FURNACE is built up of a number of elements of annular form, made of magnetic material. Longi-

tudinal insulated conducting bars of high ohmic resistance pass through holes in the elements parallel to the axis of the furnace, and are connected so as to form one or more complete turns. When an alternating current is passed through the conductor, the heating effect is due partly to resistance and partly to induction effects. The separate elements may be spaced by insulating air gaps or by layers of refractory material in order to modify the power factor of the furnace.—C. A. K.

Electric resistor; Zig-zag carbon —. Electric furnace for reducing oxidised zinc concentrates by carbon. J. Thomson, New York. U.S. Pats. (A) 1,321,682 and (B) 1,321,683, 11.11.19. Appl., (A) 7.10.18, (B) 27.1.19.

(A) Two contiguous zig-zag carbon resistors, connected with each other at one end and separated by a narrow slot. (B) A furnace for the reduction of zinc concentrates is heated by a carbon resistor of the above type, the surface of which is relatively non-conducting and immune from oxidation. The resistor is surrounded by the charge to be reduced, and is non-reactive with the reducing agent (carbon) in the centre of the furnace.—C. A. K.

Blast furnaces; Process for reduction and smelting in —. A. Torkar, Vienna. Ger. Pat. 313,619, 3.1.18. Int. Conv., 13.12.17.

REDUCING gases are introduced at the top, and in their passage downwards through the charge reduce the ore to the metallic state and bring the whole to fusion by combustion with preheated air supplied at the tuyères. The products of combustion pass through openings in the wall of the crucible and away into flues at the ground level.—H. J. H.

Converter. A. Nielsen and A. Birkedal. Ger. Pat. 313,756, 11.10.17.

A CONVERTER for melting metal by means of gas or sprayed fluid fuel, and subsequent purification by blowing air through the molten metal, is provided at one side with a container for the pig iron, which melts down into the working space of the vessel, the throat being closed with a cover. The products of combustion are led out through a flue leading from the working space of the converter to the container for the pig iron. The air pipe is provided with a reversing switch, so that the air can be passed to the burners or to the tuyères as desired.—T. H. Bu.

Siemens-Martin furnace, with built-in gas producer. P. Ostendorf, Vienna. Ger. Pat. 313,781, 20.4.18.

THE gas producer communicates directly with the hearth by slit-shaped ports, through which the gas enters. The sensible heat of the producer gas is therefore made effective in attaining a high temperature in the hearth. The air heated in regenerators is introduced above the gas ports and under such pressure as to drive the flame down upon the bath.—H. J. H.

Aluminium or aluminium alloys; Method of tin-soldering and tin-coating —. A. G. Bloxam, London. From Stabilimenti "Biak" Ing. A. Pouchain, Turin, Italy. Eng. Pat. 131,315, 30.10.18. (Appl. 17,739/18.)

A COATING of iron is deposited electrolytically on aluminium or an alloy of aluminium, the electrolyte consisting of a mixture of equal parts of a 4% solution of ammonium oxalate and a 2% solution of ferric sulphate. A current density of about 0.15 ampère per sq. dm. of cathode is used. The surface may then be tin-soldered or coated, using an acid solution of zinc chloride as cleaning agent.

—C. A. K.

Solder for aluminium. J. G. Kelly, Assignor to H. Hall, Sedalia, Mo. U.S. Pat. 1,321,529, 11.12.19. Appl., 10.2.19.

Zinc, lead, block tin, and a flux-like ingredient containing stearic acid are mixed in equal proportions and heated together with a small amount of skunk oil.—J. W. D.

Metal alloy. J. A. Weiger, Union Hill, N.J., Assignor to The Independent Lamp and Wire Co., Weehawken, N.J. U.S. Pat. 1,321,170, 11.11.19. Appl., 25.8.19.

An alloy containing 77—80% Ag, 18—20% Cu, and 2—3% Pt.—J. W. D.

Alloy. F. M. Becket, Assignor to Electro-Metallurgical Co., Niagara Falls, N.Y. U.S. Pat. 1,322,158, 18.11.19. Appl., 7.6.18.

An alloy of magnesium, silicon, and manganese, containing magnesium in excess of the equimolecular proportion to the silicon.—W. E. F. P.

Lead-tungsten alloys; Manufacture of —. H. Falkenberg, Weetzen. Ger. Pat. 299,052, 23.5.14.

A LITTLE finely-divided iron is added to a mixture of lead and tungsten before heating the same to form an alloy. By this means homogeneous alloys may be prepared containing lead and tungsten in any proportion.—L. A. C.

Tin or antimony; Alloy suitable as substitute for —. W. Zimmer, Giessen. Ger. Pat. 306,382, 21.10.17.

An alloy of arsenic and cadmium in approximately equal proportions may be used as a substitute for tin or antimony in the manufacture of alloys of the same with lead or copper, such as hard lead, bearing metal, type metal, etc.—L. A. C.

Alloy. G. Fuchs and A. Kopietz, Berlin. Ger. Pat. 310,041, 24.1.18. Addition to Ger. Pat. 307,764 (this J., 1919, 910 A).

THE tungsten content of the alloy specified in the chief patent may be increased to about 60%. The alloy may consist, for example, of W 57%, Fe 33%, Ti 5%, C 3%, and Ce 2%.—T. H. Bu.

Defects in metallic articles; Method of developing —. F. G. Brettell, Birmingham. From The Sneed and Co. Iron Works, Jersey City, N.J., U.S.A. Eng. Pat. 134,791, 5.7.19. (Appl. 16,850/19.)

METALLIC articles, such as tubes, are placed between electrical contacts and a current of low voltage and high amperage is passed so as to heat the tubes to the decalescence point. Seams, segregations, or other defects, which produce increased resistance to the passage of the current are indicated by overheating of the particular portion of the tube. This method of heating may be used at the same time for heat treatment of the article, which may be examined visually, after cooling, for overheating effects.—C. A. K.

Compressing powdered tungsten, tantolum, and the like; Means and method of —. C. A. Pfanstiehl, Highland Park, Ill., Assignor to Pfanstiehl Co., Chicago, Ill. U.S. Pat. 1,321,125, 11.11.19. Appl., 24.10.16.

POWDERED refractory metals are pressed into irregularly formed articles by placing the powder between a metallic cup and a die and subjecting the interior of the cup to fluid pressure, whereby the cup is expanded to compress the powder between it and the die.—J. W. D.

Metals of high melting point [tungsten, molybdenum, etc.]; Manufacture of articles from —. L. Reimann, Berlin. Ger. Pat. 302,650, 8.8.14.

IN the manufacture of articles from tungsten,

molybdenum, or the like, by compression and subsequent heating, a portion of the article is formed from a mixture of the powdered metal with a small percentage of another metal of melting point higher than that of copper, e.g., nickel, cobalt, or platinum; subsequent heating causes this portion of the article to contract and bind itself firmly to another part of the article. An anticathode, for example, may have a face of pure tungsten with a backing of tungsten containing a small quantity of platinum. A rod fitting tightly in a hole bored in the backing becomes permanently fixed when the backing contracts on heating. A suitable powder may be prepared by heating tungsten oxide moistened with a solution of a suitable salt at 1000°—1200° C. in a current of hydrogen.—L. A. C.

Pickling bath. W. H. Allen, Detroit, Mich. U.S. Pat. 1,321,182, 11.11.19. Appl., 10.5.17.

SURFACES of iron and steel having patches of oxides of iron are cleaned by immersing in a bath wherein the oxides will be reduced by sulphurous acid and the reduced metal dissolved by sulphuric acid. The articles are then removed and treated to prevent rusting.—J. W. D.

Metals of low melting points and different specific gravities; Apparatus for separating mixtures of —. F. Hoffmann, Berndorf, Austria. Ger. Pat. 299,724, 2.7.19. Int. Conv., 23.6.16.

A VESSEL for containing the alloy of metals of low melting point to be separated into its constituents, e.g., an alloy of lead with tin or zinc, a delivery pipe with regulating valve, and a centrifugal separator similar to a hydro-extractor are contained in a heating chamber. A current of a pre-heated reducing gas containing little carbon dioxide, such as producer-gas, water-gas, power-gas, or hydrogen, is passed through the chamber, and to ensure uniform heating the gas is led in from all sides through a number of adjustable openings. On leaving the chamber the gas is mixed with air and burnt, and the heat produced is utilised to heat the fresh supplies of gas.—L. A. C.

Metallic coatings on the inner walls of tubes, etc., of small diameter; Process for depositing —. R. Mylo, Charlottenburg. Ger. Pat. 312,409, 27.11.17.

THE wire anode is stretched between two vessels of insulating material attached to the ends of the pipe or channel to be coated, which vessels serve also for the admission and discharge of the electrolyte. The tautness of the anode may be regulated by an electro-magnet. The anode and the walls of the channel to be coated may move relatively to each other, either in the longitudinal direction or about the axis or in both directions. The arrangement is described in connection with the copper plating of the interior of a channel extending longitudinally through an arc-light electrode, a copper wire being used as anode.—J. F. B.

Electric arc welding and repairing. C. J. Holslag, South Orange, N.J., U.S.A. Eng. Pat. 128,186, 21.11.18. (Appl. 19,147/18.) Int. Conv., 11.6.18.

Coating sheet metal with tin or other metal [on one side only]; Machine for —. J. Powanda, Ansonia, Conn., U.S.A. Eng. Pat. 135,327, 4.12.18. (Appl. 20,076/18.)

Manganese steel; Method of recovering —. E. C. R. Marks, London. From American Manganese Steel Co., Chicago, Ill., U.S.A. Eng. Pat. 134,917, 12.11.18. (Appl. 18,525/18.)

SEE U.S. Pat. 1,291,656 of 1919; this J., 1919, 261 A.

Manganese steel; Method of making —. E. C. R. Marks, London. From American Manganese Steel Co., Chicago, Ill., U.S.A. Eng. Pat. 131,918, 12.11.18. (Appl. 18,526/18.)

SEE U.S. Pat. 1,291,655 of 1919; this J., 1919, 261 A.

Steel, iron, or other magnetisable metal; Method of and means for indicating the condition of — during heat treatment. L. W. Wild and E. P. Barfield, London. U.S. Pat. 1,321,347, 11.11.19. Appl., 22.5.18.

SEE Eng. Pat. 112,891 of 1917; this J., 1918, 153 A.

Ores; Concentration of —. W. A. Scott, Chicago, Ill., U.S.A. Eng. Pat. 118,627, 11.8.18. (Appl. 13,209/18.) Int. Conv., 17.8.17.

SEE U.S. Pat. 1,246,665 of 1917; this J., 1918, 62 A.

Alloying furnaces. F. L. McGahan, Los Angeles, Cal., U.S.A. Eng. Pat. 131,927, 13.11.18. (Appl. 18,616/18.)

SEE U.S. Pat. 1,290,268 of 1919; this J., 1919, 183 A.

Alloys; Manufacture of metal —. W. L. Turner, Atherton, and H. A. Blackwell, Blackpool. U.S. Pat. 1,321,684, 11.11.19. Appl., 7.1.18.

SEE Eng. Pat. 123,103 of 1917; this J., 1919, 225 A.

Zinc; Recovery of — by electrolysis. R. H. Stevens, Risdon, Tasmania, Assignor to Electrolytic Zinc Co. of Australasia Proprietary, Ltd., Melbourne, Australia. U.S. Pat. 1,322,071, 18.11.19. Appl., 6.5.19.

SEE Eng. Pat. 126,296 of 1919; this J., 1919, 727 A.

Zinc-bearing ores; Treatment of — for the recovery of zinc by electro-deposition. H. W. Gepp and D. Avery, Melbourne, and R. H. Stevens, W. C. Snow, and W. M. Whitecotton, Risdon, Tasmania, Assignors to Electrolytic Zinc Co. of Australasia Proprietary, Ltd., Melbourne, Australia. U.S. Pat. 1,322,101, 18.11.19. Appl., 6.5.19.

SEE Eng. Pat. 126,296 of 1919; this J., 1919, 727 A.

Roasting blende, pyrites, or other minerals; Rotary furnace for —. H. C. Bingham, Assignor to Huntington, Heberlein and Co., London. U.S. Pat. 1,322,223, 18.11.19. Appl., 25.3.19.

SEE Eng. Pat. 118,940 of 1917; this J., 1918, 659 A.

Potassium salts from blast-furnace slag. Eng. Pat. 134,665. See VII.

Electric resistance alloy. U.S. Pat. 1,321,294. See XI.

Electrolytic method. U.S. Pat. 1,322,494. See XI.

XI.—ELECTRO-CHEMISTRY.

Mercury; Preparation of colloidal — by cathodic disintegration. A. Gutbier and G. L. Weise. Kolloid-Zeits., 1919, 25, 97—100.

MERCURY sols may be prepared by striking an arc between a clean mercury surface and a thin platinum foil cathode under water or a solution of a protecting colloid such as gum arabic. The sols are grey in colour and not very stable. They coagulate irreversibly on the addition of very small quantities of electrolytes. (See also J. Chem. Soc., Jan., 1920.)—J. F. S.

Colloids; Synthesis of — by means of oscillating arcs. G. Börjeson and T. Svedberg. Kolloid-Zeits., 1919, 25, 151—158.

FAIRLY stable colloidal solutions of antimony, lead, bismuth, zinc, gold, platinum, silver, copper,

aluminium, and cadmium are prepared by burning an oscillating arc under alcohol. Using a capacity 12.8×10^{-2} MF and a current of 1.5 amps. at 400 volts the colloidal solutions are produced rapidly.

—J. F. S.

Electrical endosmosis of molten sodium chloride into carbon. Wa. Ostwald. Kolloid-Zeits., 1919, 25, 115—116.

WHEN molten sodium chloride is electrolysed between carbon electrodes there is scarcely any evolution of chlorine or formation of sodium unless special conditions are established, but the negative electrode increases in bulk and is partially destroyed. On cooling and treating with water the electrode falls to powder, whereas the positive electrode is unaffected either during electrolysis or on treatment with water. This behaviour is due to the cataphoresis of molten sodium chloride.—J. F. S.

Luminous phenomena observed in the neighbourhood of a plate of graphite raised to a high temperature by means of an electric current. G. A. Hemsalech. Comptes rend., 1919, 169, 915—918.

WHEN a sufficiently powerful current is passed through a graphite plate held in a horizontal position and having its upper surface covered with carborundum powder, certain luminous phenomena are observed. When the plate is incandescent yellow vapours appear from the carborundum, and as the temperature rises these turn bluish, become luminous, and give a continuous spectrum. When the temperature reaches 2500° — 2700° C. the space bounded by the plate and the air convection currents set up becomes filled with a luminous vapour, which gives a spectrum of rays and bands. At 3000° C. a red fringe, also giving a spectrum of rays and bands, appears on the lower surface of and in contact with the plate, and its position can be controlled by means of a powerful magnetic field set up by an electro-magnet. (See further J. Chem. Soc., Jan., 1920.)—W. G.

Thermal conductivity of solid insulators. W. M. Thornton. Phil. Mag., 1919, 38, 705—707.

IN the case of solid insulators such as quartz, glass, graphite, paraffin wax, marble, and the like, the author finds that the thermal conductivity κ is related to the density ρ of the solid insulator, and the velocity, v , of sound therein, by the relation $\kappa = v^2 \rho^2$.—J. S. G. T.

Chlorine cells. Wheeler. See VII.

Reduction of barium nitrate by alternating current. Wenger and Lubomirski. See VII.

Electrometric analysis. Müller. See XXIII.

PATENTS.

Electric furnaces. H. A. Kent, London. Eng. Pat. 133,485, 28.10.18. (Appl. 17,543/18.)

A RESISTER or crucible is mounted between conductive parts, or electrodes carried by the conductive parts, which may be partly rotated in bearings or equivalent supports, the conductive parts carrying discs or the like dipping into mercury in receptacles in connection with the electric supply mains. Means are provided for cooling the conductive parts or electrodes, and good contact is maintained between the resistor or crucible and the conductive parts or electrodes by means of a spring device.

—B. N.

Electric furnace. C. Masera, Turin, Italy. U.S. Pat. 1,320,881, 1.11.19. Appl., 9.12.18.

ELECTRODES in an electric furnace are raised or lowered by means of a terminal clamp secured to the electrode and to a ring which carries nuts through which screws pass. A cooling jacket

telescoping with the clamping cylinder is fitted on the furnace roof.—C. A. K.

Electrolytic cell. R. Q. Brown, Kansas City, Mo. U.S. Pat. 1,318,724, 14.10.19. Appl., 21.2.17.

THE cell comprises a receptacle forming the cathode, and provided at its upper end with flanges projecting outwards, a cover being bolted on, but insulated from the flanged end. A pair of bolts with angular heads is secured to and against the outer side of the cathode, and against the under side of one of the flanges, but spaced from and projecting upwards beyond the cover. The latter is provided with a bell depending into the cathode, with a sack attached to the lower end of the bell and out of contact with the cathode. The water level within the cathode is indicated by a float, and a pair of independent gas tubes are provided, within and without the bell, extending downwards but insulated from the covering, and terminating short of the lower end of the bell. A plate anode is disposed within the bell and sack, but out of contact with both, and is provided with a pair of headed bolt extensions extending up through but insulated from and clamped to the cover.—B. N.

Electrolytic cell. H. A. Hallum, Seattle, Wash. U.S. Pat. 1,318,993, 14.10.19. Appl., 30.7.18.

THE cell, acting as a negative electrode and a container for the electrolyte, is provided with an inner peripheral flange about its upper end to which is fastened a plate with apertures. A positive electrode with a threaded stem passes through and is insulated from the plate, and an oxygen bell is mounted upon, but insulated from the stem. The bell is provided with an oxygen outlet passing through an aperture in the plate, but insulated therefrom. A diaphragm secured to the marginal edge of the bell surrounds the positive electrode, and means are provided for holding the lower portion of the diaphragm spaced apart from the positive electrode.—B. N.

Electrolytic apparatus. E. A. le Sueur, Ottawa, Canada, Assignor to The Diamond Match Co., Chicago, Ill. U.S. Pat. 1,319,715, 28.10.19. Appl., 30.3.17.

A RELATIVELY heavy "feeder," provided with a support, is in electrical contact with and throughout the length of a narrow marginal portion of a metallic anode sheet, the marginal portion and the adjacent surface of the feeder being coated with insulating material. Lateral members embrace the feeder and the anode sheet, clamping together the portions in contact.—B. N.

Electrolytic method, apparatus, and product. M. M. Merritt, South Middleton, Mass. U.S. Pat. 1,322,494, 18.11.19. Appl., 15.4.19.

IN a method of depositing metal electrolytically on wire or other flexible cathode, the latter is passed in helicoidal form about two tiers of sheaves, the sheaves of each tier being free to rotate relatively to the co-operating sheaves, in order to equalise the strain on the wire, whilst the electrolyte is circulated past the runs of wire between them.

—W. E. F. P.

Electric battery. E. A. Sperry, Brooklyn, N.Y. U.S. Pat. 1,321,947, 18.11.19. Appl., 24.9.15.

A BATTERY plate is prepared by depositing lead electrolytically on a copper base provided with openings, coating the plate so prepared with lead oxide and a carbonaceous substance, and highly heating the surface of the coating.—W. E. F. P.

Electric cell. H. E. Evans, Buffalo, N.Y. U.S. Pat. 1,322,486, 18.11.19. Appl., 1.6.18.

AN electric cell consists of a receptacle containing a depolarising fluid (bichromate) in which the posi-

tive electrode (a porous carbon cup) is immersed. The latter contains a solution of sodium zincate in which the negative electrode (zinc) is suspended.

—W. E. F. P.

Gases; Apparatus for the electrical treatment of —. C. W. J. Hedberg, New Britain, Conn., Assignor to Research Corporation, New York. U.S. Pat. 1,319,706, 28.10.19. Appl., 26.2.18.

A COLLECTING electrode comprises a treater chamber and a number of vertically spaced pivoted plates having aligned orifices to form a conduit for the gases. A discharge electrode extends into the conduit, and means are provided for turning the plates about their pivots and for rapping them during the turning movement.—B. N.

Electrode. H. B. Conover, Steubenville, Ohio. U.S. Pat. 1,322,163, 18.11.19. Appl., 24.11.16.

A DISCHARGE electrode disposed centrally within a tubular collecting electrode consists of a metal rod upon which metal discs are fastened in spaced relation, the edges of each disc being rounded to increase its discharge surface.—W. E. F. P.

Electric-resistance element and alloy therefor. F. L. Driver, jun., Newark, N.J., Assignor to Driver-Harris Co., Harrison, N.J. U.S. Pat. 1,321,294, 11.11.19. Appl., 7.2.19.

AN iron alloy contains a low percentage of chromium, together with manganese and a metal such as nickel or cobalt. The nickel and manganese contents lie within the approximately constant resistance zone and may be 50% Ni and 10% Mn.

—C. A. K.

Electrode. J. C. King, Montreal, Canada. U.S. Pat. 1,322,491, 18.11.19. Appl., 21.3.17. Renewed 7.4.19.

AN electrode having a surface coating consisting of silica, 25—90, and graphite, 75—10%.—W. E. F. P.

Insulation for electrical machines and transformers; Drying —. A.-G. Brown, Boveri et Cie., Baden, Switzerland. Ger. Pat. 313,993, 14.7.17.

THE insulation of electrical machines, e.g., oil-transformers, is placed, before assembling the parts, in a heated chamber containing a hygroscopic substance for absorbing any moisture. A current of an inert gas, such as air from which the oxygen has been removed, is passed through the chamber, and thus oxidation of the oil and other insulating material is prevented.—L. A. C.

Electric furnaces; Electrodes of —. Soc. Electro-Metallurgique Française, Paris. Eng. Pat. 121,282, 29.11.18. (Appl. 19,756/18.) Int. Conv., 3.12.17.

SEE U.S. Pat. 1,315,992 of 1919; this J., 1919, 870A.

Alkaline chloride solutions which are to be electrolysed; Purification of —. C. N. Riiber, Trondhjem, Norway. Eng. Pat. 135,141, 12.6.19. (Appl. 14,875/19.)

SEE U.S. Pat. 1,308,509 of 1919; this J., 1919, 630A.

Hydrogen peroxide. Eng. Pat. 120,045. See VII.

Concentrating solutions. Eng. Pat. 134,593. See VII.

Magnesia. Eng. Pat. 134,626. See VII.

Oxidation of nitrogen. Ger. Pat. 298,952. See VII.

Electric resistor. U.S. Pat. 1,321,682. See X.

Sterilising. U.S. Pat. 1,319,238. See XIXA.

Water purifying. U.S. Pat. 1,320,118. See XIXB.

XII.—FATS; OILS; WAXES.

Cottonseed oil; Colour standards for —. H. V. Army, C. Kish, and F. Newmark. J. Ind. Eng. Chem., 1919, 11, 950—953.

It was found by Priest (Proc. Soc. Cotton Products Analysts, 1913, 6) that there were considerable variations in the colours of different standard sets of Lovibond glasses. On the other hand, it is not possible to use authentic samples of cottonseed oil as standards, since even when sealed up in vacuum cells the oils change in colour in a few months. These difficulties are obviated by the use of standard cobalt-ferric-copper solutions and ammoniacal cobalt-chromate-copper solutions, which are easily prepared, uniform, and permanent (compare this J., 1915, 925). The "Co-Fe-Cu" fluids are prepared by blending a red N/2 cobalt chloride solution in 15% hydrochloric acid (1474 grms. Co per litre) with a yellow N/2 ferric chloride solution in 15% hydrochloric acid (9308 grms. iron per litre), and a blue N/2 copper sulphate solution in 15% hydrochloric acid (158925 grms. of copper per litre). The "Co-Cr-Cu" fluids are prepared by mixing N/10 cobaltamine solution in 2.8% ammonia water (29485 grms. Co per litre) with N/10 ammonium chromate solution in 2.8% ammonia water (0.8666 gm. Cr per litre), and N/10 cuprammonium solution in 2.8% ammonia water (31785 grms. Cu per litre). Samples of these liquids mixed in various proportions had not faded to any appreciable extent after six years, showing the same values when compared with the same Lovibond glasses. In matching cottonseed oil with these standard fluids the two liquids are placed side by side in 0.5 in. cells in the Lovibond instrument. In the results of typical tests, given in tabular form, "Prime White" samples of oil were matched by blends ranging from a mixture of 6 c.c. of N/2 ferric chloride solution and 0.4 c.c. of N/2 cobalt chloride solution, with sufficient water to give 50 c.c., to a mixture of 16 c.c. of ferric chloride solution, 1.4 c.c. of cobalt chloride solution, and water to 60 c.c. Analogous limits are given for other grades of cottonseed oil. The results were in accordance with the Lovibond readings.

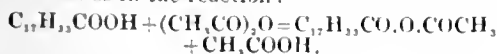
—C. A. M.

Oil from sumac (Rhus glabra). H. W. Brubaker. J. Ind. Eng. Chem., 1919, 11, 950.

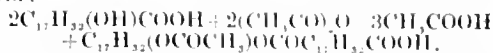
The air-dried seeds of the common sumac (*Rhus glabra*) yielded on extraction with ether an average of 11.71% of a deep yellow oil, with pleasant odour and taste. When cooled it solidified to a semi-solid mass at -16°C ., and exposed in a thin film to the air increased 1.66% in weight in seven days, as compared with linseed oil 9.30%, and cottonseed oil 0.65% under the same conditions. It had the following average characters: Sp. gr. at 15°C ., 0.92577; refractometer reading (Abbé) at 20°C ., 1.4710; acid value, 0.9; saponif. value, 192.6; acetyl value, 9.23; iodine value, 126.78; soluble fatty acids, 0.766%; insoluble fatty acids, 93.54%. Fatty acids: M. pt., 17°C .; solidif. pt., 6°C .; refractive index, 1.470; and iodine value, 121.8. The oil is readily saponifiable, forming a semi-solid sodium soap. It might be used as an edible oil or as a semi-drying oil in the paint industry.—C. A. M.

Acetyl value [of fats]; Errors in the determination of the — and their remedy. A. Grün. Oel u. Fettind., 1919, 1, 339—341, 364—367. Chem. Zentr., 1919, 90, IV., 645—646.

The tendency of hydroxy-acids to form inner anhydrides is a source of error which is not entirely eliminated in Lewkowitsch's method of determining the acetyl value, since mixed anhydrides may be formed as in the reaction:—



When such anhydrides are formed too much acetic acid will be found by the distillation method. A more serious source of error is that caused by the formation of inner esters of hydroxy-fatty acids. For example, ricinoleic acid, after eight hours' boiling with acetic anhydride, was converted not into acetylricinoleic acid but into acetyldiricinoleic acid:—



In the case of this by-reaction the acetyl values determined by Lewkowitsch's method will be too low, whilst by Benedikt and Ulzer's method they will be too high. An objection to the ordinary methods of determining the acetyl values of neutral fats is that reciprocal esterification between the triglyceride and acetic anhydride may take place, whilst it is difficult to distinguish between the different forms of hydroxylated constituents indicated by the value. To obviate these difficulties a method has been devised in which the fatty acid esters are acetylated. These esters are prepared by heating the fat with methyl or ethyl alcohol and 1 to 2% of hydrochloric or sulphuric acid, evaporating the alcohol and neutralising, washing, and drying the residue. Neither acid anhydrides nor inner esters can be formed on acetylating these esters, which is preferably done by the original method of Benedikt and Ulzer, after removal of volatile esters. The acetyl value of free acids is obtained by multiplying the acetyl value of the esters by the theoretical acetyl value of the free acids and dividing by the theoretical acetyl value of the esters. In the case of the methyl esters, calculation into the triglyceride is unnecessary, since the difference between the groups C_3H_5 and the group C_3H_7 is negligible. For the determination of the amount of mono- and diglycerides the neutralised fat is acetylated and the acetic acid content of the product estimated. The difference between the acetyl value of the neutral fat and that of the mixture of methyl esters prepared from it corresponds to the amount of mono- and diglycerides. Acetylation of the fatty acid esters instead of the free acids or neutral fat is also advisable for the determination of the hydroxyl value.—C. A. M.

Soap; Lathering of —. J. Leimdörfer. Seifensieder-Zeit., 1919, 46, 273—274, 295—296, 317—318, 339—340. Chem. Zentr., 1919, 90, IV., 751—755.

The difference between the behaviour of castor oil soaps and that of palm-kernel and coconut oil soaps is explained by the relations of these oils towards electrolytes. The author has studied the concentrations of electrolytes required to precipitate the various soaps. Castor oil soap is the most resistant to flocculation, and is salted out only by sodium hydroxide. The reduced lathering power of the salted-out castor oil soap is connected with its high content of electrolyte. The capacity for swelling of the soap in water is lowered by the solution of the electrolyte. Stearin soap is another striking case; it is typical of those soaps in which the lathering power tends to be suppressed through the predominance of hydrolysis. Sulphonated oils, even those prepared from rape oil, are far less sensitive to electrolytes than coconut oil soaps. One hundred parts of sulphonated rape oil can substitute 110 parts of coconut oil, and 70 of sulphonated rape oil is equivalent to 100 of palm-kernel oil. Soaps made from rape oil in combination with sulphonated rape oil by the cold process have excellent lathering power and are quite hard.—J. F. B.

PATENTS.

Copra; Process of and apparatus for washing and drying —. C. P. Park, Caulfield, Victoria. Eng. Pat. 134,982, 27.11.18. (Appl. 19,609/18.)

Is apparatus for washing and drying copra the

cracked nuts are fed through a hopper on to an endless conveyor, by means of which they are carried through an alkaline solution and are then dropped through an opening in the side of a vertical dryer on to the top of a series of drying tables. The material is carried over the tables, in opposite directions on alternate tables, by means of endless conveyors. The drying vessel is heated, *e.g.*, by means of a number of coils containing superheated steam. Washing with an alkaline solution previous to drying removes "milk" from the surface and prevents the formation of fungus and mildew on copra.—L. A. C.

Oils; Refining vegetable — C. L. Riley, North Plainfield township, N.J., Assignor to Clark, MacMullen, and Riley, New York. U.S. Pat. 1,320,093, 28.10.19. Appl. 16.11.17.

A MIXTURE containing a partially-refined vegetable oil and soap is introduced into a solvent for soap, and portions of the soapy solvent are withdrawn. A fresh supply of solvent is introduced and the diluted solvent re-circulated.—B. N.

Hydrogenation catalysts; Process of re-activating spent — H. Schlinek und Co. A.-G., Hamburg. Ger. Pat. 313,192, 22.1.96.

SPENT catalyst from hydrogenation processes is freed from adherent organic matter and treated with acid in insufficient quantity for complete solution of the whole of the catalytic metal, and the dissolved portion is then reprecipitated upon the undissolved portion. The activity of the catalyst is in this way as completely restored as if the whole of the metal had been dissolved. Sodium bisulphate may be used in place of acid for dissolving the metal.—C. A. M.

Cleaning compound. L. C. Gorius and F. B. Hays, Indianapolis, Ind. U.S. Pat. 1,322,009, 18.11.19. Appl., 7.6.15. Renewed 25.4.19.

THE solid matter precipitated from a mixture of 27% to 42% of corn (maize) syrup, 25% to 28% of water, and 48% to 30% of a mixture of sodium hydroxide and soda ash in equal volumes, is separated from the liquor and mixed with two parts by weight of water to form a cleaning compound.—L. A. C.

Soap substitute. J. Perl und Co., Berlin. Ger. Pat. 313,319, 1.10.16. Addition to Ger. Pats. 308,609 and 311,160 (this J., 1919, 21 A, 545 A).

THE prepared cakes are treated with water after they have been separated for a short time in order to prevent them subsequently becoming hard. Most of the uncombined magnesium chloride is thereby dissolved away.—J. F. B.

Soap or saponaceous composition and process of manufacturing the same. W. Feldenheimer, London, and W. W. Plowman, East Sheen. U.S. Pat. 1,321,516, 11.11.19. Appl., 22.3.19.

SEE Eng. Pat. 125,491 of 1918; this J., 1919, 427 A.

Waxes, oils, etc. Eng. Pat. 133,989. See IIa.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Hiding power of white pigments and paints. A. H. Pfund. J. Franklin Inst., 1919, 188, 675—681.

THE hiding power of a paint is defined as the thinnest layer which will hide the background as effectively as does an infinitely thick layer. A "cryptometer" for determinations of hiding power on this principle is described. A plate of glass

(14×5×0.6 cm.) has its lower surface coated with black baking enamel to serve as a black background. The upper surface is optically flat and has a transverse groove 2 mm. deep and 1 cm. wide cut in it. Another glass plate (7×3.5×0.6 cm.) with its under surface optically flat has a narrow strip of steel 0.45 mm. thick attached to one edge of its under side, and rests on the larger plate so that a wedge-shaped layer of white paint under examination may be formed between the plates. This wedge of paint terminates abruptly at the "infinitely thick" layer formed by the paint in the transverse cut, the line of demarcation being visible so long as the hiding is not complete. The wedge is moved backwards and forwards along a direction parallel to the length of the plates, and the mean of the points between which the line of demarcation with the "infinitely thick" layer is visible and that at which it disappears is taken. By taking ten pairs of readings the average deviation from the mean is found to be 3—4%. From a knowledge of the angle of the wedge and the reading of a scale on the lower plate the thickness of the critical layer lying immediately above the point found may be calculated. A viewing device consisting of an arrangement of blackened shields to cut out extraneous light and to allow the plates to be illuminated from one side of the observer only may be used. From a knowledge of the relative proportions of pigment and oil and the thickness of the critical layer, hiding powers of pigments are returned as area in sq. cm. covered by 1 gm. The hiding powers of paints are similarly returned as area in sq. ft. covered by 1 gal. In determinations of hiding powers of a number of pigments it is not expedient to prepare pigment-oil mixtures of constant numerical composition as the different "oil absorptions" of the pigments would thereby result in the production of paints of varying and in some cases impracticable consistency. A standard "painting consistency" over a range of samples is therefore aimed at, and allowances made in the calculation for the different proportions of pigments in the paints. The results for characteristic pigments and paints are given in a table in which the non-relation of the order of the hiding powers of pigments to that of their corresponding paints is noteworthy, the divergence being merely due to differences in oil absorption. Comparative determinations of hiding powers of three paints by the craftsman's method of application of a sufficient number of coats of paint to produce obliteration of a black cross on a white undercoat agreed within reasonable limits with those using the cryptometer.—A. de W.

Lead chromate; Iodometric determination of chromic acid in — M. Gröger. Z. anorg. Chem., 1919, 108, 267—272.

THE chromic acid in lead chromate can be estimated iodometrically by dissolving the chromate in hydrochloric acid if the strength of the latter is so chosen that no reduction of the chromic acid takes place. Experiments showed that when hydrochloric acid of 1.25*N* strength or less was used, in the proportion of 25 mols. HCl to 1 mol. of chromate, no reduction took place even after boiling the solution for three hours. With stronger acid, however, the rate of reduction increased rapidly with the concentration of the acid. The analysis is carried out as follows. About 0.3 gm. of the lead chromate is heated gently with 50 c.c. of 1.25*N* hydrochloric acid until all has dissolved. The solution is cooled. 1 gm. of potassium iodide added, allowed to stand for ten minutes, diluted with 100 c.c. of water and the free iodine titrated with sodium thiosulphate. The precipitated lead iodide does not interfere with the end point. A short time after the titration is finished, free iodine again appears, due to atmospheric oxidation of the hydriodic acid, a reaction which appears to be catalysed by lead iodide. This

method can be used for determining the chromic acid in basic lead chromates and in pigments containing lead chromate. The results always tend to be about 0.5% too high.—E. H. R.

Tetralin, a new turpentine substitute. Vollmann. *Farben-Zeit.*, 1919, 24, 1689—1690. *Chem. Zentr.*, 1919, 90, IV., 668.

"TETRALIN" or tetrahydronaphthalene and "tetralin extra," a mixture of tetralin with "dekalin" or decahydronaphthalene, are serviceable turpentine substitutes. Their high boiling points, by diminishing the risk of fire when working in open pans, simplify the manufacture of lacquers and varnishes, and owing to their low volatility the loss by evaporation is small. The solvent power for natural gums, coumarone, indene, and tar resins for linoxyn, oils, and fats is far superior to that of almost all known liquids. "Tetralin extra" has a somewhat smaller solvent action and higher vapour tension than pure tetralin. It is therefore to be preferred as an addition to lacquers. Lacquers prepared by the addition of "tetralin" or "tetralin extra" have excellent covering power. "Tetralin extra" behaves as an oxygen carrier.

—H. J. H.

Oil from sumac. Brubaker. See XII.

Ultramarine blue. Kalshoven. See XVII.

PATENTS.

Water-proof paint. S. Kuroki and M. Makayama. Tokyo, Japan. Eng. Pat. 135,132, 16.5.19. (Appl. 12,343/19.)

WHEAT flour (8½ lb., i.e., about 3850 grms.) is kneaded with 2000 grms. of water for two hours, and is then washed with water to remove the starch. The gluten is maintained at 75°—80° F. (about 24°—27° C.) for 5 to 7 days until a viscous liquid readily miscible with either water or oil is obtained. One part of camphor oil is added to 5 parts of the liquid, and then 3 parts of formalin is added to 10 parts of the mixture. The product may be used as a waterproof paint, or may be mixed with china clay and a pigment, dried and ground, to form a material for use in the manufacture of paint.

—L. A. C.

Shellac, resin, or the like; Production of synthetic —. (b) (c) *Substitutes for celluloid, vulcanite or the like.* W. T. Robinson-Bindley, A. W. Weller, and E. Duleken, London. Eng. Pat. 134,563, 27.2.18. (Appl. 3501/18.)

SYNTHETIC shellac, resin, or the like is obtained by adding a mineral acid catalyst, e.g., hydrochloric acid, to formaldehyde or an equivalent quantity of a substance adapted to yield formaldehyde, which is then condensed with *m*- or *p*-cresol by heating to about 80° C. The temperature is reduced when the mixture bubbles, heating being discontinued when the viscous mass separates from the cloudy liquid. The viscous mass is poured off, washed free from cresol and free hydrochloric acid by means of steam at about 120° C., and finally heated in a vacuum to extract all liquid, after which it is cast into moulds. The product may be toughened by further heating in an autoclave to about 140° C. at 100 lb. pressure whilst in the moulds. A spirit-soluble product is obtained when 70 parts of *m*-cresol is condensed with 50 parts of 40% formaldehyde and 5 parts of 30% hydrochloric acid, an oil-soluble product being produced by condensing 50 parts of *p*-cresol with 70 parts of 40% formaldehyde and 5 parts of 30% hydrochloric acid.—A. de W.

Paints and methods of making same. E. T. Goldthorpe, Chicago, U.S.A. Eng. Pat. 135,368, 20.1.19. (Appl. 1462/19.)

SEE U.S. Pat. 1,295,730 of 1919; this J., 1919, 330 A.

Printers' roller composition; Reclaiming of used —. T. H. Grozier, Longueville, N.S.W. U.S. Pat. 1,321,789, 11.11.19. Appl., 13.6.18.

SEE Eng. Pat. 121,911 of 1918; this J., 1919, 378 A.

Pyroxylin composition. U.S. Pat. 1,320,458. See V.

Ethylidene esters. Ger. Pat. 313,696. See XX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Grinding machines [for sawdust to be used in rubber mixings or for leather substitutes]. T. P. D. Marshall, Bradford. Eng. Pat. 131,268, 11.10.18. (Appl. 16,566/18.)

SAWDUST is reduced to a fine powder in a roller mill with granite or plain or fluted metal rollers; the flutes of the last may be made with cutting edges. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents Designs Act, 1907, to Eng. Pat. 27,587 of 1903 and 16,055 of 1907, this J., 1905, 149.)—B. M. V.

Rubber; Process of recovering plastic — from fabrics. T. F. Furness, Cynwyd, Pa., Assignor to Acushnet Process Co. U.S. Pat. 1,321,200, 11.11.19. Appl., 20.4.17. Renewed 25.9.18.

RUBBER-COATED fabric is progressively disintegrated in presence of water, and the plastic rubber is simultaneously stripped from the fabric and from the threads and fibres resulting from its disintegration.—E. W. L.

Rubber and cotton; Process for reclaiming — from waste. P. E. Young, Fairhaven, Mass., Assignor to Acushnet Process Co. U.S. Pat. 1,321,201, 11.11.19. Appl., 19.9.18.

RUBBER-CANVAS waste is wetted and then sheeted by passing it between rollers. The sheet is fed gradually to a high-speed picker, whereby the fabric is torn from the sheet in the form of threads and fibres, and the rubber in small balls or particles.

E. W. L.

Rubber; Art of reclaiming —. C. W. Bedford. Assignor to Goodyear Tire and Rubber Co., Akron, Ohio. U.S. Pat. 1,321,501, 11.11.19. Appl., 13.4.18.

FRESH rubber stock containing added protein is vulcanised, and the product is reclaimed and re-vulcanised.—E. W. L.

Rubber; Process for regeneration of — from old rubber and scrap. W. North and H. Loosli, Hannover. Ger. Pat. 313,551, 8.8.17.

THE material is heated with water under a pressure of 200—600 atm. at a high temperature, e.g. 210° C., and in presence of alkaline substances such as caustic soda. Free and also combined sulphur as well as organic and inorganic filling materials are more completely removed than by existing processes, and the rubber is not affected.—H. J. H.

Rubber substitute and process of producing the same. M. Gregory, Tacoma, Wash., Assignor to Western Rubber Co. U.S. Pat. 1,321,788, 11.11.19. Appl., 13.3.16.

SEE Eng. Pat. 123,114 of 1917; this J., 1919, 227 A.

Vulcanite substitute. Eng. Pat. 131,561—5. See V.

XV.—LEATHER; BONE; HORN; GLUE.

Hide and pelt; Biological and chemical history of —. Relation of elastin to the tanning process. W. Möller. Collegium, 1918, 105. Chem.-Zeit., 1919, 43, Rep. 263.

THE elastin of the corium is a complex of a number of colloidal constituents. Its physical properties suggest that it has already undergone a tanning process in the living tissues, and its greater resistance, as compared with collagen, to the action of acids and alkalis is thereby accounted for. Tryptic enzymes are more active towards elastin than towards collagen. The action of the "limes" is to saponify and remove sulphur from elastin, the properties of which then approximate to those of collagen. It is necessary to de-tan these elastic fibres as completely as possible without damaging the collagen, and this is effected by the preliminary processes in the case of sole leather. In the case of upper leather the bating process almost completely removes the elastin, and it is mainly the absence of these fibres which accounts for the difference between a bated pelt and one which has only been delimed with acid, and which gives to the finished leather its characteristic "plastic" feel. It follows that hides rich in elastin tend to give a loose upper leather.—E. W. L.

Aldehyde tannage. W. Möller. Collegium, 1918, 25. Chem.-Zeit., 1919, 43, Rep., 263.

THE course of the aldehyde tannage affords evidence that chemical theories of tanning are incorrect. It is not the presence of a certain radical which determines the tanning action, but the possibility of the formation of a colloidal substance in presence of a "peptising" agent. This is shown by the fact that aromatic aldehydes do not tan when the aldehyde group is attached to the nucleus, but only when it is in an aliphatic side-chain. Tannage with aldehydes therefore depends upon the fact that by the addition of acid or alkali polymerisation and condensation are intensified and the quantity of "peptised" substance thereby increased.—E. W. L.

Gelatin jellies; Relationship between shrinking and diffusion structure in tanned —. W. Moeller. Kolloid-Zeits., 1919, 25, 101—109.

THE structures produced by the diffusion of solutions of sodium hydroxide into sodium chloride-gelatin jellies, which have been hardened by formaldehyde, are due to changes in the internal structure of the micellar envelope. The thin tanned layer attaches itself closely to the underlying coarse structure and follows completely the direction of the coarser structure. Under tension shrinkage lines are formed radially from the diffusion centre, and concentric ring systems are produced by contractions of the micellar envelope.—J. F. S.

Oil from sumac. Brubaker. See XII.

PATENTS.

Scrap and waste leather; Method of and means for the treatment and utilisation of —. S. C. Lansdown, Melbourne, and P. Magnus, North Fitzroy, Victoria. Eng. Pat. 134,592, 29.10.18. (Appl. 17,623/18.)

SCRAP leather is ground to a coarse powder, cleansed, and rendered gelatinous by treatment with a tepid 2% solution of oxalic acid, and allowed to dry in a shady place. The dried, cleansed powder (2 lb.) is mixed with 1 lb. of a mixture consisting of hot liquid glue, 85; boiled linseed oil, 10; "carbolic oil," 21; and oil of cloves, 2½. The resulting mass is formed into sheets or pieces of suitable size and thickness in moulds under a pressure of 4 tons per sq. in., the sheets being after-

wards allowed to dry in the atmosphere. The product is suitable for use for soles and heels of boots and shoes.—E. W. L.

Casein; Process for preparation of horn-like material from —. R. Weiss, Hamburg. Ger. Pat. 313,881, 29.4.17.

CASEIN is kneaded with sulphonated fatty acids, pressure and heat being applied at the same time. The use of a mixture of 3 parts of Turkey-red oil and 17 parts of water leads to products of great plasticity and strength, not brittle nor easily cracked.—B. V. S.

Plastic compositions from vegetable ivory; Manufacture of —. G. C. Devonshire, London, and E. T. Foord, Birmingham. Eng. Pat. 134,619, 5.11.18. (Appl. 18,140/18.)

Plastic material [from gelatin]. A. P. Galley, Assignor to Soc. Française du Ceramoid, Paris. U.S. Pat. 1,321,429, 11.11.19. Appl., 29.1.18.

SEE Eng. Pat. 112,196 of 1917; this J., 1918, 89 A.

Hop-bines. Ger. Pat. 304,387. See V.

Slabs [from leather waste]. Eng. Pat. 124,415. See IX.

Grinding [sawdust for leather substitutes]. Eng. Pat. 134,268. See XIV.

XVI.—SOILS; FERTILISERS.

Cyanamide; Transformation of — into urea by the microbes of the soil. P. Mazé, Vila, and M. Lemoigne. Comptes rend., 1919, 169, 921—923.

NUMEROUS common species of bacteria abundant in all well-cultivated soils can grow in a medium containing one part of cyanamide per thousand and are capable of rapidly converting the cyanamide into urea.—W. G.

Superphosphate manuring of indigo. Davis. See IV.

Enzyme of soya beans. Wester. See XVIII.

XVII.—SUGARS; STARCHES; GUMS.

White sugar; Iron content of plantation —. W. H. T. Harloff. La. Planter, 1919, 63, 61—62. Int. Sugar J., 1919, 21, 574.

REFERRING to the work of Schneller and Zerban (this J., 1918, 778 A; 1919, 192 A) on the formation of colour by the reaction between ferric salts and the polyphenols present in the juice, the author considers that in the manufacture of white sugar under present conditions in Java such compounds are not of great influence. If syrups and molasses high in iron are strongly sulphited (as is common practice in some countries), the sugar crystallising from the acid liquor will contain only slight traces of iron. The yellowish tinge of plantation white sugar is probably due rather to the presence of the calcium salts of acids resulting from the decomposition of reducing sugars during heating in an alkaline medium.—J. P. O.

Ultramarine blue; its composition and examination [for use in sugar refining]. H. Kalshoven. Archief Suikerind. Nederl.-Indië, 1919, 27, 1041—1051. Int. Sugar J., 1919, 21, 575.

ACCEPTING the view that ultramarine may be regarded as a solid solution of colloidal sulphur in different substances, as silica, alumina, phosphorus pentoxide, boron trioxide, it follows that its quality is dependent more upon the manner in which the

mixing of the several ingredients is accomplished than upon their relative proportions. In the examination of ultramarine for use in sugar refining, in addition to the usual determinations of its comparative fineness and colouring power, the following tests may be applied: On boiling with water, no hydrogen sulphide should be evolved; on boiling with alcohol, and with sodium hydroxide, no colour should pass into solution; and on heating with hydrochloric acid, complete decolorisation with evolution of hydrogen sulphide should occur.

—J. P. O.

Dextrose; Action of alkaline-earth carbonates on — H. Murschhauser. *Biochem. Zeits.*, 1919, 97, 97—113.

A SOLUTION of dextrose boiled with calcium carbonate becomes brown and gradually loses its dextro-rotation, which after prolonged boiling is entirely eliminated; the sugar may even become laevo-rotatory. The reducing power of the dextrose is also diminished, but to a much smaller extent. Distilled water previously shaken with calcium carbonate produces the same change, but the reduction in the rotation becomes constant after some time because the dissolved carbonate is neutralised by the acid formed in the reaction. It is concluded that the dextrose is changed into levulose and eventually into other laevo-rotatory or weakly dextro-rotatory sugars.—S. S. Z.

Sucrose; Catalytic action of ferric oxide on — C. Thomae. *Chem.-Zeit.*, 1919, 43, 747.

SUCROSE after contact with tobacco ash is readily brought into violent decomposition by the application of a lighted match at the point of contact. This phenomenon is apparently due to the presence of ferric oxide in the tobacco ash, and sugar which has been rubbed with a piece of dry rusty iron, preferably slightly warm, can be set on fire by means of a match in a similar way.—G. F. M.

Mannitol; Physical properties of — and its aqueous solutions. J. M. Braham. *J. Amer. Chem. Soc.*, 1919, 41, 1707—1718.

PURE mannitol may be obtained from the commercial product by two crystallisations from aqueous alcohol. The pure substance melts at 166.5° C., has $[\alpha]_D^{25} = -0.244 \pm 0.002$; sp. gr. (room temperature) 1.487, specific heat $C(28^\circ \text{C.} - 100^\circ \text{C.}) = 0.3271$ cal./deg. $C(14^\circ - 26^\circ) = 0.315$ cal./deg. and heat of combustion 4.00° cal./gram.—J. F. S.

Hydrolysis of starch. Sherman and Walker. See XVIII.

PATENTS.

Sugar moulds; Method and apparatus for cooling masscuite in — G. A. Widström, Landskrona, Sweden. Eng. Pat. 109,621, 12.9.17, (Appl. 13,092/17.)

MASSECUTE contained in moulds such as are used in the Adant process of manufacturing sugar cubes, is cooled by irrigating both the cylindrical surfaces of the moulds with cold water. A rotary device, supplied from above with cooling water, is fitted centrally to a frame bolted to the top of the mould, and is easily removable. It carries two tubular arms which revolve with it and discharge the water against the inner and outer cylindrical surfaces respectively of the mould.—J. H. L.

Saccharine materials; Decolorising and purification of — J. J. Hood, London, J. Clark, Hutton, Essex, and P. G. Clark, London. Eng. Pat. 134,607, 4.11.18. (Appl. 18,042/18.)

CRUDE saccharine solutions, e.g., the juice expressed from sugar cane or beet, solutions of crystallised saccharine bodies or of artificially prepared glucose,

or the like, are filtered through or agitated with ignited precipitated magnesia, precipitated alumina, magnesite, bauxite, or mixtures of the same. In certain cases, the addition of small quantities of salts such as aluminium nitrate, aluminium chloride, calcium phosphate, sodium carbonate, or the like, to the solution before filtration increases the degree of decolorisation. The filtering medium, when it ceases to be effective, is revived by washing, and igniting at dull redness with the exclusion of air.—L. A. C.

Starch products; Process for the manufacture of — R. W. G. Stutzke, Assignor to The G.A. Buhl Co., Chicago, Ill. U.S. Pat. 1,320,719, 4.11.19. Appl., 19.8.16.

A STARCH solution is desiccated and simultaneously modified by being subjected in finely divided form to a current of heated gas.—A. E. D.

Inulin and larulose; Production of — from plant juices. A. Daniel, Charlottenburg. Ger. Pat. 313,986, 19.9.16.

By heating with strong alkalis, foreign matter is precipitated or converted into an innocuous form, the concentration of alkali being limited to a degree which prevents precipitation in the further treatment of the juice. For example, inulin is obtained by treatment of dahlia tubers with alkali hydroxides, carbonates, and alkaline earths. From pure inulin it is possible to prepare its degradation products, levulose, caramel, and dextrin.

—H. J. H.

Potato starch. Ger. Pat. 301,679. See XIXA.

XVIII.—FERMENTATION INDUSTRIES.

Hydrolysis of starch; Influence of aspartic acid and asparagin on the enzymic — H. C. Sherman and F. Walker. *J. Amer. Chem. Soc.*, 1919, 41, 1866—1873. (Compare Sherman, Walker, and Caldwell, this J., 1919, 651 A.)

THE action of saliva, pancreatin, and purified pancreatic amylase on alkali-washed potato, wheat, maize, and rice starches and on Lintner soluble starch is accelerated by the addition of small amounts of boiled, neutralised water extract of potato, whilst the action of the vegetable amylases tested was not influenced by the addition of potato extract. The addition of neutralised aspartic acid or asparagin accelerated the action of saliva, pancreatin, and purified pancreatic and malt amylases. Clear evidence of activation was not obtained in the case of malt extract or of the preparations made from *Aspergillus oryzae*. The addition of both sodium aspartate and asparagin to the same digestion mixture produces practically the same activation as does one of these substances alone, the activating effects of these substances being interchangeable rather than additive. Activation is not due in these experiments to change in hydrogen ion concentration nor merely to a more favourable concentration of electrolyte. The amino-compounds to be tested were added to a substrate which already contained the optimum concentrations of sodium chloride and phosphate, the reported activation being thus additional to the activity induced by the chloride and phosphate. Moreover, sodium aspartate is not interchangeable with sodium chloride in the activation of purified pancreatic amylase.

—H. W.

Pancreatic amylase preparations; Proteolytic activity of — H. C. Sherman and D. E. Neun. *J. Amer. Chem. Soc.*, 1919, 41, 1855—1862.

HIGH-GRADE commercial pancreatin was purified as described previously (Sherman and Schlesinger,

this J., 1915, 627; Sherman and Neun, this J., 1918, 526 A), except that in the final precipitation the usual 1:1 alcohol-ether mixture was replaced by a 2:1 mixture. A precipitate (A) was thus obtained, after removal of which a second precipitate (B) was isolated by adding more ether to the filtrate. The amylolytic activity of (A) was found to be lower than that of (B), but the latter was more active than the usual amylase preparations; the proteolytic activity was higher in precipitate (A) than in precipitate (B). Interpretation of the results is rendered difficult by the exceedingly unstable nature of the substance under purification and the impossibility of pushing the fractionation further because of the great tendency to loss of amylolytic activity when pancreatic amylase in the absence of salts and carbohydrates is held in solution or subjected to precipitation. On the whole, it seems probable that a partial separation of a mixture of amylase and protease was accomplished, but that amylolytic activity was partially lost because of the extra manipulation, since it deteriorates much more rapidly than proteolytic activity, at least under conditions such as obtained in these experiments. The further possibility that there are enzyme particles which have both amylolytic and proteolytic activities is not excluded.

—H. W.

Yeasts; Analysis of —. E. Vautier. Ann. Chim. Analyt., 1919, 1, 345—347.

THE presence of brewers' yeast in pressed yeast cannot be ascertained by determining the volume of carbon dioxide evolved when the yeast is grown in 10% sucrose solution, since, in given periods of time, both species of yeast yield the same volume of gas. Herzfeld's method may be used as a qualitative test for the purpose. The yeast is added to 1% raffinose solution contained in an Einhorn fermentation apparatus and maintained at 30° C. for 24 hours. If the sample consists of pressed yeast, the sugar is not attacked, but if of brewers' yeast, or a mixture of the same with pressed yeast, the sugar is fermented and carbon dioxide evolved. A control test is run at the same time, using the same weight of yeast and water, but no raffinose. A correction may thus be made for any carbon dioxide evolved as the result of auto-digestion of the yeast. An approximately quantitative test has been proposed by Bau, as follows: 10 c.c. of 1% raffinose solution is placed in each of three test-tubes, to each is added 0.4 gm. of the yeast, and the mixtures are kept at 30° C. After 24 hrs. the contents of one tube are filtered and 3 c.c. of the filtrate is heated with 1 c.c. of Fehling's solution for 5 mins. in a boiling water-bath; if the blue colour is not discharged, the yeast contains at least 10% of brewers' yeast. Should the colour of the Fehling's solution be destroyed, showing that raffinose is still present, the contents of the second tube are tested similarly after 48 hrs.; the non-discharge of the colour of the Fehling's solution in this case indicates the presence of about 5% of brewers' yeast. A blue coloration in the third test after 72 hrs. incubation shows that the yeast contains about 1% of brewers' yeast. If the Fehling's solution remains blue in this third day test, the yeast is free from brewers' yeast.—W. P. S.

Glycerol; Mechanism of the "fixation" method of the decomposition of sugar into acetaldehyde and— C. Neuberg and J. Hirsch. Biochem. Zeits., 1919, 98, 141—159.

At every stage during the process of fermentation of sugar in the presence of sodium sulphite, acetaldehyde and glycerol are produced in equimolecular proportions. As alcohol and carbon dioxide are also produced independently at the same time in equivalent proportions, the fermentation process can be followed by estimating the alcohol and aldehyde at the various stages.—S. S. Z.

Soya-bean extract [urease]; Peculiarity of —on heating at 37° C. D. H. Wester. Chem. Weekblad, 1919, 16, 1461—1463.

If the extract is heated for 3 days at 37° C., its urease-activity is reduced. Further treatment at this temperature does not uniformly reduce its enzymic action, the graph representing the urea equivalents after various periods of heating being a zig-zag.—W. J. W.

Enzyme action; Urease and the radiation theory of — H. P. Barendrecht. Proc. K. Akad. Wetensch., 1919, 22, 29—45.

THE addition of 5% alcohol to urea solutions reduces the activity constant of the urease hydrolysis from 0.00381 to 0.00335. Above a definite concentration of urease the specific activity is constant, but below this value it decreases. (See also J. Chem. Soc., Jan., 1920.)—J. F. S.

Soya-beans; Enzyme of — [Urease.] D. H. Wester. Chem. Weekblad, 1919, 16, 1442—1443.

THE author has investigated the factors which influence the conversion of urea into ammonium carbonate by means of urease. The urea solution must always be freshly prepared; but urease solutions retain their activity on keeping. The concentration of the urea does not influence the process, nor do decomposition products formed act deleteriously. Glycerin may be added to the medium used for extracting urease from soya beans up to 50% of its volume; larger quantities reduce the urease content, and addition of glycerin to the urease-urea mixture retards enzymic action. The extract of canavalia beans is in some respects similar to that obtained from soya beans.

—W. J. W.

Methyl and ethyl alcohols; Differentiation of — Panwitz. Pharm. Zentr., 1919, 60, 441—442.

COPPER sulphate is insoluble in ethyl alcohol, but dissolves in anhydrous methyl alcohol, giving a blue solution. The presence of a small quantity of water, however, destroys the blue coloration, and the quantity of added water must be increased to 35% by volume before the blue colour reappears. At this dilution, ethyl alcohol also dissolves copper sulphate, and the test is therefore untrustworthy for distinguishing between the two alcohols. Water also interferes with the borax flame test for methyl alcohol.—W. P. S.

PATENTS.

Cellulose; Fermentation of — [for the production of acetic acid]. Power Gas Corporation, Ltd., and H. Langwell, Stockton-on-Tees. Eng. Pat. 134,265, 1.10.18. (Appl. 15,942/18.)

A MASH of cellulosic material, such as sulphite pulp or straw, is inoculated with fermenting vegetable matter, e.g., stable manure, and maintained under aerobic conditions at a temperature between 25° and 60° C. The cellulose undergoes acetic fermentation, and calcium carbonate or other suitable substance is added to neutralise the acid as it is produced. The mash must contain nutrient salts and nitrogenous matter, and some sugar or other readily fermentable substance may be added to induce a vigorous fermentation at the commencement. The temperature employed, within the limits stated above, influences the rate of fermentation, but not the yield of acid.—J. H. L.

Hop-bines. Ger. Pat. 304,387. See V.

Yeast. Eng. Pat. 134,168. See XIXA.

Garbage. U.S. Pat. 1,319,515. See XIXb.

XIXA.—FOODS.

Aluminium vessels; Use of — in dairies. Utz. *Z. angew. Chem.*, 1919, 32, 345—346.

ALUMINIUM is not attacked at ordinary temperature by 1% lactic acid solution and only very slightly dissolved when in contact with the solution at 100° C. (about 0.07 grm. in 10 hrs.); serum from spontaneously curdled milk has still less action. The quantities of dissolved aluminium are not injurious to health, and aluminium vessels may be used without hesitation for the storage, transportation, etc., of milk and milk products.—W. P. S.

Bacteria found in milk; Study of the alkali-forming —. S. H. Ayers, P. Rupp, and W. T. Johnson, jun. *U.S. Dept. Agric., Bull.* 782, 1919, 39 pages.

The group of bacteria studied is defined as consisting of those which produce an alkaline reaction in milk without peptonisation of the casein. This group produced the reaction in five days at 30° C. The reaction was not due to ammonia, as ammonia was produced by a few cultures only, and then not until after more than seven days. All the citric acid in the milk was used up and an amount of carbonate corresponding to about half the citric acid was produced. The chief sources of these organisms were milk, soil, and water. Of the species studied 6 were cocci and 62 were bacilli; all were non-sporing. The action of each organism on certain organic compounds was examined, using cultures in a sodium ammonium phosphate medium containing the substance to be tested as the only source of carbon. The change in the hydrogen-ion concentration in the medium was taken as the measure of the alkalinity produced. Dextrose and galactose were fermented by 44 cultures, lactose by 11, sucrose by 2, and raffinose by none. Ethyl, propyl, and amyl alcohols were more readily fermented than mannitol and glycerol. Most of the organisms fermented the sodium salts of pyruvic, citric, malic, lactic, succinic, acetic, propionic, butyric, valeric, caproic, mucic, glyceric, tartaric, malonic, formic, benzoic, and salicylic acids, but oxalic and glycollic acids were unacted on. It was found that urea, uric acid, and hippuric acid could act as the sole sources of both carbon and nitrogen, and also that the organisms could utilise the nitrogen in nitrites and nitrates. (See also *J. Chem. Soc., Jan.*, 1920.)—J. H. J.

Milk analysis; Application of Ackerman's refraction method to —. H. Langkammerer. *Milchw. Zentr.*, 1919, 48, 249—254.

The formulæ given by Wiegner (this J., 1910, 1076) for calculating the non-fatty solids of milk from the refractive index of the milk serum have been applied to milks of various districts and the results obtained compared with those found by the use of Ackermann's formula (*loc. cit.*). Generally, the results agree, but with some milks (from the Frankfurt district) there is a distinct difference; the winter milk contains, apparently, 0.25% more albumin than does the summer milk.—W. P. S.

Milk; Determination of added water in —. J. Goldan, jun. *Ann. Chim. Analyt.*, 1919, 4, 342—345.

WHEN corrected for the volume of fat and casein, the "simplified molecular concentration constant" (see Ferris, this J., 1917, 1245) affords a trustworthy indication of the presence or absence of added water in milk. For genuine milks the value does not fall below 70, the average value being 74. It shows less variation than do the usual analytical values for genuine milks.—W. P. S.

Butyrometer; Composition of the sediment obtained in Gerber's —. F. Reiss and G. Diesselhorst. *Milchw. Zentr.*, 1919, 48, 237—238.

THE small quantity of heavy sediment which is found at the bottom of the centrifuge tube in Gerber's method for the determination of fat in milk consists of hydrated calcium sulphate, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$; it is free from calcium phosphate.—W. P. S.

Butter fat; The Crismer value of —. L. Vandam. *Ann. Falsif.*, 1919, 12, 260—269.

THE Crismer value (critical temperature of solution) of butter fat is determined with alcohol containing 0.9% by weight of water (sp. gr. 0.7976 at 15°/15° C.), but alcohol of other strength may be used provided that the amount of water it contains is known and a correction applied to the results obtained. A mixture of petroleum and vaseline may be employed for determining the quantity of water in alcohol; this mixture is standardised against alcohol-water mixtures of known composition by determining its critical temperature of solution in the same. The results are plotted in a curve and reference to this will give the strength of any alcohol when tested with the petroleum mixture. The following corrections are applied to the Crismer values of butter fats determined with alcohols of various strengths in order to obtain the true Crismer value: For each 0.1% of water between 0 and 0.4%, +1.0; between 0.5 and 0.8, +0.9; between 1 and 1.1, -0.9; between 1.2 and 2.3%, -0.8; between 2.4 and 2.9%, -0.7. The Crismer value decreases as the acidity of the fat increases; this influence is corrected by adding to the value found the number of c.c. of N/10 sodium hydroxide solution required to neutralise 4 grms. of the fat.—W. P. S.

Bromine in mineral waters. Baughman and Skinner. *See VII.*

Oil from sumac. Brubaker. *See XII.*

Yeasts. Vautier. *See XVIII.*

Tyrosine. Weiss. *See XX.*

PATENTS.

Yeast; Process of treatment of brewers' or distillers' — whereby an article of food is obtained resembling extract of meat. J. J. C. A. and F. M. O'Sullivan, Wolsley Bridge, Staffs, and M. L. Marsan, Paris. *Eng. Pat.* 131,168, 30.4.19. (Appl. 10,715/19.)

A MODIFICATION of *Eng. Pat.* 19,161 of 1897 (this J., 1898, 784). Pressed yeast, after undergoing autolysis, is introduced into boiling water and subjected to successive boilings and skimmings, with stirring after each removal of scum, whereby the bitter constituents are completely removed. For example, after the first boiling and skimming the liquid is filtered and again boiled for several hours and skimmed, then left to ripen for some days, and finally concentrated in an open, jacketed pan, with constant stirring, the scums which form being carefully removed. Concentration is continued until the product sets to a solid mass on cooling. Throughout the process the material should be exposed to daylight as far as possible.—J. H. L.

Granulating or finely dividing pasty or semi-fluid materials [e.g. milk]; Apparatus for —. J. C. MacLachlan, St. Paul, Minn., U.S.A. *Eng. Pat.* 134,761, 29.1.19. (Appl. 10,619/19.)

THE liquid, e.g. milk, is discharged through a rotating spraying head at the top of a condensing or evaporating chamber, through which it falls as

rain, meeting warm air moving upwards. The partially evaporated liquid is withdrawn by a pump from the conical bottom of the chamber and passed again through the same chamber or through a second similar chamber, wherein it is completely dried. The rotary spraying head consists of a propeller with downturned overlapping blades, which are rotated at a very high speed, and the liquid which is fed through the hollow spindle is emitted from holes which are not exactly on the axis of the beater, and is thrown by centrifugal force violently against the revolving arms. In addition to a current of hot air induced by external means, the beaters may draw in supplementary currents of cold air through adjacent ducts.—B. M. V.

Sterilising and preserving milk and other fluids; Method and apparatus for —. J. F. Moscrop, Omaha, Nebr. U.S. Pat. 1,319,238, 21.10.19. Appl., 21.3.19.

A TANK constructed of insulating material has baffles of insulating material standing on the bottom and secured alternately to opposite sides of the tank. Vertical conducting plates are placed close together between and with their ends in contact with the baffles, alternate plates standing on the bottom of the tank and the others being spaced therefrom. Alternate plates are connected to different poles of a source of electricity.—J. H. J.

Drying alimentary and other substances; Apparatus for —. A. F. Spawm, Los Angeles, Cal. U.S. Pat. 1,318,931, 11.10.19. Appl., 27.11.18.

A CHAMBER is supplied with heated air from a furnace at one end by flues passing along the bottom. It is divided by a vertical partition into two compartments, which are connected together at the end farthest from the furnace. Drying trays are supported in tiers in each compartment.—J. H. J.

Foodstuffs; Process for preparing — in the manufacture of potato starch. Stärke-Zuckerfabrik A.-G. vorm. C. A. Koehlmann und Co., Frankfurt. Ger. Pat. 301,679, 4.4.16.

THE juice of the potatoes is separated by pressing and is then evaporated and dried. The potatoes, after removing the juice, are used for the production of starch, mixed with pressed or dry pulp, and again dried. The evaporation residue from the juice is mixed.—A. B. S.

Feeding stuff; Production of a meal for use as — from the green parts of plants, especially foliage leaves. Versuchsstation für die Konservindustrie, Berlin. Ger. Pat. 302,426, 17.4.17.

THE material is extracted with alcohol of definite strength (88—92%) at temperatures below freezing point (–1 to –3° C.), whereby only the resinous and colouring matters are removed whilst the fats remain undissolved. Maple-leaf meal prepared in this way contains 17.91% of mineral matter, 24.18% of protein, 2.65% of fat, 24.88% of crude fibre, and 32.98% of nitrogen-free extractives.

—J. H. L.

Milk; Process for removing cream from —. R. Clavel, Basle, Switzerland. Ger. Pat. 314,090, 24.1.18. Int. Conv., 28.11.17.

MILK is converted into a froth by passing gases through it and is then poured on to a sieve whereby the cream is separated from the milk on account of its lower surface tension. By treating the separated milk intensively with gases casein and albumin may be separated in a similar manner.

—A. B. S.

XIXB.—WATER PURIFICATION; SANITATION.

PATENTS.

Water purifying and filtering device. E. L. Brillhart, Chicago, Ill., Assignor to Electric Specialty Co., New London, Wis. U.S. Pat. 1,320,118, 28.10.19. Appl., 13.8.17.

A STREAM of water is directed spirally round the inside of a tank and around and between electrodes placed therein in a diametrical plane. From the tank the water passes through scum tanks and filter tanks. Means are provided for reversing the flow through the scum and filter tanks.—J. H. J.

Garbage; Process of treating — and production of carbonic acid gas therefrom. F. Danks, Troy, N.Y. U.S. Pat. 1,319,515, 21.10.19. Appl., 1.4.18.

GARBAGE is degreased, disintegrated, and comminuted, then washed and pressed. The expressed liquid is boiled with dilute acid until the carbohydrates are converted into sugar, then neutralised and fermented, the carbon dioxide evolved being collected.—J. H. J.

Hypochlorite preparations; Process for the production of stable solid — for wound treatment. Farbenfabriken vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 309,106, 15.3.17.

CALCIUM hypochlorite or bleaching powder is mixed with an acid salt, such as acid sodium sulphate, acid sodium oxalate, or acid sodium phosphate. For use the mixture is treated with water or other suitable solvent, a weakly acid hypochlorite solution being obtained which has less irritant action in wounds than has the solution of the calcium salt itself.—B. V. S.

Arsenate of lead [; Preparation containing —]. H. B. Goodwin, Grand Junction, Colo., Assignor to The Latimer Chemical Co. U.S. Pat. 1,322,008, 18.11.19. Appl., 5.8.18.

SUFFICIENT tannin is mixed with lead arsenate to increase its capacity of remaining suspended in water.—L. A. C.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Glucosides; Distillation of certain — under diminished pressure. A. Pictet and H. Goudet. Helv. Chim. Acta, 1919, 2, 698—703.

THE distillate obtained from salicin consisted of an aqueous liquid containing a little furfural and a pasty mass from which levoglucosan, m. pt. 179°—180° C., $[\alpha]_D^{20} = -66.73^\circ$ in aqueous solution, was isolated. Similarly, arbutin gave an aqueous portion containing acetic acid and furfural and a pasty mass from which levoglucosan and quinol were obtained, whilst phloretic acid, phloroglucinol, and levoglucosan were isolated from the semi-solid product from phloridzin. The authors suggest that the glucosides are in general derivatives of levoglucosan (thus accounting for their laevo-rotatory power) and not of dextrose, which is only formed as a secondary product of their hydrolysis in acid solution. (See also J. Chem. Soc., Jan., 1920.)

—H. W.

Quinoline bases; Modification of Skraup's synthesis for preparing —. Their conversion into stannichlorides. J. G. F. Druce. Chem. News, 1919, 119, 271—273.

THE method adopted for the synthesis of quinoline from aniline stannichloride (Chem. News, 1918, 117,

346) has been applied successfully, though with diminished yields, to the synthesis of its homologues from aniline homologues. Thus the *o*-, *m*-, and *p*-toluidine stannichlorides, heated with glycerol and sulphuric acid for 2–3 hours and then diluted and diazotised with sodium nitrate gave respectively 8-, 7-, and 6-methylquinoline. The stannichlorides of *m*- and *p*-phenylenediamine gave, on the other hand, very poor yields of phenanthroline and 2-phenanthroline respectively. The stannichlorides of the methylquinolines were prepared by adding stannic chloride to a hydrochloric acid solution of the base. The chlorine in these salts, and also in similar tin salts of both aromatic and aliphatic bases, was estimated with a fair degree of accuracy by simple titration with *N*/10 sodium hydroxide with phenolphthalein as indicator. (See further J. Chem. Soc., 1920, i., 88.) —G. F. M.

Optical isomers; Properties of — from the biological side, A. R. Cushny. Pharm. J., 1919, 103, 483–484.

THE power possessed by moulds and bacteria of discriminating between optical enantiomorphs is also shared by certain tissues of the higher mammals. The nerve ends appear to be the most susceptible of discrimination, whilst such tissues as the brain and muscle seem to be acted upon equally by both isomerides. Organs capable of differentiation must themselves be optically active, and it is noteworthy that in the large majority of cases the optical isomeride which occurs in nature is the one which is reactive towards, or is destroyed by, the living organism, the artificial enantiomorph being immune, or nearly so, from attack. Thus, *l*-asparagine is sweet to the taste, whilst the artificial *d*-asparagine is non-reactive and tasteless. Similarly, *l*-hyoscyamine paralyses the peripheral nerve ends, whilst atropine (*dl*-hyoscyamine) is only half as powerful and *d*-hyoscyamine has only about 1-50th the power of the *l*-isomeride. On the other hand, atropine is more active on the spinal cord of the frog than *l*-hyoscyamine and the *d*-isomeride appears therefore to be stronger than the *l*-isomeride in this case. Adrenaline from the suprarenal gland is laevo-rotatory and causes twice the rise in blood pressure produced by a similar quantity of synthetic adrenaline. It is only possible to produce an optically active substance either directly or indirectly through the agency of life, and optical activity is the most persistent evidence of life which we possess. The optical activity of petroleum is evidence that it must have originated from living tissues.—G. F. M.

Arsenicals for chemotherapeutic research; Plan of procedure for the syntheses of —. Aromatic arsenic compounds. I. W. A. Jacobs and M. Heidelberger. J. Amer. Chem. Soc., 1919, 41, 1581–1587.

THE general plan of a very comprehensive study of the chemotherapeutic value of arsenic compounds is developed. The authors consider that the Salvarsan type of these substances has been fully investigated and therefore turn their attention to products containing quinquavalent arsenic, using, as starting point, *p*-amino- or *p*-hydroxy-phenyl-arsonic acid or a similar derivative, and uniting this with an accessible group of simple type which is readily capable of chemical modification. They regard simplicity in chemical manipulation as a further essential to success. From these points of view, the following classes of compounds have been studied: diazoamino-compounds $A-N:N'NR'$ (in which A is the arylarsonic radical and R and R' hydrogen, alkyl, aryl, or substituted aryl groups); azo dyes, $A-N:N-R$ (in which R is the aromatic

coupler); *N*-substituted amides of *N*-phenylglycine-*p*-arsonic acid, $A-NH-CH_2-CONRR'$ (in which R and R' are hydrogen, alkyl, aryl, or substituted aryl groups); β -substituted ureides of *N*-phenylglycine-*p*-arsonic acid, $A-NH-CH_2-CONH-CONHR$; substituted *N*-phenylglycyl-derivatives of arsanilic acid, $A-NH-CO-CH_2-NHR$; substituted α -phenylglycollyl-derivatives of arsanilic acid, $A-NH-CO-CH_2OR$; and substituted amides of α -phenylglycollic acid *p*-arsonic acid; $A-\alpha-CH_2-CONHR$. Of these the third type has up to the present yielded the most important results.—H. W.

N-Arylglycinecarsonic acids; Amides and alkyl-amides of —. Aromatic arsenic compounds. II. W. A. Jacobs and M. Heidelberger. J. Amer. Chem. Soc., 1919, 41, 1587–1600. (Compare preceding abstract.)

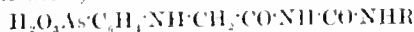
THE condensation of sodium arsanilate with chloroacetic acid to yield phenylglycine-*p*-arsonic acid has been described previously (Ger. Pat. 201,644); it is now found that reaction occurs readily between sodium *p*-aminophenylarsonate, *o*-arsanilate, or certain aminotolylarsonates and the amide or alkylamides of chloroacetic acid, yielding substances of the general formula,



in which R and R' may be hydrogen, alkyl, benzyl, or substituted benzyl groups. The best experimental conditions consist in boiling aqueous solutions of the salt and the simpler chloroacetyl-alkylamines for $\frac{1}{2}$ –2 hours; in the case of the chloroacetylbenzylamines, 50% alcohol is the most serviceable medium and the addition of sodium iodide is found to be necessary. All the glycine-amidearsonic acids are colourless substances which are generally sparingly soluble in the usual neutral media and possess high melting or decomposition points, the latter depending greatly on the rate of heating. They dissolve in alkalis or alkali carbonates to form neutral salts from which they are entirely displaced by acetic acid. They are more feebly basic than arsanilic acid itself, their hydrochlorides being stable only in the presence of concentrated hydrochloric acid. On boiling with excess of alkali or with mineral acids, the amide linking is hydrolysed with formation of the glycine-arsonic acid and the amine. (See also J. Chem. Soc., Jan., 1920.—H. W.)

N-Arylglycinecarsonic acids; Ureides and β -substituted ureides of —. Aromatic arsenic compounds. III. W. A. Jacobs and M. Heidelberger. J. Amer. Chem. Soc., 1919, 41, 1600–1610. (Compare preceding abstracts.)

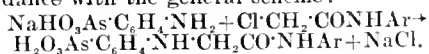
BY replacing the amides of chloroacetic acid by the ureide and its alkyl or aryl derivatives in the reaction described in the preceding abstract, the ureides and substituted ureides of arylglycine-arsonic acids,



(where R may be hydrogen or an alkyl or aryl radical) are obtained. The new series of substances closely resembles the amide series; they form stable and soluble neutral salts with the alkali metals. The ureide linkage, like that of the amides, is easily ruptured, this often occurring even at room temperature in solutions containing an excess of fixed alkali. A lengthy series of compounds derived from *o*-, *m*-, and *p*-arsanilic acid and from methyl-substituted arsanilic acids has been prepared, the most interesting member of which from the physiological point of view is the methylureide of *N*-phenylglycine-*p*-arsonic acid, since it has been found to exert a definite therapeutic effect in experimental syphilis and trypanosomiasis in rabbits. (See also J. Chem. Soc., Jan., 1920.)—H. W.

N-Arylglycinearsonic acids; Aromatic amides of —. Aromatic arsenic compounds. IV. W. A. Jacobs and M. Heideberger. J. Amer. Chem. Soc., 1919, 41, 1610—1644. (Compare preceding abstracts.)

A VERY extensive series of compounds, derived from *o*-, *m*-, and *p*-arsanilic acids and from methyl-substituted *p*-arsanilic acids, has been prepared in accordance with the general scheme:



In the cases of the more reactive chloroacetyl derivatives, reaction may be effected in boiling aqueous solution, but, with the more stable compounds, 50% alcohol is a more suitable solvent and sodium iodide should be added. In general, it is found that *m*-arsanilic acid condenses more readily than the *o*- and *p*-isomerides. The compounds have generally weakly basic and acidic functions; the free arsonic acids do not as a rule possess sharp melting or decomposition points, the values obtained depending greatly on the rate of heating. They are, on the whole, but sparingly soluble in the usual media. The sodium salts, on the other hand, dissolve more or less readily in water, and were prepared for convenience in biological testing and also as a means of purifying the compounds. (See also J. Chem. Soc., Jan., 1920.)—H. W.

Arsenic compounds used as drugs; Behaviour of certain organic — in Marsh's apparatus. D. Ganassini. Boll. Chim. Farm., 1919, 58, 385—390.

THE introduction of platinum chloride into the hydrogen flask of Marsh's apparatus retards or prevents the appearance of the arsenic deposit in the case of cacodylic acid. In the presence of a large excess of platinum chloride cacodylic oxide forms a yellow precipitate of cacodyl platino-chloride. If, however, only a small amount of platinum chloride is introduced, cacodylic oxide is evolved sooner or later. Even for the detection of mineral arsenic it is not advisable to use platinum chloride, since arsenic may be fixed as platinum arsenide, with a loss of as much as 50% of the arsenic present. The yellow ring, probably of erythrasine, produced, together with the ordinary black deposit, by cacodylic acid in the Marsh test is a characteristic reaction. It may be obtained quantitatively by immersing the evolution flask of Marsh's apparatus in a bath of molten lead (about 335° C.). Atoxyl and salvarsan give the ordinary arsenic deposit in Marsh's test, but neosalvarsan and methylarsinic acid behave in an analogous manner to cacodylic acid, giving a yellow ring under the same conditions. In the case of cacodylic acid, however, the gas forms dense white fumes, and when conducted into Bettendorf's reagent (stannous chloride solution saturated with hydrogen chloride) it does not form a yellow precipitate. In the case of methylarsinic acid the formation of the yellow ring is not accompanied by the appearance of white fumes, and the gas gives an abundant precipitate with Bettendorf's reagent. The lemon-yellow ring given by neosalvarsan may be distinguished from the other two by the fact that it is only formed at a high temperature, does not change to black, and is rapidly dissolved by ammonia solution. (See also J. Chem. Soc., Jan., 1920.)—C. A. M.

Tyrosine; Quantitative estimation of — by means of Millon's reaction. M. Weiss. Biochem. Zeits., 1919, 97, 170—176.

A 1/50,000 SOLUTION of tyrosine to which Millon's reagent is added is used as a standard. The solution to be examined is diluted until it gives with Millon's reagent a coloration equal to that of the

standard. The method can be employed for the estimation of tyrosine in hydrolysed proteins.

—S. S. Z.

Saccharin-bicarbonate tablets (110); Chemical alteration in the composition of —. O. Beyer. Chem.-Zeit., 1919, 43, 751—752.

SACCHARIN-BICARBONATE tablets (110 strength) after keeping several months are soluble in water with difficulty and without effervescence. Analysis indicates a corresponding diminution in carbonate radicle and in free saccharin, the latter falling in 12 months from 20% to as low as 0.58% in some cases. Even in the freshly made tablets a certain amount of the saccharin is combined, the quantity varying from 3 to 5% after 14 days. By mixing and compression, therefore, the formation of the soluble sodium sulphinate and liberation of carbon dioxide is already induced, and this continues progressively on keeping the tablets, until eventually, when nearly all the saccharin is in combination, there is practically no evolution of gas on placing the tablet in water, and, its disintegrating influence being removed, the rate of solubility of the tablet is accordingly reduced.—G. F. M.

Cyclohexanones; Transformation of — into catechols. G. Cusmano. Atti R. Accad. Lincei, 1919, 28, ii., 30—33.

METHYLSOPROPYLCATECHOL, $\text{C}_{10}\text{H}_{14}\text{O}_2$, may be obtained by heating monobromo-Buchu-camphor above its melting point, while its diacetyl-derivative is formed when the same bromo-compound is heated with acetic anhydride and sodium acetate. (See also J. Chem. Soc., Jan., 1920.)—T. H. P.

Formaldehyde; Colour reactions of — with certain aromatic compounds. A. Rossi. Boll. Chim. Farm., 1919, 58, 265—270.

SOLUTIONS of a few centigrams. of the following ten aromatic compounds in sulphuric acid give colour reactions with 0.1% and 0.001% solutions of formaldehyde. Gallic acid gives a red-brown ring at the zone of contact of the liquids, and on shaking the tube a green liquid changing to grey-green is obtained. Tannic acid gives a red-brown ring, whilst on mixing the colour is greenish-blue at the bottom, and subsequently brownish-red throughout. Pyrogallol gives a wine-red ring and red-brown liquid. The test is capable of detecting 0.00001% of formaldehyde. Salicylic acid gives a light-rose coloured ring and solution. Catechol gives a violet-red ring and wine-red liquid, and resorcinol a wine-red ring and liquid. β -Naphthol gives a violet-red ring, whilst the liquid becomes brownish-black with green fluorescence, and similar colorations are obtained with benzonaphthol. Phenyl salicylate gives a carmine-red ring and a reddish-yellow liquid, becoming pink on dilution. Phenolphthalein does not form a ring, but gives an orange liquid changing to carmine-red when shaken. On dilution with water a carmine precipitate separates whilst the solution remains colourless.—C. A. M.

Acrolein; Stabilisation of —. III. Preparation of acrolein. C. Mouren and A. Lepape. Comptes rend., 1919, 169, 885—889.

A MIXTURE of potassium bisulphate and potassium sulphate in the proportion of 5:1 is recommended as a dehydrating agent in the preparation of acrolein from glycerol. To such a mixture is added one-quarter of its weight of glycerol, and the whole is gradually heated until the reaction commences, the temperature during the greater part of the operation remaining at 195° C. The vapours first pass through a condenser so regulated that the uncondensed vapours issue from it at about 70° C. These then pass to a second condenser, from which the

acrolein is collected. As the reaction proceeds and the glycerol becomes used up more is added, until the dehydrating agent has decomposed nearly ten times its weight of glycerol. By this method 2311 kilos. of glycerol was dehydrated by 240 kilos. of the dehydrating agent and gave a yield of pure acrolein equivalent to 67.5% of the theoretical yield.—W. G.

Rose oil: German extracted —. F. Elze. Chem.-Zeit., 1919, 43, 747.

OIL of rose obtained in 0.015% yield by extraction with volatile solvents had the following constants: Sp. gr. = 0.9894 at 20° C., $n_{D,20} = +0.6^\circ$, acid value 3.15, ester value 2.9, acetylation value 317.5. Phenylethyl alcohol was the chief constituent, and in addition geraniol, citronellol, nerol, and the aliphatic sesquiterpene alcohol, farnesol, were identified. The stearopten contained several olefinic hydrocarbons with melting points ranging from 20° to 46° C.—G. F. M.

Colloidal copper. Protective colloids. Second Series. Cetraria islandica as protective colloid. IV. A. Guthrie and E. Sauer. Kolloid-Zeits., 1919, 25, 145–153.

COLLOIDAL solutions of copper may be prepared by reducing an ammoniacal solution of copper sulphate with hydrazine hydrate in the presence of a 0.2% solution of the aqueous extract of Iceland moss. The sols are fairly stable, but in 14 days' deposit a reversible gel. Sols which have a clear red colour both by reflected and transmitted light deposit reversible gels, whilst those which are turbid by reflected light deposit irreversible gels. The solid colloid is obtained by adding $\frac{1}{2}$ –1 volume of alcohol, filtering under pressure and drying in a vacuum over sulphuric acid. The limit of reversibility in the solid product is reached with 13.43% of copper. (See also J. Chem. Soc., Jan., 1920.)

—J. F. S.

Nitrobenzoic acids. Bigelow. See III.

Colloidal mercury. Guthrie and Weise. See XI.

PATENTS.

Choline; Manufacture of salts or compounds of — and of its higher homologues. J. Y. Johnson, London. From Verein. Chem. Werke A.-G., Charlottenburg, Germany. Eng. Pat. 8031, 30.3.14.

CHOLINE, or a higher homologue, is combined with suitable acids, other than hydrochloric, hydrobromic, nitric, hydriodic, chloroplatinic, or sulphuric acids. The aqueous solutions of the resulting compounds, whilst having the same effect on the cells as choline solutions when injected into the blood, are more certain in action owing to their greater stability and the consequent non-formation of poisonous decomposition products. Suitable acids are, for instance, boric, salicylic, *o*-, *m*-, or *p*-iodobenzoic, *p*-aminophenylarsonic, 4-amino-3-iodophenylarsonic, and formic acids, and the compounds or salts are produced by dissolving either an equivalent proportion or an excess of the acid in an aqueous or alcoholic solution of choline, or a higher homologue.—G. F. M.

Synthetic drugs [adrenaline]; Manufacture of —. E. C. R. Marks, London. From N. Nagai, Tokyo, Japan. Eng. Pat. 118,298, 13.7.17. (Appl. 10,183/17.)

DIACETYLPROTocatechic aldehyde, produced by the interaction of protocatechic aldehyde and acetyl chloride or acetic anhydride, is condensed with nitromethane by the action of a solution of a weak alkali, such as an alkali carbonate or bicarbonate, pyridine, etc., at normal temperature, on equimolecular proportions of the two substances. The

condensation product, diacetoxyphenylmethanol, $(CH_3COO)_2C_6H_3CH(OH)CH_2NO_2$, which separates in crystalline form, is filtered off, washed free from unchanged protocatechic aldehyde with ether and treated with acetic acid and zinc dust in presence of a 35% formaldehyde solution containing one molecular equivalent of formaldehyde, whereby simultaneous reduction and methylation take place with the production of diacetoxy-phenyl-methylaminoethanol,



The zinc is precipitated from the reaction mixture as sulphide and to the filtered solution the requisite amount of hydrochloric acid is added to effect hydrolysis and combine with the liberated base. On evaporation at a low temperature in a vacuum crystals of dihydroxyphenylmethylaminoethanol hydrochloride (adrenalin hydrochloride) are obtained.—G. F. M.

β -Halogen-ethylaminobenzoic esters; Manufacture of —. Soc. Chim. des Usines du Rhône, Paris. Eng. Pat. 128,553, 30.5.19. (Appl. 13,761/19.) Int. Conv., 20.6.18.

β -HALOGEN-ETHYL-*p*-AMINOBENZOIC esters of the general formula, $NHCH_2CH_2X.C_6H_4.CO_2R$, where X is a halogen, are obtained by treating the corresponding hydroxyethyl compounds with halogenating reagents, such as phosphorus or sulphur halides, thionyl chloride or bromide, etc. For example, ethyl β -hydroxyethyl-*p*-aminobenzoate is treated with an equal weight of phosphorus pentachloride in presence of dry benzene, the reaction is completed by heating on a water-bath, and, after evaporating off the benzene and phosphorus oxychloride, the residual chloro-ester is purified by distillation under reduced pressure. It is a crystalline substance, m.p. 69° C., and boiling at 183° C. at 3 mm. pressure. Alternatively thionyl chloride might be used to produce the same compound with dimethylaniline as a diluent.—G. F. M.

β -Alkylaminoethylaminobenzoic alkyl esters; Manufacture of —. Soc. Chim. des Usines du Rhône, Paris. Eng. Pat. 128,554, 30.5.19. (Appl. 13,762/19.) Int. Conv., 20.6.18.

β -ALKYLAMINOETHYL-*p*-AMINOBENZOIC acid esters are obtained by heating for several hours in a closed vessel at a temperature of about 100° C. a mixture of an alkylamine and a β -halogenethyl-*p*-aminobenzoic ester (see preceding abstract). Thus diethylamine and ethyl β -chloroethyl-*p*-aminobenzoate give ethyl β -diethylaminoethylaminobenzoate, an oily liquid which yields a monohydrochloride when evaporated to dryness with the requisite quantity of dilute hydrochloric acid. On recrystallising from alcohol white needles are obtained, melting at 156° C.—G. F. M.

Substituted benzoic acid esters; Manufacture of new —. Soc. Chim. des Usines du Rhône, Paris. Eng. Pat. 128,912, 7.6.19. (Appl. 14,550/19.) Int. Conv., 26.6.18.

DI- β -HALOGENETHYL-*p*-AMINOBENZOIC acid esters of the general formula, $(XCH_2CH_2)_2N.C_6H_4.CO_2R$ are obtained from the corresponding di- β -hydroxy compounds, by treating them with halogenating agents such as phosphorus or sulphur chlorides, thionyl chloride, etc., using tertiary amines etc. as diluents as described in Eng. Pat. 128,553 (preceding). Ethyl β -dichloroethyl-*p*-aminobenzoate prepared in this way melts at 53° C. and boils at 215° C. under 3 mm. pressure with slight decomposition. These dichloro-esters may be converted into dialkylamino esters of the general formula $(R.N.CH_2CH_2)_2N.C_6H_4.CO_2R'$, where R is a hydrogen atom or an alkyl group, and R' an alkyl group, by treating with an alkylamine as previously described (*loc. cit.*). The dialkylamino esters give

neutral dihydrochlorides which can be crystallised from alcohol.—G. F. M.

Acetone and carbonic acid; Manufacture of —. Soc. Anon. des Acieries et Forges de Firminy, Firminy, France. Eng. Pat. 131,143, 18.2.19. (Appl. 4019/19). Int. Conv., 16.11.18.

ACETIC acid of 90–100% strength is decomposed by the aid of a catalyst of manganese peroxide, either prepared by precipitation, or preferably natural pyrolusite, crushed and screened to pieces, of a diameter of 5–7 mm. Catalysis occurs without appreciable subsidiary reaction between wide limits of temperature, for instance from 350° to 450° C. The catalyst is packed in a cylinder and heated either externally, or preferably by mixing with 1–1½ volumes of crushed coke of similar dimensions and passing an electric current through the mixture by means of two iron or aluminium electrodes. Acetone is recovered from the product of catalysis by means of condensers and finally by scrubbing the uncondensed gas in water sprinkling towers, and the washed carbon dioxide is suitably stored for use. The spent catalyst can be revived by merely heating in a current of air.—G. F. M.

Dental cements. J. V. G. Andresen, Copenhagen. Eng. Pat. 134,757, 9.4.19. (Appl. 9048/19.)

A DENTAL cement is prepared by working up a mixture of 45 parts of zinc oxide and 15 parts of burnt alum into a paste with 22½ parts of water, or, to increase the local anæsthetic property of the cement, with 15 parts of oil of cloves, eugenol, or a concentrated solution of zinc sulphate, zinc chloride, copper sulphate, phosphoric acid, or mixtures of the same, or with the product obtained by heating 15 parts of eugenol with 2 parts of formaldehyde at 120° C.—L. A. C.

Maleic acid; Preparation of —. J. M. Weiss, New York, and C. R. Downs, Cliffside, N.J., Assignors to The Barrett Co. U.S. Pat. 1,318,632, 14.10.19. Appl., 27.5.18.

A MIXTURE of benzene and quinone in the vapour phase is oxidised by treatment under pressure with a gas containing oxygen at a temperature of 300°–700° C. in the presence of vanadium oxide as a catalyst. The maleic acid produced is separated and the residual benzene and quinone are again subjected to the same process.—G. F. M.

Acetylsalicylic acid; Purification of —. H. Hibbert, Mount Vernon, N.Y., Assignor to The Commercial Research Co. U.S. Pat. 1,321,307, 11.11.19. Appl., 8.4.16. Renewed 11.5.18.

CRUDE acetylsalicylic acid is dissolved in a hot dichlorinated hydrocarbon, e.g., ethylene dichloride, or the mixture obtained by chlorinating oil gas. Acetylsalicylic acid crystallises from the solution on cooling.—L. A. C.

Glycerin substitute. [Ester salts of phthalic acid.] O. Rössler, Dresden. Ger. Pat. 313,059, 23.8.17.

ESTER salts of phthalic acid soluble in water are used as glycerin substitutes. One carboxyl group of the phthalic acid is esterified with a monohydric alcohol and the other group is combined with a metallic base, such as sodium, to form a salt soluble in water. The ester salts are used in moderately dilute solution.—J. F. B.

Cholic acid; Manufacture of an acyl derivative of —. J. D. Riedel A.-G., Berlin-Britz. Ger. Pat. 313,413, 1.4.14.

ACETYLSALICYLIC acid chloride reacts with cholic acid in the presence of a compound capable of combining with hydrochloric acid, e.g., pyridine, to form a white crystalline powder, m.pt. 120° C., insoluble in water, and soluble in alcohol. The

product is tasteless and has no action on the digestion, but passes unchanged into the intestines, where it is dissolved and combines the cholagogic action of cholic acid with the antineuralgic properties of salicylic acid.—L. A. C.

Ethylidene esters; Production of —. Chem. Fabr. Griesheim-Elektron, Frankfurt. Ger. Pat. 313,696, 11.11.15.

ORGANIC vinyl esters are treated with organic or inorganic acids so as to couple one molecule of acid to the ester, e.g., equimolecular proportions of the components, with or without catalysts, are heated together under pressure until combination is complete; one vinyl ester or a mixture of several may be used. Vinyl acetate boiled with acetic acid and a little strong sulphuric acid gives ethylidene acetate. Vinyl benzoate and dry hydrogen chloride give chloroethyl benzoate. Vinyl acetate and benzoic acid give ethylidene acetobenzoate $\text{CH}_3\text{CH}(\text{CO}_2\text{CH}_3)(\text{CO}_2\text{C}_6\text{H}_5)$. Vinyl acetate and hydrogen chloride give chloroethyl acetate $\text{CH}_3\text{CO}_2\text{CHClCH}_2$. The products are generally excellent solvents for artificial and natural resins, nitro- and acetyl-celluloses, and can be used for the preparation of lacquers, films, impregnating preparations, etc.—H. J. H.

Organic substances ordinarily insoluble in water; Process of preparing aqueous solutions of —. E. Kolshorn, Munich. Ger. Pat. 313,726, 13.12.16.

AQUEOUS solutions of esters of carbanic acids are used to render certain organic compounds soluble in water without chemical change. Among the compounds which may be dissolved in this way by means of urethane are alcohols (amyl alcohol, benzyl alcohol, geraniol); bases (aniline, phenylhydrazine, quinoline, isoquinoline); alkaloids (quinine, morphine); drugs of various composition (paraldehyde, sulphonal, antipyrine, thymol, phenacetin, camphor); essential oils, and colouring matters. Ethylurethane, and higher and lower esters of carbanic acid, e.g., the methyl, propyl, isoamyl, allyl, and glyceryl esters, behave in the same way. The solutions prepared with such esters are stable and can be sterilised. By evaporating the solutions solid preparations are obtained consisting of a mixture of the solvent and dissolved substance, which on the addition of water are readily dissolved again.—C. A. M.

Organic compounds; Manufacture of heterocyclic —. M. Wiernik, Berlin-Schöneberg, G. and O. I. Grüttner, Charlottenburg. Ger. Pat. 313,876, 4.7.15.

A DIHALOGENPENTANE or a similar hydrocarbon derivative containing two reactive halogen atoms and an arsenic halide or an organo-arsenic halide are treated with sodium or magnesium in the presence of an indifferent solvent. A compound of any other metal or metalloid, with the exception of silicon and mercury, may be used in place of that of arsenic. The products are decomposed by halogens with the formation of the original dihalogenhydrocarbons. Examples: Cyclopentyl-ene-phenyl arsine, a colourless oil, sp. gr. 1.2420, b. pt. (20 mm.) 153°–154° C., is prepared by the action of sodium or magnesium on a mixture of 1.5-dibromopentane and phenyldichloroarsine. Cyclo-tetramethylenephénylphosphine, a colourless oil, sp. gr. 1.0502, b. pt. (16–18 mm.) 131°–134° C., is prepared from phosphorus phenylchloride and the magnesium derivative of 1,4-dibromobutane. A number of other examples are given.—L. A. C.

p-Phenetol-urea; Manufacture of —. J. D. Riedel A.-G., Berlin-Britz. Ger. Pat. 313,965, 22.3.17.

PHENETIDINE hydrochloride is added to an aqueous

solution of an alkali cyanide containing an alkaline oxidising agent such as sodium hypochlorite or sodium peroxide. *p*-Phenol-urea separates immediately in good yield and purity.—L. A. C.

Furfural; Manufacture of —. E. Ricard, Melle, France. U.S. Pat. 1,322,054, 18.11.19. Appl., 22.11.17.

See Eng. Pat. 129,025 of 1917; this J., 1919, 658 A. *Benzaldehyde*. U.S. Pat. 1,321,959. See III.

Anthranilic acid. U.S. Pat. 1,322,052. See III.

Hydrogenation catalysts. Ger. Pat. 313,192. See XII.

Fermentation of cellulose. Eng. Pat. 131,265. See XVIII.

Urine. Eng. Pat. 131,710. See XXIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Sharp-cut selective [light] filters; Experiments in the use of — in spectro-photometric researches. I. *Dependence of gradation on wave-length with gelatino-bromide of silver*. A. Hnatek. Z. wiss. Phot., 1916, 15, 271—288.

SOME of the filters described in a previous paper (see preceding abstract) were used for determining the relation between the wave-length of the light, to which a plate is exposed, and the gradation of the resulting image. Exposures were impressed by means of a tubes-photometer (Eberhard's modification) with revolving plate-holder, which was first carefully calibrated by the aid of a series of uniformly exposed and developed plates of various densities which were found to be free from scattering effect and without selective spectral absorption. In order to obtain similar effective exposures with the same exposure-time for different colour-mixers and different plates, use was made of the same series of neutral densities to modify the intensity of the light in the gradation experiments. This use of an intensity scale permitted the employment of the expression $S = a + bm + cm^2$, in which S is the density of an image produced by a light-intensity I , $m = -2.5 \log. I$ and a , b , and c are constants which can be determined experimentally for each plate. Gradation, G , is then $dS/dm = b + 2cm$; in the case of certain plates, such as Seed plates with a very long straight-line portion of the sensitiveness-curve, $S = a + bm$ and $G = b$, i.e., is independent of the intensity of the light. The wave-lengths of the colour filters were taken as the "effective" wave-lengths, i.e., in each case the wave-length indicated by the mass-centre of the area included between the horizontal axis and the spectrum curve of the particular light-filter and plate; effective wave-lengths were determined for the Elko plate as representing ordinary plates, and for the colour plate as representing orthochromatic plates. A number of tables are given of the results with five brands of ordinary plates and three brands of colour-sensitive plates (not panchromatic). The filters were selected in each case to cover the range of spectral sensitiveness of the plate. The curves obtained by plotting gradation against wave-length show in all cases an S-formation, rising from short wave-lengths up to a maximum, for ordinary plates at about $\lambda = 4500\mu$, falling to a minimum, and then rising again to about 4900μ ; the chromatic plates show more variation both as to the position of the maximum and its height.—B. V. S.

PATENT.

Coloured [photographic] picture; Process and apparatus for production of a —. J. H. Christensen, Sölleröd, Denmark. Ger. Pat. 313,836, 24.7.18. Int. Conv., 13.8.17.

In a colour process in which three colour records

are taken on one double-coated film and one single-coated film, the two films after completion of the partial colour pictures being cemented together, e.g., with glycerin, to form the complete picture, one or both of the coated films is stretched in a special holder in which it remains until the various processes and the cementing are completed. Where two such frames are used registration is secured by an arrangement of pins and sockets, and one (or both) of the frames is provided with a spring edge under the film, in order to assist in obtaining perfect contact of the two films.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Nitrocellulose. Oddo. See V.

XXIII.—ANALYSIS.

Temperature; Methods of determining —. Their application and reliability. K. Scheel. Z. angew. Chem., 1919, 32, 317—349.

A DESCRIPTION of various mercury, alcohol, toluene, and pentane thermometers, thermo-couples, resistance pyrometers, radiation pyrometers, and Seger cones. For standardising thermometers, especially mercury thermometers, the following fixed points are given:—*Melting points*: Mercury, -38.89° ; ice, 0° ; tin, 231.85° ; cadmium, 320.9° ; zinc, 419.4° ; antimony, 630° ; silver, 960.5° ; gold, 1063° ; copper, 1083° ; palladium, 1157° ; platinum, 1761° C. The transition point of crystallised sodium sulphate is 32.38° C. *Boiling points*: Oxygen, -183.0° C. + $0.01258(p-760) - 0.000007(p-760)^2$; carbon dioxide, -78.5° C. + $0.01595(p-760) - 0.000011(p-760)^2$; naphthalene, 217.96° C. + $0.058(p-760)$; benzophenone, 305.9° C. + $0.0663(p-760)$; sulphur, 444.55° C. + $0.0908(p-760) - 0.000047(p-760)^2$. p is the barometric pressure.—W. P. S.

Filtration; Method for rapid [laboratory] —. J. D. van Leeuwen. Chem. Weekblad, 1919, 16, 1424—1425.

THE author has obtained satisfactory results with the method proposed by Noyes (Int. Sugar J., May, 1919), in which, by tearing off a corner from one or two thicknesses of the folded filter paper, an unbroken stream of liquid is obtained in the stem of the funnel, filtration being thereby accelerated.

—W. J. W.

Charcoal sticks; Preparation of — for reduction tests. N. C. Alexandrescu. Bul. Soc. Chim. Romania, 1919, 1, 11—12.

SMALL wood sticks, as used for making matches, are boiled for two minutes with a 25% solution of ammonium phosphate and then dried at a temperature not exceeding 60°C ., and stored in a stoppered bottle. To carry out the ordinary qualitative charcoal test, half of a stick is burnt, and then the test is carried out in the usual manner on the substance previously mixed with sodium carbonate.

—W. G.

Gas analysis apparatus. M. Bürgerhausen. Chem.-Zeit., 1919, 43, 731.

In the compact apparatus described the absorption vessels are so constructed that the gas, on entering the vessel, bubbles through the solution; a three-way tap allows the return of the gas to the measuring burette. "Dead space" in the connecting capillary tubes is reduced to a minimum. The wires of the combustion pipette are fitted through a tubulure at the top of the pipette.—W. P. S.

Electrometric analysis with potassium ferrocyanide. E. Müller. Z. angew. Chem., 1919, 32, 351—352.

THE electrometric titration of metals with

potassium ferrocyanide solution fails in many cases owing to the fact that the precipitates produced are not of constant composition. The method, however, is trustworthy for the determination of lead or zinc; in the case of zinc, the method is more reliable than is the ordinary method of titration, using uranium nitrate as an external indicator, since small quantities of cadmium do not interfere.

—W. P. S.

Iron; Iodometric estimation of — I. M. Kolthoff. Pharm. Weekblad, 1919, 56, 1565—1568.

THE estimation of ferrous iron by means of iodine and a pyrophosphate gives low results owing to oxidation of the iodine by dissolved oxygen. Removal of the oxygen by means of sulphuric acid and sodium bicarbonate reduces the error. A more convenient method consists in adding potassium bromate and phosphoric acid to the acidified iron solution, allowing to stand, and then adding potassium iodide and a few drops of molybdate solution, the iodine being finally titrated back with thiosulphate. (See also this J., 1919, 444 A.)

—W. J. W.

Iron, aluminium, chromium, glucinum, titanium, and zirconium; Separation of — by means of sodium carbonate. P. Wenger and J. Wuhrmann. Ann. Chim. Analyt., 1919, 1, 337—339.

WUNDER and Wenger have shown (this J., 1912, 664) that fusion with sodium carbonate affords a means of separating iron and glucinum oxides from aluminium and chromium, the two latter forming compounds which are soluble in water, and the authors now extend the method to the analysis of similar mixtures also containing titanium or zirconium oxides. Titanium oxide, however, interferes with the separation of iron from zirconium, and the latter renders impossible the separation of iron and titanium oxides. In the absence of titanium, iron and zirconium oxides, obtained as an insoluble precipitate after fusion as described with sodium carbonate may be separated from each other by taking advantage of the insolubility of zirconium oxide in hydrochloric acid (1:1). If zirconium is absent, iron and titanium oxides, even in the presence of glucinum oxide, may be separated by one of the known methods.—W. P. S.

Zirconium; Estimation of — by the phosphate method. G. E. F. Lundell and H. B. Knowles. J. Amer. Chem. Soc., 1919, 41, 1801—1808.

Zirconium can be quantitatively precipitated as secondary zirconium phosphate in cold or tepid solutions containing 2—20% by weight of sulphuric acid, provided that a 10—100-fold excess of the precipitant, di-ammonium phosphate, is used. Hydrolysis, which occurs when the phosphate precipitate is washed with water, can be almost entirely avoided by the use of a cold 5% ammonium nitrate solution for washing. Zirconium pyrophosphate, for which the factor (ZrO_2) is 0.4632, is obtained on ignition of secondary zirconium phosphate which has been washed with ammonium nitrate solution. No definite composition can be assigned to the compound resulting when secondary zirconium phosphate, which has been washed with water, is ignited. Zirconium can be quantitatively separated as phosphate in a 20% sulphuric acid solution from iron, aluminium, chromium, cerium, and thorium. The separation from titanium can also be effected provided that hydrogen peroxide is present.

—J. F. S.

Complex internal salts in quantitative analysis. I. I. Bellucci and A. Chiucini. Gazz. Chim. Ital., 1919, 49, ii., 187—216.

THE work published on the analytical applications

of α -nitroso- β -naphthol and cupferron (nitroso-phenylhydroxylamine-ammonium) is summarised and criticised, and the cases indicated in which these reagents may be used for the separation and estimation of metals. For the separation of copper neither of the reagents offers any real advantages over known methods, except in the case of the separation of copper from arsenic, and especially from antimony, by means of nitroso- β -naphthol. Cupferron is preferable to nitrosonaphthol for the separation of iron, the permissible limit of free mineral acid in the solution being higher and the precipitate less voluminous. Nitrosonaphthol is of value chiefly as a precipitant for cobalt, and in lesser degree for palladium. Cupferron is a valuable precipitant for iron and also for titanium, zirconium, and vanadium. (See J. Chem. Soc., Jan., 1920.)—T. H. P.

Molybdenum and tungsten; Colour reactions of — II. G. A. Barbjeri. Atti R. Accad. Lincei, 1919, 28, i., 390—392. (See this J., 1919, 881 A.)

IF the acetic acid of Braun's reagent is replaced by hydrochloric or sulphuric acid (see Péchard, Comptes rendus, 1894, 118, 804), the compound $NH_4SCN.(NH_4)_2O.4MoO_3.5H_2O$, is always formed, no matter whether ammonium permolybdate or the tri- or tetra- or normal molybdate is used with the ammonium thiocyanate. From potassium trimolybdate and thiocyanate in presence of acetic acid, the corresponding potassium compound, $KSCN.K_2O.4MoO_3.5H_2O$ is formed. (See also J. Chem. Soc., Jan., 1920.)—T. H. P.

Sodium thiosulphate; Titration of solutions of — I. M. Kolthoff. Pharm. Weekblad, 1919, 56, 644—657.

THE purification and application of various substances for the titration of thiosulphate solutions are described, and their relative merits discussed. A method for the detection and estimation of small quantities of free chromic acid or potassium chromate in potassium bichromate is based on the fact that the conductivity of a bichromate solution containing chromic acid decreases slightly on the addition of standard alkali until the free acid has been neutralised. After this point the conductivity increases rapidly on the further addition of alkali. A similar discontinuity in the curve is obtained when acid is added to a bichromate solution containing chromate. Pure bichromate for analytical purposes should be fused in an electric furnace before use. All the other substances examined—iodine, crystallised oxalic acid, cyanogen iodide, potassium iodate and bromate are easily purified and are equally suitable for the estimation of thiosulphate, the error in the titration being in each case less than 0.1%. The greatest error, 0.07%, was observed in titrating with bichromate.

—W. S. M.

Cerium; Detection of — F. Feigl. Oesterr. Chem.-Zeit., 1919, 22, 124—126. Chem. Zentr., 1919, 90, IV, 592.

CERIUM oxide, moistened with an acetic acid solution of benzidine gives a bright blue coloration. Other cerous and ceric compounds give the reaction, with the exception of cerous fluoride, cerous carbonate, and cerium bisulphate. The most sensitive reaction is obtained by rendering the solution alkaline, boiling, filtering a portion, and sprinkling the filter with the benzidine solution; 0.02 mgrm. of cerium in a litre can be recognised in this manner. The other metals of the ammonium sulphide group of the rare earths, except thallium, do not give the reaction, which depends upon oxidation by means of an oxygen-carrier, and all other oxidising agents, especially manganese, cobalt, thallium, and chromium must be absent. Feebly acid iron solu-

tions also give the reaction; alkaline solutions do not. Since ferric hydroxide carries down considerable amounts of cerium, the latter is best precipitated as fluoride in the presence of iron and converted into hydroxide.—J. H. J.

Uranium; Colorimetric determination of small quantities of —. Müller. Chem.-Zeit., 1919, 43, 739—740.

The red coloration obtained when a dilute (e.g., 0.02%) uranium salt solution is treated with sodium salicylate solution may be utilised for the colorimetric determination of the metal, the coloration being compared with that given by a known amount of uranium under similar conditions. Free mineral acids and acetic acid must not be present; if the solution contains free mineral acid, sodium acetate should be added and the acetic acid then expelled by evaporation. Further, iron salts, alcohol, and acetone interfere with the reaction, but neutral alkali salts appear to be without effect.—W. P. S.

Hydrocyanic acid; Detection of — in a case of poisoning. Its post-mortem transformation into thiocyanic acid. L. Chelle. Comptes rend., 1919, 169, 852—854. (See also this J., 1919, 899 A, 900 A.)

In the case of a dog poisoned by potassium cyanide, hydrocyanic acid as such but no thiocyanic acid was found in the brain, lungs, and blood two hours after death. Eight days after death thiocyanic acid but no hydrocyanic acid was found. Further, in the case of the brains and lungs the amount of thiocyanic acid found was far more than equivalent to the amount of hydrocyanic acid found in these organs two hours after death. To ensure estimation of all the toxic principle it is desirable to wait for a few days after death until putrefaction and consequent cytotoxicity has set in before performing the analysis.—W. G.

Acetylene, ethylene, and benzene. Treadwell and Tauber. See II A.

Wool and silk. Waentig. See V.

Mixed pulp in paper. See V.

Ferrocyanides. Kelley and Bohn. See VII.

Lead oxides. Andersen. See VII.

Bromine. Baughman and Skinner. See VII.

Molybdenum in iron and steel. Malowan. See X

Acetyl value. Grün. See XII.

Lead chromate. Gröger. See XIII.

Ultramarine blue. Kalshoven. See XVII.

Yeasts. Vautier. See XVIII.

Ethyl and methyl alcohols. Pannwitz. See XVIII.

Milk analysis. Langkammerer. See XIX A.

Added water in milk. Goldan, jun. See XIX A.

Sediment in Gerber's butyrometer. Reiss and Dieselhorst. See XIX A.

Butter fat. Vandam. See XIX A.

Tyrosine. Weiss. See XX.

Arsenic compounds. Ganassini. See XX.

Formaldehyde. Rossi. See XX.

PATENTS.

Pyrometer or temperature alarm instrument. J. F. J. Malone, Newcastle-on-Tyne. Eng. Pat. 134,498, 27.5.19. (Appl. 13,357/19.)

The portion of the pyrometer to be immersed in the medium of which the temperature is required consists of members composed of two materials having different coefficients of linear expansion; and the relative changes in length of these members is transmitted to an indicating device by connecting and supporting rods made of the same materials

and having the same cross-sectional areas, so that the indications are not affected by thermal changes in the rods. In one form of the apparatus one of the working members is a tube (steel) surrounding the other member (invar), and the free end of the latter is secured to a sleeve adapted to slide within the tube. One end of the tube is attached to a fixed coupling, to which one set of transmitting rods is also secured, the other set of transmitting rods being secured to the sliding sleeve. The apparatus may be provided with an alarm device.

—W. E. F. P.

Still head. S. F. Dufton, Leeds. Eng. Pat. 134,629, 11.11.18. (Appl. 18,415/18.)

In a still head, a spiral passage is arranged within an annulus, the width of the latter being graduated from top to bottom so that the passage is only just large enough at any point to allow the vapour and condensed liquid to pass each other freely at the rate of distillation desired. The apparatus is constructed by winding a wire round a core, or by making a similar arrangement by casting or turning, and fitting the core inside a tube. A number of units may be combined to form a multiple still head; and the loss of heat from a single or multiple head may be regulated by surrounding the apparatus with a vacuum vessel. (See also this J., 1919, 45 T.)—W. E. F. P.

Urine; Medical analysis of — by the combined use of tungstic acid and other reagents. H. C. Ross, London. Eng. Pat. 134,710, 7.11.18. (Appl. 2267/19.)

When 0.2 c.c. of a reagent composed of an aqueous solution of colloidal tungstic acid 12%, acetic or tartaric acid 10%, and sodium potassium tartrate 2.5%, is added to 2 c.c. of urine, the presence of albumin will cause a precipitate. If the tungstic acid is impure, excess of urates also produces a precipitate, but this re-dissolves on boiling. In the latter case a diabetic condition is indicated, and the hot solution is further tested for reducing sugars by adding a tablet containing copper sulphate 0.02 gm., sodium carbonate 0.01 gm., and calcium oxide 0.01 gm. If desired both reagents may be prepared in tablet form.—G. F. M.

Gas-testing machine. L. A. Stenger, Denver, Colo. U.S. Pat. 1,320,584, 4.11.19. Appl., 14.10.18.

In a gas analyser, a gas chamber, which communicates with a solution tank and a pressure gauge, is charged with a predetermined volume of gas and closed to the atmosphere. The solution, in the form of spray, is then caused to pass through the gas chamber and back to the tank.—W. E. F. P.

[Combustible] gases; Method and apparatus for testing —. A. B. Lamb and A. T. Larson, U.S. Army. U.S. Pat. (A) 1,321,063 and (N) 1,321,064, 4.11.19. Appl., 17.2.19.

In apparatus for determining combustible gases at low concentrations, the gaseous mixture is caused to pass (A) over catalytic material in the vicinity of a thermo-junction, or (N) around one of a pair of electrically heated wires the change in resistance of which is measured.—W. E. F. P.

Inflammable gases in gas mixtures; Estimation of —. R. Naumann, Berlin-Schlachtensee. Ger. Pat. 313,858, 18.1.18.

The inflammable gas is estimated by burning at a catalytic surface, the rise in temperature of the latter being noted. The speed of flow of the gas mixture can be varied, and that speed is used which gives the greatest rise in temperature.—W. P.

Absorbent. A. B. Lamb, U.S. Army, and C. R. Hoover, Middletown, Conn. U.S. Pat. 1,321,061, 4.11.19. Appl., 28.12.18.

As oxidising, absorbent material is prepared by

treating iodic anhydride with sulphuric anhydride in the presence of a small amount of water.
—W. E. F. P.

Carbon monoxide in hydrogen. Eng. Pat. 134,243.
See VII.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Abogado. Process and apparatus for heating liquids. 30,834 and 30,835. Dec. 9.
Arraghi. Filters. 30,675. Dec. 8.
Beanes. Machines for drying and/or roasting granular substances. 31,945. Dec. 19.
Bourdet. Apparatus for treating pulverulent materials. 31,482. Dec. 16.
Bury, and Skinningrove Iron Co. 31,066. See XI.
Harris. Settling vats. 31,299. Dec. 13.
Hort. Method of evaporation. 30,821. Dec. 9.
Imbery. Furnaces. 31,324. Dec. 15.
Norske Aktieselskab for Elektrokemisk Industri. Manufacture of porous material. 31,371. Dec. 15. (Norway, 16.12.18.)
Rostados. Method of drying and dry storage of merchandise, foodstuffs, etc. 31,987. Dec. 20.
Soc. Anon. des Etabl. A. Olier. Filter presses. 31,298. Dec. 19. (Fr., 31.1.19.)
Soc. l'Air Liquide. Separation of constituents of gaseous mixtures liquefiable at very different temperatures. 31,696. Dec. 17. (Fr., 4.2.14.)

COMPLETE SPECIFICATIONS ACCEPTED.

10,453, 10,458, and 10,459 (1918). Elektro-Osmose A.-G. See XI.
20,253 (1918). Barron and Barron. Impact grinders. (135,955.) Dec. 17.
20,739 (1918). Fuller and Bedford. Furnaces. (136,270.) Dec. 24.
21,057 (1918). Webster. Filter presses. (135,976.) Dec. 17.
21,756 (1918). Acton. Apparatus for separating solid particles from liquids. (135,992.) Dec. 17.
21,759 (1918). Alexander (United Filters Corporation). Continuous vacuum filters. (136,309.) Dec. 24.
2097 (1919). South Metropolitan Gas Co., and Chandler. Gas furnaces. (136,356.) Dec. 24.
13,336 (1919). Mond (International Precipitation Co.). Separation of suspended material from gases. (136,464.) Dec. 24.
19,318 (1919). Chenard. Apparatus for fractional distillation. (130,992.) Dec. 17.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Brown and Hoover. Working oil shales. 30,930. Dec. 10.
Dale. Coal etc. briquettes, and manufacture thereof. 31,820. Dec. 18.
Davidson. Preparation of peat fuel for producer gas. 31,074. Dec. 11.
Davidson. Preparation of peat fuel for general purposes. 31,075. Dec. 11.
Duckham, and Woodall, Duckham, and Jones. Introduction of steam into vertical retorts or ovens. 31,799. Dec. 18.

France. 31,821. See X.
Goold (Universal Oil Products Co.). Process of converting hydrocarbons. 31,138. Dec. 12.
Knibbs. Treatment of hydrocarbon oil. 32,019. Dec. 20.

McEwen, and Underfeed Stoker Co. Carbonisation or destructive distillation. 31,073. Dec. 11.
Marshall. Manufacture of incandescent gas mantles. 31,853. Dec. 19.

Rushton. Composite fuel, and manufacture of same. 31,196. Dec. 12.
Soc. Indus. de Prod. Chimiques. Recovery of ammonia from coke-oven etc. gases. 31,688 and 31,689. Dec. 17. (Fr., 27.5.18.)

COMPLETE SPECIFICATIONS ACCEPTED.

10,972 (1917). Wellington. Production of gas. (136,186.) Dec. 24.
3715 (1918). Lynn. Producer-gas plant. (136,189.) Dec. 24.
15,809 (1918). Rowntree and Co., and Fryer. See III.
18,392 (1918). Dellwik, and Techno-Chemical Laboratories, Ltd. Combustion of fuel. (136,212.) Dec. 24.
20,002 (1918). Umsted. Purifying gas and obtaining by-products. (135,931.) Dec. 17.
20,392 (1918). Climie and Lees. Gas-producing plant. (135,959.) Dec. 17.
901 (1919). Bamber and Abrahams. Gas-producers. (136,333.) Dec. 24.
2097 (1919). South Metropolitan Gas Co., and Chandler. See I.
5002 (1919). Cummins. Vertical gas retort settings. (136,398.) Dec. 24.
9903 (1919). Berglund. See X.
11,177 (1919). Marks (U.S. Industrial Alcohol Co.). Liquid fuel. (136,452.) Dec. 24.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

Badier and others. 31,663 and 31,664. See XX.
Boot's Pure Drug Co., and Marshall. 31,660. See XX.
Green and others. 31,978. See XX.

COMPLETE SPECIFICATIONS ACCEPTED.

15,809 (1918). Rowntree and Co., and Fryer. Combustion of pitch. (135,886.) Dec. 17.
10,864 (1919). Marks (Soc. Franco-Belge de Fours à Coke). Continuous distillation and fractionation of crude benzols etc. (136,450.) Dec. 24.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

Imray (Soc. Chem. Industry in Basle). Manufacture of monoazo dyestuffs. 30,839. Dec. 9.
Ward. Scarlet dye. 31,234. Dec. 13.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Fort, Lumsden, Mackenzie, and Robinson. Treatment and purification of yarns and fabrics composed of vegetable fibres. 31,292. Dec. 13.
Frood. Manufacture of fibrous fabrics or compositions for frictional and wearing purposes. 30,760. Dec. 9.
Great Northern Paper Co. Manufacture of paper. 31,790. Dec. 18. (U.S., 14.3.18.)
McCredie. Retting flax, hemp, ramie, etc. 31,289. Dec. 13.
Müller. Manufacture of cellulose compounds. 31,377. Dec. 15.
Soc. Gillet et Fils. Treatment of vegetable fibres. 31,188—31,192. Dec. 12. (Ger., 13.12.18, and 12.6.23.7, and 2.9.19.)

VI.—BLEACHING; DYEING; PRINTING; FINISHING.**APPLICATIONS.**

Chambers. Process of metallising lace etc. 31,039. Dec. 11.
McKittrick and Pethick. Printing or dyeing patterns or ornamenting fabrics. 31,052. Dec. 11.
Maupai. Process of dyeing raw silk. 30,951. Dec. 10.

COMPLETE SPECIFICATION ACCEPTED.

21,134 (1919). Calico Printers' Assoc., Turner, and Fournaux. Ageing-machines. (136,507.) Dec. 24.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.**APPLICATIONS.**

Douglas. Apparatus for manufacture of sulphate of ammonia. 31,970. Dec. 20.
Field, and Metals Extraction Corporation. Purification of zinc solutions. 31,936. Dec. 19.
Harger. Manufacture of hydrogen. 31,849. Dec. 19.
Marks (West Coast Kalsomine Co.). Manufacture of iron compounds. 30,832. Dec. 9.
Newall. Means for calcining magnesite etc. 30,816. Dec. 9.
Pedersen. Extraction of sulphur from sulphide ores. 30,977. Dec. 10.
Pestalozza. Apparatus for direct production of hypochlorites. 31,908 and 31,909. Dec. 19.
Soc. Indus. de Produits Chimiques. 31,688 and 31,689. *See II.*
Welter. Production of soda or mixtures thereof with other substances. 31,816. Dec. 18. (Ger., 23,318.)

COMPLETE SPECIFICATIONS ACCEPTED.

3792 (1918). Partington, Jones, and Brownson. Production of ammonium nitrate. (136,190.) Dec. 24.
17,364 (1918). Haslup. Fixation of atmospheric nitrogen and production of ammonia. (135,889.) Dec. 17.
21,659 (1918). Norske Aktieselskab for Elektrokemisk Industri. Production of alumina poor in iron. (125,578.) Dec. 24.
1442 (1919). Parsons and Jones. Purification of ammonia. (136,342.) Dec. 24.
7872 (1919). General Chemical Co. Apparatus for the synthetic production of ammonia. (124,762.) Dec. 17.
14,950 (1919). Bergve. Production of alkali sulphides. (129,629.) Dec. 24.

VIII.—GLASS; CERAMICS.**APPLICATION.**

Laycock and Laycock. Annealing lehrs or kilns for glass etc. 31,877. Dec. 19.

COMPLETE SPECIFICATION ACCEPTED.

19,688 (1919). Marlow. Ovens or kilns for the manufacture of tiles, pottery, or other ware. (136,127.) Dec. 17.

IX.—BUILDING MATERIALS.**APPLICATIONS.**

Armstrong, Lee, and Pearson. Production of bricks, tiles, etc. 30,758. Dec. 9.
Bagguley. Manufacture of bricks etc. 30,894. Dec. 10.
Laurie, Sutcliffe, and Sutcliffe, Speakman and Co. Mixtures for blocks or slabs for building etc. 30,805. Dec. 9.

COMPLETE SPECIFICATIONS ACCEPTED.

2035 (1919). Scarlett. Cement. (136,355.) Dec. 24.

9678 (1919). Dale. Manufacture of concrete. (136,075.) Dec. 17.
15,154 (1919). Scott, Scott, and Scott. Cement. (136,478.) Dec. 21.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.**APPLICATIONS.**

Adams and Melrose. Solder for aluminium and its alloys. 30,788. Dec. 9.
Annable, and Nickel Concentration, Ltd. Extraction of nickel from its ores. 31,699. Dec. 17.
Ballantine. Production of alloys. 31,761. Dec. 18.
British and Foreign Chemical Producers (Rheinische Kampferfabrik). Pickling iron or steel. 30,960. Dec. 10.
Bury, and Skinningrove Iron Co. 31,066. *See XI.*
Collins. Purification of tin. 30,950. Dec. 10.
Collins. Winning tin. 30,951. Dec. 10.
Elmore. Treatment of argentiferous lead-zinc ores. 31,791. Dec. 18.
Elmore. Treatment of argentiferous sulphide ores. 31,797. Dec. 18.
France. Mineral washing processes. 31,821. Dec. 18.
Hadfield. Manufacture of alloy steel. 30,724. Dec. 8.
Hadfield. Manufacture of steel. 30,725. Dec. 8.
Johanson. Treating weakly magnetic iron ore containing sulphides. 31,814. Dec. 18.
Pedersen. 30,977. *See VII.*

COMPLETE SPECIFICATIONS ACCEPTED.

18,529 (1918). Harvey. Metal-melting furnace. (136,213.) Dec. 24.
20,541 (1918). Tucker, Edser, and Minerals Separation, Ltd. Concentration of ores. (136,255.) Dec. 24.
20,550 (1918). Haywood. Copper-aluminium alloy. (135,963.) Dec. 17.
20,761 (1918). Amalgamated Zinc Co., and Ganelin. Recovery of lead and silver from sulphide ores etc. (135,968.) Dec. 17.
9903 (1919). Berglund. Apparatus for sintering concentrates, distilling shale, etc. (136,076.) Dec. 17.
10,755 (1919). Lenoir. Manufacture of manganese and its alloys in the electric furnace. (126,303.) Dec. 17.

XI.—ELECTRO-CHEMISTRY.**APPLICATIONS.**

British Thomson-Houston Co., Hastings, and Laycock. Electric furnaces. 31,899. Dec. 19.
Bury, and Skinningrove Iron Co. Apparatus for electrostatic deposition of particles from gases. 31,066. Dec. 11.
Gush. Apparatus for purifying liquids by electrolytic treatment. 31,642. Dec. 17.

COMPLETE SPECIFICATIONS ACCEPTED.

10,453, 10,458, 10,459 (1918). Elektro-Osmose A.-G. Apparatus for electro-osmotically removing water etc. from materials. (135,815, 135,819, 135,820.) Dec. 24.
19,671 (1918). Bibby. Electric arc shaft furnaces. (135,905.) Dec. 17.
20,988 (1918). Joel. Electric accumulators. (136,282.) Dec. 21.
3024 (1919). Cooke. Storage batteries. (136,026.) Dec. 17.
3555 (1919). Moore. Electric arc furnaces. (136,034.) Dec. 17.
10,755 (1919). Lenoir. *See X.*
17,988 (1919). Winne. Electric furnace control apparatus. (136,500.) Dec. 21.

XII.—FATS; OILS; WAXES.

APPLICATION.

Marlborough. Liquid detergents etc. 30,653. Dec. 8.

COMPLETE SPECIFICATION ACCEPTED.

20,079 (1918). Blichfeldt. Apparatus for use in the manufacture of margarine or the like. (135,939.) Dec. 17.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Ioco Proofing Co., Vickers, Ltd., and Nuttall. Condensation of phenolic bodies with aldehyde compounds. 31,391—31,393. Dec. 15.

Pettigrew and Scudder. Phenol resin condensates. 31,550. Dec. 16.

COMPLETE SPECIFICATIONS ACCEPTED.

20,208 (1918). American Zinc Lead and Smelting Co. Preparing pigment zinc oxide. (122,170.) Dec. 17.

21,352 (1918). Robinson, and Damard Lacquer Co. Phenol formaldehyde condensation products. (136,298.) Dec. 24.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Bashford. Rubber compound. 30,814. Dec. 9.

Conner and Lyttlemore. Process for vulcanising indiarubber. 30,739. Dec. 9.

Willard. Process for devulcanising vulcanised rubber. 30,870. Dec. 9.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATION.

Reuhig and Seemann. Process of tanning skins. 31,065. Dec. 11. (Ger., 2,10,18.)

COMPLETE SPECIFICATIONS ACCEPTED.

7720 (1918). Clark (Chem. Fabr. Worms A.-G.). Tanning animal hides. (136,193.) Dec. 24.

19,902 (1918). Drury. Leather compositions. (135,921.) Dec. 17.

5124 (1919). Dufour and Dufour. Tanning and liming of hides and skins. (125,362.) Dec. 24.

XVI.—SOILS; FERTILISERS.

APPLICATION.

Field, Grinberg, and Grinnell. Treatment of organic matter for fertiliser purposes. 31,584. Dec. 16. (U.S., 27,4,17.)

XVII.—SUGARS; STARCHES; GUMS.

COMPLETE SPECIFICATION ACCEPTED.

20,836 (1918). Martin. Manufacture of lactose or milk sugar. (135,969.) Dec. 17.

XVIII.—FERMENTATION INDUSTRIES.

COMPLETE SPECIFICATION ACCEPTED.

10,315 (1919). Franks. Preserving non-alcoholic beer and other fermentable liquids. (136,448.) Dec. 24.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Ballochmyie Creamery Co., and McCrone. Food product. 31,741. Dec. 18.

Broek, and Broek and Sen. Baking-powder. 31,759. Dec. 18.

Bruff. Preserving peas, beans, etc. 32,030. Dec. 20.

Collins, Hawkins, Sisson, and Travis. Treatment of sewage, trade waste, etc. 31,548. Dec. 18.

Gush. 31,642. See XI.

Hawker. Insecticide. 31,354. Dec. 15.

Hort. Manufacture of protein food products. 30,820. Dec. 9.

Rostados. 31,987. See I.

Spence and Wyllie. Treatment of milk. 30,773. Dec. 9.

Townsend. Milk foods. 30,978 and 30,979. Dec. 10.

COMPLETE SPECIFICATIONS ACCEPTED.

14,970 (1918). Kestner. Food product containing cacao. (136,205.) Dec. 24.

20,079 (1918). Blichfeldt. See XII.

20,197 (1918). Cronstoe and Warnecke. Food-stuffs from marine plants. (121,295.) Dec. 17.

20,216 (1918). Wilton, Hepworth, and Fuessly. Softening, treating, and filtering water. (136,225.) Dec. 24.

20,363 (1918). Gee. Animal foodstuffs. (136,237.) Dec. 24.

20,671 (1918) and 3955 (1919). Candy. Purification of water. (136,266.) Dec. 24.

6962 (1919). Kawanishi. Method and means of disinfecting. (136,419.) Dec. 24.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Badier, Turner, and British Dyestuffs Corporation. Introduction of alkyl or benzyl radicals into amines. 31,663. Dec. 17.

Badier, Turner, and British Dyestuffs Corporation. Manufacture of diphenylamine derivatives. 31,664. Dec. 17.

Boake, Roberts, and Co., and Durrans. Manufacture of esters. 31,888. Dec. 19.

Boot's Pure Drug Co., and Marshall. Manufacture of primary and secondary amines. 31,660. Dec. 17.

Chem. Fabr. vorm. Sandez. Isolation of principal alkaloid of ergot in pure crystallised state. 32,012. Dec. 20. (Switz., 10,3,19.)

Dreyfus. Manufacture of acetic anhydride. 31,294. Dec. 13. (Fr., 30,6,14.)

Fabr. de Prod. Chim de Thann et de Mulhouse. Manufacture of borneol. 30,706. Dec. 8. (Fr., 12,6,19.)

Green, and British Dyestuffs Corporation. Manufacture of phthalic acid and anhydride. 31,978. Dec. 20.

COMPLETE SPECIFICATIONS ACCEPTED.

5589 (1917). Wieland. Manufacture of addition products of desoxycholic acid. (105,769.) Dec. 17.

484 (1918). Jöncs (Chem. Fabr. J. A. Wülfing). Preparation of double salts of caffeine with alkali salts of acetylsalicylic acid. (136,187.) Dec. 24.

21,402 (1918). Matthews and Strange. Separation of *o*- and *p*-toluene sulphochlorides. (135,987.) Dec. 17.

3695 (1919). Thomson. Preparation of detoxicated vaccine. (136,036.) Dec. 17.

16,438 (1919). Marks (Union Carbide Co.). Preparation of ethylene dichloride. (136,489.) Dec. 24.

19,573 (1919). Elektrizitätswerk Lonza. Manufacture of alcohol. (134,521.) Dec. 17.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Greene. Photographic solutions. 31,360. Dec. 15.

Greene. Colour photography. 31,997. Dec. 20.

XXII.—EXPLOSIVES; MATCHES.

APPLICATION.

Dynamit A.-G. vorm. A. Nobel and Co. Manufacture of gelatinous nitroglycerin explosives proof against fire-damp. 31,201. Dec. 12. (Ger., 22,3,19.)

I.—GENERAL; PLANT; MACHINERY.

Oiliness and lubrication. R. M. Deeley. Phys. Soc., 28.11.19. Engineering, 1919, 108, 788.

IN recent years the property of "oiliness" has become associated with the presence of unsaturated compounds, which constitute 20–40 of most lubricating oils. Hardy (this J., 1919, 77) concludes "that lubrication depends wholly on the chemical constitution of a fluid," and that "the true lubricant is always a fluid which is absorbed by the solid face." The static coefficients of various oils between metals were measured by allowing three pegs, each 5/32 in. diam., to rest on a flat metal plate which could be rotated. The pegs were attached to a weighted upper disc, which actuated a spindle, to which a spiral spring and recording pointer were attached. The movable disc on which the pegs rested lay in a circular dish which could be filled with oil. To ensure surfaces as clean as possible the rubbing metals were ground in water with carborundum flour, polished, and warmed to remove all traces of moisture. When no lubricant is used the static coefficient increases as the surfaces continue to rub against each other. Such clean surfaces are very sensitive to contaminating influences, e.g. moisture. The static coefficient of friction, and efficiency, obtained for a number of oils tested are shown below, the efficiency being calculated from the formula, $100 - (\text{static coefficient} \times 100) = E$;—

Surface flooded with oil.

Oil. Mineral=M. Animal=A. Vegetable=V.		Mild steel on cast iron.		Mild steel on lead-bronze.*	
		Static coeff.	Efficiency.	Static coeff.	Efficiency.
H.B. clock	M.	0.271	72.9	0.275	72.5
Castor	V.	0.183	84.7	0.159	83.1
Trotter	A.	0.123	87.7	0.152	84.8
Rape	V.	0.119	88.1	0.136	85.8

* The lead-bronze had the composition Cu 53.88, Sn 5.71, Pb 4.0, Zn 4.19, Fe 0.1, Ni 0.12%.

It is interesting to note that the oils which have the smallest coefficient of friction are those which have proved the best lubricants in practice. It would appear that the unsaturated molecules of the lubricant enter into a firm physico-chemical union with the metallic surfaces, forming a friction surface which is a compound of oil and metal. This surface would also appear to be of more than molecular thickness. Thin films of this kind cannot be removed by merely wiping, but must be ground off under water, or a thin layer of metal must be cut off.—C. A. K.

Liquid air; Vessels for the transport and handling of —. E. Moser. Z. angew. Chem., 1919, 32, 365–367.

THE extensive use of liquid air as an explosive in the mining industry in Germany in the latter years of the war led to a great demand for Dewar vacuum vessels. Glass containers were found unsuitable on account of their short life, whilst iron or copper vessels, although more durable, were much less efficient, especially in the smaller sizes. Double-walled vessels of hard glazed, or even unglazed, porcelain, silvered internally, were introduced, and the difficulties of obtaining an airtight seal after evacuation were eventually overcome. Such vessels possessed to a much lesser degree the disadvantages of glass or metal containers and showed an efficiency in the case of the larger transport vessels of 15–25 litres capacity, of 0.6–1.0%

evaporation per hour, whilst the smaller operating containers gave 1.2–2.1 evaporation per hour, compared with double that amount in the case of metal containers.—G. F. M.

PATENTS.

Refrigerating; Process of and apparatus for —. Apeldoornsche Maschinfabriek en Metaalgietterij voorheen Loog Landaal, Apeldoorn, Holland. Eng. Pat. 120,923, 20.11.18. (Appl. 19,084/18.) Int. Conv., 20.11.17.

THE refrigerating liquid is passed through a common pressure-regulating valve, and is evaporated part at high and the remainder at low pressure in separate evaporators with a liquid-vapour separator between the two. The level of the liquid in the separator is controlled by a valve mainly regulated by a float, but the valve opens towards the low-pressure evaporator, so that if the difference between the high and low pressures becomes too great the excess pressure acting on the valve will force it open independently of the float. A single compressor may be used to re-compress the two vapours by admitting the low-pressure vapour during nearly the whole of the outstroke of the piston (e.g. through an automatic inlet valve in the head of the hollow piston), while the high-pressure vapour is admitted through ports in the cylinder wall, which are uncovered by the piston near the outer dead centre only.—B. M. V.

Freezing media. A. J. Instone, London. Eng. Pat. 135,567, 22.11.18. (Appl. 19,244/18.)

A FREEZING mixture for maintaining cold storage chambers at a constant temperature is prepared ready for use by mixing sodium or calcium chloride with a suitable quantity of water, according to the temperature it is desired to maintain; the mixture is cooled nearly to congealing point and sprayed on to a still cooler revolving drum, whence the "ice" is scraped off as it is formed. The "ice" may be compressed into blocks if desired.—B. M. V.

Liquid-cooling apparatus. J. Elger, Chicago, Ill. U.S. Pat. 1,323,587, 2.12.19. Appl., 24.1.18.

THE liquid to be cooled flows from an inlet at the top of a casing down through a perforated plate, around a nest of air tubes, arranged transversely to the current of liquid, and to the outlet at the bottom of the casing. The inlet ends of the air tubes are enclosed in a chamber which has an opening for the inlet of cooling air, also one communicating with the top of the liquid compartment and serving as an expansion vent.—B. M. V.

Cooler for gases and vapours. E. Bergfried, Charlottenburg. Ger. Pat. 313,958, 27.8.16.

THE cooler consists of an outer cylindrical vessel surrounding a cylindrical collecting vessel which extends upwards beyond the top of the outer vessel. Mixed gases and vapours etc. are led into the projecting upper part of the inner vessel, thus allowing condensed liquid to run down into the lower portion, whilst a connecting pipe permits the passage of uncondensed vapours and gases to the annular cooling space surrounding the inner vessel. Uncondensed gases finally pass away by an exit arranged near the bottom of the outer vessel. The upper part of the inner vessel may be provided with a separator whereby heavier liquids such as tar may be collected in the inner vessel, whilst lighter liquids, such as ammonia liquor, are run off by a suitable pipe.—T. St.

Cooling apparatus composed of separate tubes. Bergedorfer Eisenwerk A.-G., Sande. Ger. Pat. 314,124, 1.2.18.

THE apparatus consists of separate tubes arranged

horizontally in a vertical plane and supported at the ends by two vertical members which consist each of one piece only and are bored out lengthwise through the centre. The horizontal tubes pass completely through one vertical boring, alternately at opposite ends, and just to the inner wall of the other vertical boring. The ends of the tubes which pass through the vertical boring are provided with holes in the upper side to afford communication with the vertical boring. The cooling liquid passes left and right through successive horizontal tubes in its passage from the bottom to the top of the apparatus, and the necessity of making water-tight joints is obviated.—T. St.

Heat interchanging apparatus. E. L. Pease, Darlington. Eng. Pat. 135,274, 18.11.18. (Appl. 18,910/18.)

A NUMBER of vertical heat-radiating columns are arranged so that the air or other fluid to be heated passes up them both inside and out. The vertical columns are heated by a hot fluid passing through superposed horizontal tubes in good metallic contact with the vertical tubes. Various diaphragms or concentric tubes may be placed within and between the vertical tubes to increase the radiating surface.—B. M. V.

Grinding, crushing, and pulverising mills. J. F. Wake, Darlington. Eng. Pat. 135,322, 29.11.18. (Appl. 19,770/18.)

A SERIES of loose rings running one within another, the outer diameter of one being less than the inner diameter of the next, are contained in a casing, the axes of all being substantially horizontal. The casing is rotated and grinding takes place between the cylindrical faces of the rings, which may be slightly tapered (say 6°) to the axes.—B. M. V.

Centrifugal attritional pulveriser. J. H. and J. Macartney, Assignors to H. T. Rudisill, Los Angeles, Cal. Reissue 14,764, 2.12.19, of U.S. Pat. 1,253,619, 15.1.18. Appl., 19.8.18.

THE pulveriser comprises two rotary hollow dished members placed face to face so as to form a shallow horizontal circular chamber. The two dished members are slightly separated so as to form a narrow peripheral outlet from the chamber. Material to be pulverised is supplied to the chamber through the hollow shaft, and is thrown against the outer wall of the chamber by centrifugal force. The two dished members rotate in opposite directions.

—W. F. F.

Corrosion in steam turbine plants; Method of preventing. A. Jude, and Belliss and Morcom, Ltd., Birmingham. Eng. Pat. 135,452, 27.8.19. (Appl. 21,041/19.)

OIL is admitted in regulated quantities to some point in the steam service of a turbine plant during the active period of service.—B. M. V.

Precipitating materials from solution; Process and apparatus for. T. B. Crowe, Victor, Colo., Assignor to The Merrill Co., San Francisco, Cal. U.S. Pat. 1,321,985, 18.11.19. Appl., 17.3.17.

THE precipitation process is facilitated by the simultaneous removal of gas from the solution, the presence of such gas interfering with the normal course of precipitation. (See also this J., 1918, 626 A.)—D. F. T.

Air dryer. B. Macfadden, New York. U.S. Pat. 1,322,804, 25.11.19. Appl., 25.3.19.

THREE vertical chambers are arranged side by side and separated by partitions having openings for the passage of horizontal endless belts of ab-

sorbent material. The first chamber is provided with means for projecting sprays of water upwards from the bottom of the chamber, and the third chamber is provided with means for heating and drying the air at the bottom of the chamber. Horizontal baffles are provided in this chamber between the heating means and the lowest endless belt. The first chamber acts as an "eliminating" chamber, the second as a cooling chamber, and the third as a drying chamber; a current of air passes upwards through all the chambers. The belts are arranged so that each is out of alinement with those immediately above and below.—W. F. F.

Solid substances; Methods of obtaining — from solutions carrying the same. G. A. Buhl, Assignor to The G. A. Buhl Co., Chicago, Ill. U.S. Pat. 1,322,875, 25.11.19. Appl., 8.7.15.

THE solution, freed from air, is forced through a coil heated to a temperature above its boiling point, where it is partly volatilised. The solution then passes into an expansion chamber, where it meets a current of air injected at a different angle, so that the two streams are mixed and the liquid evaporated. The vapour is withdrawn from the chamber, and the solid particles are collected.

—W. F. F.

Condensing apparatus. E. W. Christie, Sewaren, N.J., Assignor to Wheeler Condenser and Engineering Co., Carteret, N.J. U.S. Pat. 1,323,013, 25.11.19. Appl., 20.2.17.

A NUMBER of vertical condenser tubes are arranged in a cylindrical casing, and the upper horizontal tube-plate forms the bottom of a superposed reservoir. The condenser tubes terminate in the upper tube-plate, while a short vertical pipe of smaller diameter projects upward from the mouth of each tube into the reservoir, thus allowing a stream of water to flow downward over the inner surface of each condenser tube. A current of air is passed upward through the condenser tubes and short pipes into the atmosphere, and vapour to be condensed is passed through the space surrounding the condenser tubes.—W. F. F.

Filter leaf construction. E. J. Sweetland, Montclair, N.Y., Assignor to United Filters Corporation. U.S. Pat. 1,323,421, 2.12.19. Appl., 7.3.16.

THE filtering fabric is stretched on both sides of a circular ring and is turned over from both sides into a central channel running round the outer periphery of the ring. The ring is surrounded by an outer clamping ring T-shaped in cross-section. The central part of the T-section fits into the peripheral channel to secure the fabric at its edges.

—W. F. F.

Filter press-plate. Enzinger-Werke A.-G., Worms. Ger. Pat. 314,520, 22.4.17.

IN filter-press plates made of rubber or rubber substitute in the form of a corrugated sheet with reinforcing metal insertion fixed in an outer metal frame, the frame, together with a central metal portion, is constructed in one piece with the metal insertion, and the rubber or rubber substitute is fixed in place by a process of vulcanisation.

—L. A. C.

Filter; Plate suction — with continuous discharge for crystalline material. Konsolidierte Alkaliwerke, Westeregeln. Ger. Pat. 314,596, 7.7.16.

IN a plate suction filter the thickness of the layer of dried material ready for removal is adjusted by alteration of the distance between the filter plate and the scraper.—L. A. C.

Drying-machine. H. Parker, Berlin, N.H., Assignor to Brown Co., Portland, Me. U.S. Pat. 1,322,813, 25.11.19. Appl., 13.4.16.

ARTICLES to be dried are conveyed into a drying chamber by a quick-moving conveyor and then transferred to a slow-moving conveyor moving entirely within the chamber. The articles are transferred from the delivery end of the latter conveyor to another part of the quick-moving conveyor, which removes them from the chamber. Supports are provided on the conveyors to receive and hold the articles.—W. F. F.

Dryer. J. T. Le Messurier, Detroit, Mich. U.S. Pat. 1,323,481, 2.12.19. Appl., 31.5.17.

A CYLINDER having its curved surface formed of wire netting is mounted on a concentric perforated pipe, the common axis of both being horizontal. The cylinder is arranged in a casing having a longitudinal discharge conduit along the bottom and a removable receptacle for impurities mounted in the conduit. Air is drawn either from a heating device or from the atmosphere and forced into the pipe and cylinder by a blower at one end. The pipe and cylinder may be rotated alternately in opposite directions.—W. F. F.

Dehydration of moist materials. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin, Ger. Pat. 314,338, 14.7.17.

THE wet material is led from above into a chamber with an outlet at the bottom, the chamber being kept filled during the operation. The dehydrated material is deposited electro-osmotically upon a series of parallel plates inside the chamber resting on supports below and all attached to a horizontal rod above. By manipulation of this rod the plates can be variously inclined to the vertical position, and the distance between contiguous plates will vary correspondingly, being a maximum when the plates stand vertical. During the deposition of material between the plates the latter are turned towards the vertical position to allow for the accumulation of deposited material. When deposition is complete the chamber is emptied of liquid, the plates are then further turned until they are in vertical position, whereby the deposit drops upon the floor of the chamber and can be removed.

—H. J. H.

Liquefied gases; Vessel for transporting and storing — W. Rohn, and W. C. Heraeus G.m.b.H., Hanau, Ger. Pat. 302,532, 27.8.16.

THE actual container is surrounded by several insulating jackets contained one within the other. Each jacket is filled with a gas such that the average temperature prevailing in the jacket is very slightly above the condensation point of the gas, and is packed with so much insulating material that the combination gas plus insulating material yields the maximum insulation effect possible. Each jacket varies in thickness according to the difference in temperature between the inner and outer surfaces of the jacket at different points. Intermediate spaces between the jackets are provided with tubes leading to the outside, whereby vapour arising in the container may be made to circulate through more or less of the spaces before passing away. The rate of evaporation of the contents of the vessel can thus be controlled within a wide range. The vessel is suitable for containing all liquefied gases, including hydrogen.—T. St.

Bakelite; Method of coating autoclaves, vessels of metal or wood, etc., with — Arnstädter Malzfabrik H. & S. Windesheim, and C. ten Doornkaat-Koolman, Arnstadt, Ger. Pats. (a) 304,319, 20.8.16. (b) 305,179, 31.10.17, and (c) 307,699, 29.3.18.

(a) THE surface to be protected is roughened and

coated, by spraying, with bakelite lacquer, and this, before having completely dried, is covered with a layer of bakelite paste. Both layers are dried together, the temperature being first raised gradually to 90° C., and then, after a short time, to about 160° C. The coating is mechanically strong and resistant to acids and alkalis. (b) Protecting parts, such as rivet heads, rivet seams, etc., are covered by means of a brush with a mixture of equal parts of bakelite lacquer or enamel and bakelite paste. Hardening is made to take place under an air-pressure about half an atmosphere above the vapour pressure of the volatile solvent (alcohol) in the bakelite mixture at the respective hardening temperatures. The excess pressure during hardening prevents the formation of blisters in the bakelite coating. (c) With the object of heating uniformly and simultaneously all parts of a vessel which has been coated with bakelite, the vessel is enclosed in a sheet-iron container which is provided at the bottom with a warm-air distributor supplied centrally with a current of warm air.—T. St.

Gases; Automatic means for neutralising — L. Bergfeld, Heidelberg, Ger. Pat. 314,216, 11.10.16.

AN acid solution of known strength is brought in contact with the impure gas to be neutralised and is then added to an excess of a carbonate; the carbon dioxide liberated passes into a float contrivance, which automatically regulates the valve for admitting the liquid for washing the gas. Instead of treating the gas with an acid solution alone, both an alkaline and an acid solution of known strength, the latter in excess, may be employed, and in this case the two solutions are mixed after acting on the gas and then added to the carbonate solution. The motion of the float, instead of directly actuating the inlet valve for the wash liquor, may actuate the inlet and outlet valves of a second vessel provided with a float, which then controls the supply of wash liquor, and, either by adjusting this float or by control of the outlet for the carbon dioxide, the gas may be brought to any desired strength of alkalinity or acidity instead of to the neutral point.—L. A. C.

Mixing liquids and solids in powder or granular form. O. Stier, Heidelberg, Ger. Pat. 314,412, 4.8.17.

THE material is thrown outwards from a rotating disc or fan upon the walls of an enclosing chamber of funnel-shaped construction below so as to collect the material again. The angular velocity of the revolving parts can be varied at will. To avoid segregation of components of differing grades the discs must be suitably constructed and baffles provided to cause the material to be intimately mixed while falling through the air.—H. J. H.

Air or other gases; Compression of — with the addition of other previously compressed gas in the suction stroke. Melms u. Pfenninger Kommanditges., München-Hirschau, and F. Gensheimer, Fiume, Ger. Pat. 311,498, 24.8.17. Addition to Ger. Pat. 304,021.

THE original process is one in which the ordinary air or gas compression cylinder is equipped with an auxiliary cylinder in which additional gas is compressed to an intermediate pressure quite independently. The additional gas is drawn into the main cylinder towards the end of the suction stroke and compressed on the return. The compression in the main cylinder is therefore assisted by the compression in the auxiliary cylinder. In the present patent the apparatus is so constructed that the main piston operates both the compression of the addition gas in the auxiliary compressor and its transference after cooling to the main cylinder.

—H. J. H.

Absorption apparatus; Tubular packing for —. Deutsche Ton- und Steinzeugwerke A.-G., Charlottenburg. Ger. Pat. 314,597, 14.10.16.

STONEWARE tubes for packing absorption apparatus and the like are provided inside with baffles, some of which are inclined so as to form pockets in which liquid collects. A number of layers of the tubes are arranged one above another, and the liquid passing down the apparatus splashes on to the liquid collecting in the pockets and also on to the walls of the tubes, thus effecting good contact between liquid and gas.—L. A. C.

Heating liquids electrically; [Liquid-tight insulating joints in] apparatus for —. G. Bellaviti, Milan, Italy. Eng. Pat. 135,225, 2.11.17. (Appl. 16,017/17.)

High-temperature reactions; Apparatus for conducting —. N. Testrup, Assignor to Techno-Chemical Laboratories, Ltd., London. U.S. Pat. 1,322,652, 25.11.19. Appl., 19.3.18.

SEE Eng. Pat. 115,087 of 1917; this J., 1918, 357 A.

Desiccation of air or gases. G. Claude, Assignor to L'Air Liquide (Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude), Paris. U.S. Pat. 1,323,014, 25.11.19. Appl., 5.5.14.

SEE Eng. Pat. 11,094 of 1914; this J., 1914, 907.

Separation of substances of different specific gravities, such as fine coal or ores and the like; Apparatus for —. J. M. Draper, Bridgend. U.S. Pat. 1,323,174, 25.11.19. Appl., 12.11.17.

SEE Eng. Pat. 111,826 of 1917; this J., 1918, 45 A.

Loose material; Transference of —. C. E. Blyth, Stockton. U.S. Pats. 1,323,243 and 1,323,244, 2.12.19. Appl., 9.5.17 and 18.9.18.

SEE Eng. Pat. 104,775 of 1916; this J., 1917, 535.

Air or other gases; Apparatus for treating —. W. H. Yardley, Sheffield. U.S. Pat. 1,323,666, 2.12.19. Appl., 20.3.18.

SEE Eng. Pat. 116,512 of 1917; this J., 1918, 453 A.

Pumping [corrosive] liquids; Apparatus for —. A. H. Human, Gerrards Cross, Assignor to Kestner Evaporator and Engineering Co., Ltd., London. U.S. Pat. 1,323,864, 2.12.19. Appl., 13.3.18.

SEE Eng. Pat. 115,254 of 1917; this J., 1918, 358 A.

Extracts, infusions, or decoctions; Apparatus for enabling — to be obtained in a continuous manner. P. Kestner, Paris. U.S. Pat. 1,325,298, 16.12.19. Appl., 11.2.19.

SEE Eng. Pat. 132,075 of 1918; this J., 1919, 751 A.

Mixing apparatus. Eng. Pat. 135,544. See IX.

Apparatus for measuring charges of gas. Eng. Pat. 134,615. See XIXb.

Pyrometric method. U.S. Pat. 1,323,309. See XXIII.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Ozonisation of — suspended in non-aqueous media. F. Fischer and H. Tropsch. Ges. Abhandl. zur Kenntnis der Kohle, 1919, II., 160—168. Chem. Zentr., 1919, 90, IV., 842—843.

"SOFT" coal suspended in chloroform was rapidly attacked by ozone with simultaneous chlorination and a considerable increase in weight. The pale

brown reaction product was insoluble in chloroform, soluble in alcohol, less soluble in acetone, acetic acid, and ethyl acetate, sparingly soluble in ether, water, and aqueous acids, but soluble in aqueous alkalis. When suspended in carbon tetrachloride coal was not affected by ozone. In glacial acetic acid a reaction readily occurred and, on evaporation of the acetic acid *in vacuo*, a syrup was obtained from which ether precipitated a light brown strongly acid substance, the solubility of which in water increased with increasing duration of the ozone treatment. The yellow-coloured solution darkened on boiling, and had a strong odour of caramel. By the action of diazomethane on these products compounds of the nature of esters were obtained, insoluble in soda. The substance remaining dissolved in the acetic acid after precipitation with ether yielded a dark reddish-brown mass containing small quantities of oxalic acid. The main portion consisted of readily decomposable coloured substances of strongly acid character, insoluble in organic solvents, which formed with most metallic salts compounds insoluble in acetic acid. The ozonisation of lignite in acetic acid was analogous to that of ordinary coal. Oxalic acid was the primary product of the prolonged ozonisation of coal, but the oxalic acid was itself partially destroyed by ozone.—J. F. B.

Low temperature carbonisation. Tern. Ber., 1919, 52, 1836.

FACTORY plant embodying the features described by Fischer and Glund (this J., 1919, 563 A) was constructed by Tern as early as 1910.—J. C. W.

Ignition-temperatures [of gases]; Determination of — by the soap-bubble method. A. G. White and T. W. Price. Chem. Soc. Trans., 1919, 115, 1248—1264.

THE soap-bubble method for the determination of the ignition-temperature of mixtures of inflammable gases (McDavid, this J., 1917, 1264), in which a soap-bubble containing the vapour is made to impinge on a heated wire coil of known temperature, has been applied to determine the ignition temperatures of certain mixtures of ether, benzene, light petroleum, and hydrogen with air. After careful standardisation the results given by this method were found to be affected by the physical state of the igniting surface and the nature of the material of which it was made. Even the addition of small quantities of the salts used for standardisation purposes altered the results obtained. The ignition-temperature of a particular gas mixture often varied by more than 150° C. as determined by two different coils, and the results indicate that the method is not trustworthy even for comparative purposes.—E. H. R.

Dehydrogenation of hydrocarbons by means of palladium-black. J. Tausz and N. von Putnok. Ber., 1919, 52, 1573—1583.

A METHOD is described by which cyclohexanes can be estimated in mixtures with paraffins, depending on a measurement of the hydrogen liberated when a convenient quantity is circulated over a specially active palladium-black maintained at 270°—300° C. Cyclohexanes are completely converted into benzenes in this way, but the paraffins themselves also produce a little hydrogen, an equilibrium with the olefines being established. The volume of hydrogen produced by the cyclohexanes, however, is nearly 20 times that developed by equal quantities of the paraffins under the same conditions, so that quantitative results are obtained with a fair degree of accuracy if there is more than 1% of the cyclic hydrocarbons in the mixture. As an example of the application of the method, the proof of the presence of cyclohexane in a sample of hexane from American petroleum is given. Whereas 30 c.c. of

synthetic *n*-hexane only gave 338 c.c. of hydrogen, 30 c.c. of the natural oil gave 446 c.c., corresponding to 2.1% of cyclohexane in the sample. (See further, *J. Chem. Soc.*, 1920, ii., 61.)—J. C. W.

Ichthyol oils. Chemical constituents of the bituminous tar oils rich in sulphur. III. H. Scheibler. *Ber.*, 1919, 52, 1903—1910.

THE purification of the crude oil is effected as follows. The oil is heated with soda-lime, whereby it loses its obnoxious odour and becomes paler and more mobile. Then it is washed with dilute sulphuric acid, dried, and heated with sodium in a current of ammonia gas. After this it contains only a small quantity of a ketonic substance, which is destroyed by means of magnesium methyl chloride, and finally the oil is fractionated from sodium. A specimen of crude oil from the south of France gave nearly half its weight of a pleasant-smelling liquid, containing only carbon, hydrogen, and sulphur, all but the highest fractions being colourless. (See also *J. Chem. Soc.*, 1920, i., 74.)

—J. C. W.

Montan wax; Behaviour of — towards ozone. F. Fischer and H. Tropsch. *Ges. Abhandl. zur Kenntnis der Kohle*, 1919, 11., 162—172. *Chem. Zentr.*, 1919, 90, IV., 847.

DARK brown, powdered montan wax, suspended in carbon tetrachloride or glacial acetic acid, was changed to a light brown product by the passage of ozone for a short time, but after prolonged treatment products were obtained which were soluble in dilute alkalis, whereas montan wax itself is saponifiable only on boiling with caustic soda under pressure. Apparently the esters are decomposed by the action of the ozone with the formation of acids. The product obtained from montan wax in carbon tetrachloride was of a waxy nature, and on melting with water the latter showed a weak peroxide reaction; the waxy product could be fractionated by dissolving in alcohol, some of the fractions still containing neutral constituents. Montanic acid, which was isolated from the products of the saponification of montan wax, is also attacked by ozone in the presence of carbon tetrachloride, giving acids of lower molecular weight which form typical soaps with alkalis. When finely-powdered montan wax, suspended in aqueous caustic potash or sodium carbonate, was treated with ozone, no soluble products were formed after 24 hours. On melted montan wax ozone had only a bleaching action.—J. F. B.

Oiliness and lubrication. Deeley. *See I.*

Trinitrotoluene from petroleum spirit. Berl and Ziffer. *See XXII.*

PATENTS.

Damp peat; Preparation of — for the manufacture of producer gas. S. C. Davidson, Belfast. Eng. Pat. 135,348, 23.12.18. (Appl. 21,502/18.)

THE moisture in peat as taken from the bog cannot be reduced by mechanical pressure below about 65% without losing large quantities of valuable organic matter along with the expressed moisture. If, however, the peat is thoroughly mixed to a putty-like mass with enough lime to render the mixture distinctly alkaline, say 5%, the oily and resinous matter is coagulated, and the water content may be reduced to 30—35% by mechanical means.—B. M. V.

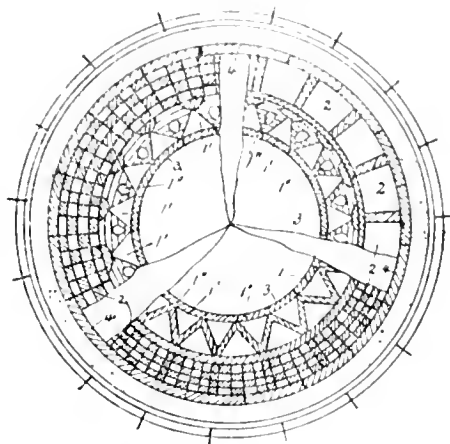
Peat briquettes; Manufacture of —. A. Pfisterer, Munich. Ger. Pat. 313,892, 19.5.18.

PEAT, humus, or the like is dehydrated by means of cloth-covered press rollers and mixed in a spiral mixer with a meal prepared from weeds or substances containing lichenin. The meal absorbs

water readily, and the mass becomes so stiff that it can easily be pressed into briquettes.—D. W.

Heating flues for combustion of gaseous, vaporous, or dust fuel, or for heating with waste gases. W. E. Davies, Penygraig, S. Wales. Eng. Pat. 135,549, 21.10.18. (Appl. 17,357/18.)

A BAND of flues lies between two parallel stretcher strips or concentrically-curved walls, 3 and 4, of brickwork, and consists of two alternating sub-series of flues, 1' and 1'', forming together a primary series of flues, and a secondary series of flues, 2. Both sets of flues of the primary series may be



used as combustion chambers, or alternate ones may be used as waste heat flues. By a suitable arrangement of primary and secondary flues the gases, after burning in the primary flues, are subdivided and sent in the opposite direction through the secondary flues. The arrangement is intended to withstand the contraction and expansion caused by the reversal during a change-over period in coke-ovens or vertical gas retorts of the regenerative type.—W. P.

Coking process. W. B. Marquard, Easton, Pa. U.S. Pat. 1,323,711, 2.12.19. Appl., 2.1.19.

A BATTERY of coke-ovens is arranged in a series of groups, each containing the same odd number of ovens. The ovens are so charged and discharged that when one oven has completed a run and is being discharged, each of the two ovens immediately adjoining the first has been running for such a time that the sum of these times is together equal to the time taken by the first for a complete run.

—W. P.

Gas producers. D. J. Smith, London. Eng. Pat. 135,319, 28.11.18. (Appl. 19,697/18.)

ALTERNATIVE means are provided for mechanically operating a producer of the type described in Eng. Pats. 120,599 and 121,416 (this J., 1919, 5A) in order that it may be operated independently of the internal combustion engine to which the producer supplies gas. A steam engine mounted on the producer is supplied with steam from a boiler heated by the producer. The steam engine operates the fuel feed, the ash discharge, and the pump for the water supply mechanically and continuously. The air supply may be controlled independently of the steam supply.—W. P.

Gas producers. D. J. Smith, London. Eng. Pat. 135,613, 9.12.18. (Appl. 20,636/18.)

SWIVELLING means are provided at the top of the producer, by means of which the lower fuel-feed tube may be readily displaced and a smoke pipe

or flue caused to register with and cover the fuel-feeding aperture. A slot arranged in some part of the feed pipe is fitted with a glass or other transparent material to enable the fuel to be watched. The invention is applicable more particularly to self-propelling vehicles (see Eng. Pats. 120,599 and 121,446; this J., 1919, 5 A), and the slot is so placed as to be within view of the driver of the vehicle.

—W. P.

Oils and semi-coke; Method for obtaining — from coal in rotating distillation apparatus. F. Fischer, Mülheim. Ger. Pat. 299,191, 14.11.16.

WHILST the coal in the retort is in a plastic condition it is rolled into a compact mass by a massive iron roller which is embedded in the charge. With a cylindrical retort 1.5 m. long and 0.5 m. in diameter, and a roller 0.1 m. in diameter, 15 kilos. of gas coal yields 11.3 kilos. of semi-coke, and 1.5 kilos. of tar oils with a calorific value of 8400 cals. The semi-coke is a transportable, dense, smokeless fuel.—T. St.

Gas producers. H. M. Iversen, Grandrup, Denmark. Eng. Pat. 135,339, 11.12.18. (Appl. 20,654/18.)

MEANS are described for improving the running conditions of producer-gas plant in which the fuel used, such as peat, lignite, or wood, gives rise to a gas which is cleaned only with difficulty. A pumping device forces the gas through a high column of washing fluid, a continuous circulation of gas being maintained by means of a relief valve and pipe.

—W. P.

Coal, water or other gases; Treatment of — and removal of substances contained therein. A. J. Pennington, Manchester, and M. C. Lamb, London. Eng. Pat. 135,577, 25.11.18. (Appl. 19,386/18.)

CONDENSED oils of the pyridine and analogous series, obtained by the destructive distillation of bones or leather, are utilised for removing naphthalene and anthracene from coal gas, or for enriching coal- or water-gas. The carbonaceous residue from the distillation of bones or leather may be treated with steam whilst still in the heated retort for the production of water-gas.—W. P.

Petrols, petroleum, benzols and the like; Process and apparatus for the continuous fractional distillation of —. E. Barbet et Fils et Cie, Paris. Eng. Pat. 117,615, 22.6.18. (Appl. 10,341/18.) Int. Conv., 3.7.17. Addition to Eng. Pat. 9088 of 1914.

IN an apparatus for the continuous fractional distillation of petroleum and the like, of the type described in the chief patent (see Fr. Pat. 468,068 of 1913; this J., 1914, 954), the oil, after dehydration, is heated under atmospheric pressure in a plate column in which the lighter fractions are separated; the residual oil is then distilled under vacuum with multiple (triple) heating effect for separation into different fractions.—L. A. C.

Shales and coals; Treatment [distillation] of —. D. E. Day, Washington, D.C. U.S. Pat. 1,323,681, 2.12.19. Appl., 4.3.18.

HOT flue gases containing not more than a trace of oxygen are utilised for distilling off the hydrocarbon oils from shales. These gases are passed, together with steam, through the material, which is subjected to agitation as it passes through the retort.—W. P.

Shale oils; Process for obtaining — in rotary carbonising retorts. Königl. Bau- und Bergdirektion, Stuttgart, and Zeller und Gmelin, Eisingen. Ger. Pat. 303,803, 1.3.17.

The shale is conveyed continuously to the upper

end of a rotary drum, and in the course of its passage through the drum it is subjected to a gradually increasing temperature and discharged at the other end, the resulting vapours being drawn off in the usual manner. The drum is divided into several sections, each surrounded by a separate heating chamber in which the temperature is raised to the desired degree. The shale is fed to the drum and the gases and the residue are withdrawn all under exclusion of air.—J. F. B.

Cooler. Ger. Pat. 313,958. See I.

Sulphur compounds. Ger. Pat. 309,159. See VII.

Purifying liquids. U.S. Pats. 1,323,239, 1,323,251, and 1,323,256. See XIXb.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

PATENTS.

Cellulose-containing materials [e.g., peat]; Treatment [wet-carbonising] of —. N. Testrup, and Wetcarbonizing, Ltd., London. Eng. Pat. 135,578, 25.11.18. (Appl. 19,389/18.) (See also Eng. Pat. 18,030 of 1914; this J., 1914, 912.)

PEAT is wet-carbonised in suitable apparatus at the lowest economical temperature. The resulting mass is filtered, the press effluent evaporated, preferably in apparatus of the type described in Eng. Pats. 12,462 and 22,670 of 1911 (this J., 1912, 971; 1913, 183), and the acid recovered from the steam or concentrated in the liquor. The recovered acid is added, together with any additional acid required, to a fresh batch of peat. The process results in a great saving of heat.—W. P.

Peat, brown-coal, lignite, sapropel, oil-shales, wood, etc.; Treatment [distillation] of —. C. Francke, Berlin. Ger. Pat. 314,337, 2.11.17.

PEAT or other material is heated in slowly-revolving drums provided at the sides with tubes for leading gas to and away from the drums. The material is first dehydrated and then distilled at temperatures up to 550° C. The drums are heated from below by gas arising from the distillation, after treatment for the recovery of condensable products. Material containing 35% of water can be treated in two hours.—T. St.

Removing gases [from X-ray tubes]; Method of — and apparatus produced thereby. W. D. Coolidge, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,323,836, 2.12.19. Appl., 6.10.16.

SEE Eng. Pat. 109,358 of 1916; this J., 1917, 1088.

Tar-containing liquids. Eng. Pat. 120,558. See III.

Liquors from straw paper factories. Ger. Pat. 314,054. See V.

Tungsten. Ger. Pat. 306,364. See X.

III.—TAR AND TAR PRODUCTS.

Nitrobenzene; Determination of the course of the catalytic reduction of —. F. F. Nord, Ber., 1919, 52, 1705—1712.

β -PHENYLHYDROXYLAMINE is known to play an important part in the chemical or electrolytic reduction of nitrobenzene to aniline, and experiments were made to determine whether it is formed immediately during catalytic reduction. *o*-Nitrobenzaldehyde and *o*-nitroacetophenone were shaken with hydrogen and colloidal platinum, and by stopping the reduction after two molecular propor-

tions of hydrogen were absorbed the author was able to isolate anthranil and methylantranil, respectively, these being the internal condensation products of the phenylhydroxylamine derivatives. (Compare J. Chem. Soc., 1920, i., 21.)—J. C. W.

Phthalic anhydride, II. *Melting point of pure phthalic anhydride.* The system: *phthalic anhydride-phthalic acid*. K. P. Monroe. J. Ind. Eng. Chem. 1919, 11, 1116—1119.

PHthalic anhydride made by the catalytic oxidation of naphthalene (Gibbs; this J., 1918, 684A) shows a m. pt. (130° C.) about three degrees higher than that previously recorded. In producing still purer preparations the material was warmed for 5 hours in a vacuum oven at 80° C., and twice sublimed *in vacuo* in the presence of phosphorus pentoxide. No differences were observed by analysis or in the m. pt. of the first and second sublimates. A differential method of titration was devised by Adams in which 0.5 gm. of the sample was dissolved in 50 c.c. of acetone, a few drops of a solution of Bromo-phenol Blue (tetrabromophenolsulphon-phthalein) added, and the solution titrated with *M*/10 normal potassium phthalate solution in 80% alcohol. In the presence of phthalic acid the end-point of the titration is reached when sufficient phthalate has been added to produce the acid phthalate, and a further addition causes the colour to change to indigo-blue. Tested by this method the second sublimate of phthalic anhydride was found to contain less than 0.1% phthalic acid. The melting point was found to be 130.84° C. Mixtures of weighed quantities of pure phthalic acid and pure phthalic anhydride were tested in the same melting-point apparatus and the results plotted in freezing-point curves. It was found that the eutectic temperature (129.74° C.) corresponded to a mixture containing 1.96% of phthalic acid. Owing to the readiness with which this acid decomposes near its melting point into phthalic anhydride and water, direct determinations only afford an approximation to the true m. pt. By graphical extrapolation the m. pt. of pure phthalic acid is indicated to be $208^{\circ}\pm 2^{\circ}$ C.—C. A. M.

Phthalic anhydride, III. *The system: naphthalene-phthalic anhydride.* K. P. Monroe. J. Ind. Eng. Chem., 1919, 11, 1119—1120.

MIXTURES of pure naphthalene (m. pt. 80.05° C.) and phthalic anhydride (m. pt. 130.84° C.) were examined, and the freezing-point curves for the system plotted. The eutectic temperature 64.9° C. corresponded to a mixture containing 29.0% of phthalic anhydride. No evidence was obtained of the existence of solid solutions. (See also J. Chem. Soc., Feb., 1920.)—C. A. M.

PATENTS.

Tar; Removing the — from tar-containing liquids. E. Barbet et Fils et Cie., Paris. Eng. Pat. 120,558, 9.10.18. (Appl. 16,457/18.) Int. Conv., 11.10.17.

AQUEOUS solutions contaminated with tar, e.g., the pyroligneous liquid obtained in the distillation of wood, are treated with organic liquids insoluble in water but capable of readily dissolving the tar, such as cresol or the like. A cylindrical vessel is packed with porcelain balls, quartz, or coke supported by a perforated iron plate placed a short distance above the bottom of the vessel, and an open space is also left at the top of the vessel. Cresol and the aqueous liquid are admitted through the side of the vessel by inlets situated respectively near the top and bottom of the portion filled with packing; the aqueous liquid rises and is scrubbed by the descending stream of cresol. In the open space at the top of the vessel any cresol suspended

in the liquid separates, and a clear solution free from tar is drawn off continuously; cresol containing tar in solution is drawn off from the space below the packing, and is purified by distillation.

—L. A. C.

Wood tar; Extraction of higher fatty acids and other organic acids from —. E. Börnstein, Berlin. Ger. Pat. 314,358, 26.1.17.

The tar is fractionally distilled under reduced pressure and the fatty acids in the fraction boiling at 190°—235° C. at 15 mm. are extracted in the usual manner. The yield varies with the nature of the tar. One kilogram of a crude pine tar yielded approximately 28 grms. of oleic acid, 48 grms. of palmitic acid, and a small quantity of liquid and solid abietic acid; arachidic acid was present in the fractions of higher boiling point.—D. F. T.

Sulphonation of hydrocarbons with sulphur trioxide. C. R. Downs, Cliffside, N.J., Assignor to The Barrett Co. U.S. Pat. 1,321,991, 18.11.19. Appl., 11.6.19.

AN aromatic hydrocarbon whilst in the state of vapour is sulphonated with a gas containing sulphur trioxide.—D. F. T.

Nitro compounds; Process for the production of —. L. J. J. Perruche, St. Gervais, France. U.S. Pat. 1,325,168, 16.12.19. Appl., 19.8.18.

See Eng. Pat. 131,982 of 1918; this J., 1919, 812 A.

Distillation of benzols, etc. Eng. Pat. 117,615. See 11 A.

Purifying liquids. U.S. Pats. 1,323,239, 1,323,251, and 1,323,256. See XIX B.

IV.—COLOURING MATTERS AND DYES.

Alizarin; Action of potassium hypochlorite on — in alkaline solutions. R. Scholl, Ber., 1919, 52, 1829—1836.

ALIZARIN is oxidised by hypochlorite in potassium hydroxide solution, the bluish-black potassium salt of 3,4,3',4'-tetrahydroxy-2,2'-dianthraquinonyl being formed. This is much poorer than alizarin as a mordant dye. When distilled with zinc dust, in an atmosphere of hydrogen, under reduced pressure, it forms 2,2'-dianthryl, and when heated with zinc chloride it loses water and gives 1,1'-dihydroxy-2,2'-dianthraquinonylene-3,3'-oxide. (See further, J. Chem. Soc., 1920, i., 61.)—J. C. W.

Phenols as mordant dyes. R. Möhlau. Ber., 1919, 52, 1730—1731.

PHENOLS which are capable of forming internally complex salts impart more or less fast shades to mordanted wool. 1,8-Dihydroxynaphthalene, α - and β -anthrols, phenanthraquinol, and 1,2-dihydroxynaphthalene-4-sulphonic acid give colours which are fast to air and light, boiling soap solution, or boiling *N*/1 hydrochloric acid. (See further, J. Chem. Soc., 1920, i., 36.)—J. C. W.

Triphenylmethane dyes; Constitution of —. H. Kauffmann. Ber., 1919, 52, 1421—1422.

THE hypsochromic effect of the introduction of a third amino-group in Malachite Green can only be explained on electrochemical lines. By converting Michler's ketone into the chloride and condensing this with *m*-dimethoxybenzene in presence of aluminium chloride a compound is obtained having two methoxy groups in the third nucleus of Malachite Green, namely, 4,4'-tetramethyldiamino-2'',4''-dimethoxytriphenylcarbinol, m. pt. 195° C. Solutions of this white substance have the following

colours; dilute acetic acid, bluish-green; dilute mineral acids, red, becoming bluish-green on dilution; concentrated sulphuric acid, orange.

—J. C. W.

Phthalic anhydride; Condensation of — with phenols in the presence of aluminium chloride. F. Ullmann and W. Schmidt. Ber., 1919, 52, 2098—2118.

HYDROXYBENZOYLbenzoic acid derivatives are prepared in uniformly good yield by the condensation of phenols with phthalic anhydride by means of aluminium chloride in presence of acetylene tetrachloride as solvent. The carbonyl-group of the phthalic anhydride attaches itself for the most part to the carbon atom in the ortho-position to the hydroxyl-group of the phenol. Starting from tetrachlorophthalic anhydride, *o*-hydroxybenzoylbenzoic acid derivatives are almost exclusively obtained which are readily converted by alkalis into xanthone derivatives. Phthalic anhydride and *p*-cresol yield 5'-methyl-2-hydroxy-*o*-benzoylbenzoic acid, $C_6H_4(CH_3)(OH).CO_2C_6H_4.CO_2H$, colourless prismatic crystals, m. pt. 194°—195° C., which is converted by sulphuric acid into 1-hydroxy-4-methyl-anthraquinone, reddish-yellow needles, m. pt. 175° C. 2'-Hydroxy-4'-methyl-5'-chloro-*o*-benzoylbenzoic acid, colourless prisms, m. pt. 205°—206° C., is prepared from *p*-chloro-*m*-cresol and is transformed by sulphuric acid monohydrate into 1-hydroxy-3-methyl-4-chloroanthraquinone, orange-red needles m. pt. 177° C. The latter is converted by *p*-toluidine in the presence of copper powder into 1-hydroxy-3-methyl-4-*p*-toluidinoanthraquinone, bluish-violet needles, m. pt. 191° C. The chlorine atom can be replaced by the hydroxyl-group by treatment with sulphuric and boric acids at 150°—160° C., whereby 1,4-dihydroxy-2-methylanthraquinone (2-methylquinizarin), carmine-red needles, m. pt. 177° C., is formed. 1-Hydroxy-3-methyl-4-toluenesulphonaminoanthraquinone, yellowish-brown needles, m. pt. 213°—214° C., is obtained by heating the hydroxymethylchloroanthraquinone with potassium acetate, *p*-toluenesulphonamide, and copper acetate in amyl alcoholic solution and is hydrolysed to 1-hydroxy-3-methyl-4-aminoanthraquinone, violet needles, m. pt. 257°—258° C. When heated with potassium and copper acetates in the presence of naphthalene the hydroxymethylchloroanthraquinone yields 1-hydroxy-3-methylanthraquinone, m. pt. 178° C. The condensation of *p*-cresol with tetrachlorophthalic anhydride gives 2'-hydroxy-5'-methyl-3,4,5,6-tetrachloro-*o*-benzoylbenzoic acid, m. pt. 232°—235° C. according to the rate of heating, which is quantitatively transformed by alkali carbonate or hydroxide into 2-methyl-5,6,7-trichloroxanthone-8-carboxylic acid, colourless prismatic needles, m. pt. 263°—266° C. Under similar conditions *m*-cresol gives 2'-hydroxy-4'-methyl-3,4,5,6-tetrachloro-*o*-benzoylbenzoic acid, pale yellow plates, m. pt. 226°—228° C., which yields 3-methyl-5,6,7-trichloroxanthone-8-carboxylic acid, long, colourless prismatic needles, m. pt. 254°—256° C. (decomp.). 2'-Hydroxy-3'-methyl-3,4,5,6-tetrachloro-*o*-benzoylbenzoic acid, yellow rhombic plates, m. pt. 222°—225° C. and 4-methyl-5,6,7-trichloroxanthone-8-carboxylic acid, colourless prismatic needles, m. pt. 270°—273° C. (decomp.) are successively prepared from *o*-cresol. Phenol yields 2'-hydroxy-3,4,5,6-tetrachloro-*o*-benzoylbenzoic acid, colourless, shining leaflets, m. pt. 216°—218° C. and 5,6,7-trichloroxanthone-8-carboxylic acid, colourless prismatic needles, m. pt. 261°—264° C., whilst β -naphthol gives 2'-hydroxy-3,4,5,6-tetrachloro-2- α -naphthoylbenzoic acid, yellowish rhombic plates, m. pt. 214°—217° C. and 4',5',6'-trichloro-1,2-naphthoxanthone-1'-carboxylic acid, needles, m. pt. 273°—275° C. (decomp.). All the recorded m. pts. are "corrected."

—H. W.

PATENT.

Azo dyestuffs dyeing on mordants and process of making same. C. Jagerspacher, Assignor to Society of Chemical Industry in Basle, Switzerland. U.S. Pat. 1,323,285, 2,12.19. Appl., 14.5.19.

DYESTUFFS prepared by combining 1,8-dihydroxynaphthalenesulphocarboxylic acids with aromatic diazo compounds which do not contain a hydroxyl group in the *o*-position to the azo-group dye wool, in an acid bath, red to blue tints. On subsequent chroming of the dyed wool, or by printing the colours together with chromium mordants on cotton, fast blue to black shades are produced.—L. A. C.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cellulose; Modification of — in the beetling process. C. F. Cross. J. Soc. Dyers and Col., 1919, 35, 271. (See also this J., 1919, 7A.)

NORMAL bleached cotton cloth was damped and beetled continuously in a layer 2 ins. thick until the fibre was tendered up to the point of destruction. In this treatment considerable heat was developed up to a maximum temperature of 200° C. The following results were obtained on examination of the fibre under the action of sodium hydroxide solution of 17.5% concentration. Original cotton:— α -cellulose, 99.7%; β -cellulose, 0.8%. Half-tendered portion:— α -cellulose, 92.3; β -cellulose, 4.9; hygroscopic moisture, 3.6%. Fully tendered portion:— α -cellulose, 78.0; β -cellulose, 16.2; hygroscopic moisture, 3.3%. The results show that the destroyed fibre was not identical with the fine powder previously described.—J. F. B.

Cellulose; Colloidal —. Investigations on the dissociation of mordant salts by cellulose. C. G. Schwalbe. Z. angew. Chem., 1919, 32, 355—357. (See also this J., 1919, 858A.)

In the manufacture of "pergamyn" (greaseproof) papers, Mitscherlich wood pulp is beaten in the hollander until it acquires a slimy condition. This type of wood cellulose is characterised by a relatively high cupric-reducing value, and if hydrocellulose or oxycellulose be similarly beaten in the hollander, the colloidal condition is reached far more quickly than in the case of cellulose itself. Moreover, if a normal cotton cellulose with low cupric-reducing power be beaten, the conversion into a colloidal condition takes very much longer, and when the modification is ultimately effected the cupric-reducing power of the product is considerably higher than that of the original material. Hence it appears probable that the formation of colloidal cellulose by prolonged beating is connected with the production or presence of hydrocellulose and oxycellulose. The slimy modifications of cellulose exhibit typical colloidal properties; they dry up in the air to tough, horny masses which swell out again considerably in an atmosphere saturated with moisture, but if the colloid be dried at a high temperature it becomes irreversible, and the tendency to absorb moisture is strongly reduced. The gelatinous celluloses produced by mechanical treatment are different from those produced by the swelling action of concentrated solutions of acids, alkalis, or salts. The latter "mercerised" celluloses are characterised by the formation of a blue compound with iodine reagents, whereas the mechanically prepared colloids have little affinity for iodine. On the other hand, the colloids produced by prolonged beating have a very pronounced influence in promoting the dissociation of aluminium sulphate with fixation of aluminium hydroxide, and this property is enhanced in the case of colloids produced by the

mechanical treatment of hydrocellulose or oxycellulose. The dissociation of neutral salts which takes place during the steaming or ageing of mordanted goods and the fixation of metallic bases and dye-stuffs are probably intimately connected with the formation of hydrocellulose or oxycellulose or both. The conditions of the reaction have considerable influence on the result, and optimum effects are obtained by restricting the proportion of water vapour present. The utility of the reaction, however, is limited by the danger of tendering the fibre. In order to avoid this it may be possible to employ external applications of artificially-prepared hydrocellulose or oxycellulose or to utilise the natural hemicelluloses, in which case it is to be noted that the colloidal properties of these substances can be developed by pressure, whereby a gelatinous modification is produced. Possibly the merits of the jigger and padding machine methods of dyeing may be attributed to this influence of pressure. Lastly, it is pointed out that the presence of gelatinised hydrocellulose or oxycellulose has an important influence upon the wetting of the goods. Goods which have been mordanted with metallic salts are difficult to wet, owing to the formation of an irreversible condition of the colloid in combination with the metallic hydroxide, similar to the effect produced by hot ironing of the colloidal cellulose in the absence of metallic mordants.—J. F. B.

Wood; Constituents of — which give colour reactions. III. H. Wichelhaus. Ber., 1919, 52, 2054—2056. (Compare this J., 1916, 1151; 1918, 129 A.)

THE active constituents have been removed from the distillates by extraction with ether and purified by distillation under greatly reduced pressure; two fractions, b. pt. 88° C. at 0.4 mm. and 95° — 105° C. at 0.4 mm., are obtained together with formic acid. Analyses of the fractions give results in agreement with those required by the formulæ, $C_{14}H_{22}O_8$ and $C_{16}H_{22}O_{11}$, respectively, which are simply related to the formulæ of brasilin and hamatoxylin.—H. W.

Gloss of papers. Kieser. See XXI.

PATENTS.

Vegetable fibrous materials; Treatment of — and production of detergents. The Amber Size and Chemical Co., Ltd., London, and C. Weygang, Maidenhead. Eng. Pat. 135,210, 15.10.18. (Appl. 16,825/18.)

STRAW, wood, flax, jute, or the like, is boiled with water containing a mixture of 1 part of an alkali metal compound of low alkalinity, such as a sesquicarbonate, or "Crex," and 1 to 4 parts of calcium carbonate. The relative proportions of the constituents, the strength of the solution, and the time of boiling are varied according to the product desired; thus, if straw is boiled for 5—10 hours in an open vessel with water containing 6—8% of a mixture of 1 part of the alkali compound and 2 parts of calcium carbonate, the fibre is reduced to a gelatinous condition and gives a horny and more or less transparent paper, but if the proportions of the mixture are halved the fibres are left more distinct, but are softer and tougher than the untreated fibres. The liquor obtained after separation of the fibrous material may be utilised as such in the manufacture of soap, or it may be allowed to stand and the sediment, either in a pasty condition or dried and ground, may be added to soap, to which it imparts increased detergent properties.—J. A. C.

Wool; Process of treating [cleaning] —. H. Y. McBride, Artesia, N. Mex., Assignor to The United States Wool Co., Denver, Colo. U.S. Pat. 1,323,611, 2.12.19. Appl., 15.1.17.

IS the process of cleaning wool, dry pulverised cleaning material and a suitable proportion of dry pul-

verised lubricating material are applied to the wool, which is then combed in the presence of a further quantity of similar cleaning material. A quantity of dry pulverised lubricant is then applied to the fibre. The cleaning operation is conducted in progressive stages in which the cleaning material moves in counter-current to the fibre.—S. S. A.

Hop bark; Process for obtaining fibre from —. U. Dammmer, Berlin-Dahlem. Ger. Pat. 299,164, 27.1.17.

THE stripped bark is steeped for a short time in a highly-dilute solution of lactic acid; it is then transferred to pure water and the fibre is separated from the bark. The mass surrounding the fibre is converted by the lactic acid into a form in which it can be easily removed by water.—J. F. B.

Willow or poplar bark; Process for utilising —. J. Kersting, Weine. Ger. Pat. 300,611, 29.2.16.

GREEN willow or poplar wood is steamed at 150° — 180° C., the bark is stripped, shredded, and boiled with water under pressure, whereby a tanning liquor is obtained, and the bark is further shredded, washed, and rolled in order to prepare the fibre for spinning.—J. F. B.

Fibre plants; Process for treating —. E. Einstein, Hechingen. Ger. Pat. 305,633, 13.3.17.

THE plants are boiled for a short time in a nearly saturated solution of caustic alkali, with or without pressure, and the treatment is repeated if necessary. Plants which are easily treated, for instance, nettle stems or isolated bast, are given first a preliminary boil in spent liquor and then treated at about 100° C. in a caustic soda lye containing about 1 kilo. NaOH to 1 litre of water for 15—30 minutes. The material is squeezed or rinsed and boiled with several changes of water. The treatment is repeated until a spinning fibre sufficiently clean and colourless is obtained. In the case of fibrous materials more difficult to treat, such as broken hops or rushes, a caustic soda bath saturated at a higher temperature is used, and solid caustic soda is added during the boiling to replace that which has been consumed; the first boiling may be prolonged to 1—2 hours.—J. F. B.

Tree bark; Process for obtaining textile fibre from —. F. Kniebel, Rühn. Ger. Pats. (A) 305,655, 12.10.17, and (B) 307,721, 18.11.17.

(A) POPLAR twigs are boiled, the bark is stripped, boiled with sodium carbonate, washed, centrifuged, shredded by carding in the moist condition, dried, and spun. (B) Instead of poplar twigs, the twigs and branches of linden, willow, and other trees may be treated in the above manner.—J. F. B.

Tree barks; Manufacture of light-weight millboards from —. O. Zimmermann, Berlin-Lichterfelde. Ger. Pat. 305,697, 16.8.17.

TREE bark is ground in a kind of bone mill, beaten in a hollander, and delivered to a strainer, from which the beaten stuff, diluted with water, passes to a millboard machine, on which it is worked up as the middle layer of the finished board. The bark pulp has a certain colouring power for boards made of waste papers, and yields a board of low specific gravity.—J. F. B.

Textile goods; Process for rendering — water-repellent. Farbenfabr. vorm. F. Bayer and Co., Leverkusen. Ger. Pat. 307,111, 16.12.16.

THE fabrics are impregnated with an aqueous emulsion of montan wax prepared by the aid of alkalis. Montan wax is heated and stirred with a 1% solution of sodium hydroxide to form a permanent, non-separating emulsion, suitable for impregnating without weakening the fibre.—J. F. B.

Oxycellulose free from saline matter; Production of — R. Adler, Vienna. Ger. Pat. 314,311, 9.1.14.

SALTS of oxycellulose are mixed with an inert solvent such as methyl or ethyl alcohol, ethyl or amyl acetate, or acetone, and then treated with an acid of which the corresponding metallic salt is soluble in the solvent used; the free oxycellulose is then washed with more of the solvent and dried at a low temperature. The dried product forms a hard transparent mass which is completely soluble in alkalis and can be used for the preparation of cellulose solutions.—D. F. T.

Sulphate pulp; Recovery process in the manufacture of — E. Olsson, East Angus, Quebec. U.S. Pat. 1,322,043, 18.11.19. Appl. 16.8.18.

SODIUM bisulphate is added to the washing liquid ("black liquor?"), the solution is evaporated, and the residue burned.—C. A. K.

Paper pulp; Process of colouring — with sulphur dyestuffs (thiophor-dyestuffs). C. Jäger, G.m.b.H., Düsseldorf. Ger. Pat. 298,826, 17.3.16.

A COLLOIDAL suspension of a sulphur dyestuff, which may be prepared by boiling the dyestuff with Turkey-red oil or "monopol oil," is reduced by an alkaline solution of hydrosulphite, and the leuco compound so obtained is added to the pulp in the course of manufacture; it has a strong affinity for the pulp and the colouring, re-oxidation, and fixation take place in the cold, the pulp being sized at the same time. If hydrogen peroxide be used as the oxidising agent and aluminium sulphate as the fixing agent, very brilliant shades are obtained.—J. F. B.

Wood or other cellulose materials; Manufacture of cellulose from — Zellstoffabrik Waldhof, and H. Clemm, Mannheim, and R. Willstätter, Munich. Ger. Pat. 304,214, 20.10.16.

THE material is treated with liquid or gaseous reagents at a comparatively moderate temperature, but at a greatly increased pressure created not entirely by heating the liquid, but largely by forcing in a liquid or gas. The digestion may be carried out under a pressure of 50 atm. in order to obtain a good penetration of coarser material and a more complete digestion. The avoidance of high temperatures while maintaining all the advantages of a high pressure gives a cellulose fibre in a better state of preservation than by the usual process, and the constituents of the spent liquors are less profoundly decomposed, so that the by-products may be more advantageously utilised. The process may also be utilised in the saccharification of cellulose.—J. F. B.

Pyroxylin solvent and composition containing the same. E. M. Flaherty, Parlin, N.J., Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,323,624, 2.12.19. Appl., 5.3.18. Renewed 26.9.19.

A PYROXYLIN composition is dissolved in a mixture of butyl acetate, xylol, and butyl alcohol.—S. S. A.

Pyroxylin; Process of treating — and products thereof. A. Schwarzman, Assignor to S. Kellogg and Sons, Inc., Buffalo, N.Y. U.S. Pat. 1,323,792, 2.12.19. Appl., 29.3.19.

To reduce the inflammability of pyroxylin it is treated with halogen until a substantial amount of the latter is chemically combined.—S. S. A.

Cellulose preparations; Process for purifying and improving —. Elektra-Osmose A.-G. (Graf Schwerin Ges.). Ger. Pat. 305,118, 18.3.17. Addition to Ger. Pat. 296,053 (this J., 1917, 593).

THE process is conducted in the presence of substances with an alkaline reaction, for which purpose

small additions of ammonia or sodium hydroxide are suitable. Owing to the continuous presence of hydroxyl ions in the intermediate chamber the migration of the acid impurities is greatly accelerated, so that the purification of the materials can be effected in a much shorter time than without such additions. In the course of the treatment the added substances are themselves removed by the electric current, so that products, such as nitrocellulose, are obtained which are free from both acid and alkaline residues.—J. F. B.

Wood cellulose as a substitute for cotton in chemical industries; Manufacture of highly-purified — W. Schacht, Weissenfels. Ger. Pat. 306,366, 14.3.18.

WOOD cellulose prepared by any of the alkaline processes is further digested by boiling with a sulphite solution. The gelatinous substances (hemicelluloses) which are present in soda-wood pulps are removed by the sulphite treatment, and a highly-purified cellulose is obtained, suitable for use as a substitute for cotton in the chemical and paper-making industries.—J. F. B.

Beating and refining engine; Continuous — for use in the manufacture of paper and the like. T. H. Nash, St. Paul's Cray. Eng. Pat. 135,733, 5.3.19. (Appl. 5479/19.)

A SERIES of beating rolls is mounted in a common trough, the bottom of which is so constructed that the pulp or the like gradually ascends whilst passing under the rolls, and is thereafter returned, by pipes or ducts passing through or beneath the bottom, to an opening in the feed plate. An adjustable deflector is fitted near each opening in the feed plate, by means of which the stream of pulp passing from the feed plate may be narrowed, thereby causing the pulp to pass under the rolls a greater number of times.—S. S. A.

Paper and other absorbent materials; Process for sizing —. Holzverkohlungs-Ind. A.-G., Konstanz. Ger. Pat. 303,925, 24.2.16.

ALKALINE solutions or suspensions containing products of the condensation of tar and aldehydes in the presence of alkalis are added to the paper pulp, and the sizing constituent is precipitated by substances with an acid reaction. Example: Beechwood tar is freed from acetic acid and other acid substances and then condensed with formaldehyde in the presence of alkali. The quantity of formaldehyde is so chosen that it is not sufficient to condense all the tar constituents. Paper can be satisfactorily sized with 1% of the product. The product may also be mixed with bakelite resins soluble in alkalis.—J. F. B.

Paper; Process of sizing —. H. Wandrowsky, Berlin. Ger. Pat. 314,146, 30.11.17.

SIZING is effected by means of the natural resins present in wood cellulose by adding substances having an alkaline reaction to the wood pulp during the beating, in order to convert the resin into a resinate, which is then precipitated on the fibre by aluminium sulphate.—J. F. B.

Straw paper and straw-board factories; Utilisation of the spent time liquors from —. R. Adam, Bremen. Ger. Pat. 314,054, 28.3.17.

THE spent liquor separated from the straw pulp by mechanical means is evaporated, dried, and the residue submitted to destructive distillation. The products resemble those from the distillation of wood and comprise acetone and methyl alcohol.—J. F. B.

[Paper] coating machines. C. W. Mayer, Rochester, N.Y., U.S.A. Eng. Pat. 135,678, 6.1.19. (Appl. 397/19.)

Conditioning of textile fibres and other materials; Moistening or —. H. Smethurst, Hollinwood. Eng. Pat. 135,748, 5.4.19. (Appl. 8634/19.)

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Anthocyanins and certain related compounds; The tinctorial properties of some —. A. E. Everest and A. J. Hall. J. Soc. Dyers and Col., 1919, 35, 275—279.

THE anthocyan pigments give beautiful shades fast to light, but not fast to washing, and very sensitive to acids and alkalis. Both the anthocyanins (glucosides) and the anthocyanidins (products of hydrolysis) derived from them dye full shades on tannin-mordanted cotton from a faintly acid bath, but on wool mordanted with a metallic oxide the anthocyanins have scarcely any tinctorial power, whereas the corresponding anthocyanidins dye readily. Thus it would appear that the affinity for the tannin-mordant is due to the pyran ring containing basic oxygen, and that the affinity for the metallic mordants is due to the phenolic hydroxyls of the catechol nucleus, which, in the form of the glucosides, are masked by the sugar residues. Experiments with violamin chloride showed that in the change from the coloured form to the colourless pseudo-base the tinctorial properties are entirely extinguished. Several synthetic compounds have been prepared and studied, among them 2-phenyl-benzopyryronium chloride, the parent substance of the anthocyanins; full dyeings of straw-yellow shade were obtained with this substance on a tannin mordant in a bath made nearly neutral with alkali or sodium acetate. Like the natural compounds of the anthocyan series, this substance also tends to pass into a colourless pseudo base in dilute aqueous solution. In the case of pure pigments the dye-bath is made up by dissolving 0.0025 gm. of the pigment in a mixture of 10 c.c. of alcohol and 40 c.c. of water and the solution acidified by five drops of 10% sulphuric acid. In this bath 1 gm. of wool or mordanted cotton is dyed at 25°—30° C. When extracting the colouring matter from flower petals, fruit, or fruit skins the same procedure may be followed, or else the materials may be extracted with alcohol containing the acid and the solution poured into water to make the bath.—J. F. B.

Colloidal cellulose. Schwalbe. See V.

PATENTS.

Woollen goods; Process for increasing the resistance to wear of dyed —. L. Cassella und Co., Frankfort. Ger. Pat. 303,231, 17.10.16.

THE material is impregnated with chromium salts, e.g. chromium acetate, and then steamed or otherwise treated so as to separate chromium hydroxide. The quantity of chromium should be at least 1% Cr₂O₃ referred to the dry material.

Colouring paper pulp. Ger. Pat. 298,826. See V.

[Chrome] leather waste. Eng. Pat. 115,421. See XV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Ammonia; Synthesis of — at very high pressures. G. Claude. Comptes rend., 1919, 169, 1039—1041.

HAVING previously shown the possibility of using very high pressures in industrial processes (this J., 1919, 885 A), the author has studied the synthesis of

ammonia at pressures ranging up to 1000 kilos. per sq. cm. At this pressure and at a temperature of 536° C. the yield of ammonia exceeds 40% as against the 13% yield at the pressures used by the Badische Anilin Co. At these high pressures the temperature range within which a reasonable reaction velocity is obtained is 500°—700° C. At a temperature of 600° C., with the catalyst commonly used, 6 grms. of ammonia at a concentration of 25% is obtained per gram of catalyst per hour, as against 0.5 gram at a concentration of 6% obtained by the Badische Anilin Co.—W. G.

Ammonia gas equilibrium; Calculation of the —. E. Maurer. Z. anorg. Chem., 1919, 108, 273—302.

A COMPARISON is made of the results obtained for the nitrogen-hydrogen-ammonia equilibrium at different temperatures and pressures by Haber and Le Rossignol (this J., 1913, 134), and Jost respectively. It is concluded that the results of these experimenters are in full agreement, and, further, that the work of Jost (Z. anorg. Chem., 1908, 57, 414; J. Chem. Soc., 1908, ii., 362, 761) was a sufficient scientific foundation for the technical synthesis of ammonia.—E. H. R.

Ammonia oxidation; Heat of reaction of —. G. B. Taylor. J. Ind. Eng. Chem., 1919, 11, 1121—1123.

A GENERAL formula for calculating the theoretical heat of reaction for any mixture of ammonia and air, at any conversion efficiency is based upon the following equations:—

- (1) $4\text{NH}_3 + 5\text{O}_2 = 4\text{NO} + 6\text{H}_2\text{O} + 214,200 \text{ cal.}$
- (2) $4\text{NH}_3 + 3\text{O}_2 = 2\text{N}_2 + 6\text{H}_2\text{O} + 300,600 \text{ cal.}$

The optimum temperature for the catalytic oxidation of ammonia to nitric acid has been shown to exceed 800° C. Both calculation and experiment have shown that the heat developed in the reaction is insufficient to maintain the optimum temperature for the catalysis, and a source of external energy is therefore necessary. Experiments have shown that electric heating, pre-heating the ammonia-air mixture, or enriching the current of ammonia and air with oxygen give equally satisfactory results.

—C. A. M.

Nitrous anhydride; Properties of — pure or in solution in nitrogen peroxide. G. Baume and M. Robert. Comptes rend., 1919, 169, 968—970.

A STUDY of the melting-point and vapour pressure diagrams of the system N₂O₄—N₂O gives the b. pt. of pure nitrous anhydride as -27° C. at 760 mm. It is not possible to distil it in a vacuum because of the immediate formation of an atmosphere of nitric oxide at temperatures at which distillation is practicable. (See further J. Chem. Soc., 1920, ii., 31.)—W. G.

Ferrieyanide and ferrocyanide; Estimation of —. I. M. Kolthoff. Pharm. Weekblad, 1919, 56, 1618—1626.

FERRICYANIDE can be readily estimated iodometrically in acid solutions, and accurate results are obtained. Rupp's method for the estimation of ferrocyanide in which an excess of iodine and sodium acetate or sodium bicarbonate are added to the ferrocyanide solution, and the excess iodine titrated with thiosulphate after one hour (this J., 1902, 1099) gives incorrect results. The author gives a theoretical interpretation of the iodometric estimation of ferri- and ferrocyanide. (See also this J., 1918, 122 A.)—W. J. W.

Lithium silicate. K. A. Vesterberg. Medd. K. Vetenskapakad. Nobel-Inst., 1919, 5, No. 30, 1—9.

AMORPHOUS silicic acid, which has been dried at 100° C., dissolves slowly in a fairly concentrated solution (approx. 2N) of lithium hydroxide at

ordinary temperatures, giving finally a solution containing 3.4 mols. SiO_2 to 1 mol. Li_2O . Thus lithium behaves similarly to potassium and sodium in giving soluble silicates containing a large excess of acid over the base (compare *J. Chem. Soc.*, Feb., 1920). In warm lithium hydroxide solution, however, silicic acid is almost insoluble, the acid being transformed into a practically insoluble lithium silicate. Lithium metasilicate occurs in two modifications, the one easily soluble and the other almost insoluble. The latter, which has the formula $\text{Li}_2\text{SiO}_3 \cdot \text{H}_2\text{O}$, is obtained as a white granular precipitate when a fairly concentrated solution of sodium metasilicate is mixed with the equivalent quantity of lithium chloride, lithium hydroxide added in approximately $N/1$ concentration, and the solution heated for a short time at 80° – 90° C. It can also be obtained by dissolving silicic acid, which has been dried at a temperature not higher than 100° C., in twice the theoretical amount of $2N$ lithium hydroxide solution at ordinary temperature, and then heating the solution for a short time at 80° C.—T. S. P.

Radioactivity of uranium. C. Staehling. *Comptes rend.*, 1919, 169, 1036–1039.

OXIDES of uranium prepared from uranyl nitrate or ammonium uranate, after suitable preliminary purification, showed a lowering in their non-penetrating activity over the period Jan., 1913, to Nov., 1919, equal to from 1 to 30.7%, the curves indicating that half the lowering took place in 6.2 months. A black-green oxide, which showed a lowering from 5.95 to 4.64%, was dissolved in nitric acid and the nitrate again calcined giving the black oxide; the activity of the latter was 5.90%, and the conversion of this black oxide into the green oxide through ammonium uranate did not appreciably lower its restored activity. Renewing the surfaces of the discs carrying the test specimens did not modify the activities observed.—W. G.

Zirconium monoxide; Question of the existence of —. R. Schwarz and H. Deisler. *Ber.*, 1919, 52, 1896–1903.

THE black powder obtained by reducing zirconium dioxide with magnesium does not contain a monoxide, but is a mixture of free metal and dioxide. The more sudden the reduction (firing by thermite) the greater is the proportion of free metal in the product. (See further *J. Chem. Soc.*, 1920, ii., 42.)—J. C. W.

Chlorine and hydrogen; Interaction of —. Influence of mass. D. L. Chapman and J. R. H. Whiston. *Chem. Soc. Trans.*, 1919, 115, 1264–1269.

BODENSTEIN and Dux have concluded from their experiments (*Z. physik. Chem.*, 1913, 85, 297) that the reaction is of the second order, the velocity being proportional to the square of the concentration of the chlorine. The present authors cannot confirm this result, for they find that, within wide limits of concentration of the interacting gases, the rate of formation of hydrogen chloride is given by the expression $\frac{k \cdot I \cdot [\text{Cl}]}{[\text{O}]}$ where k is a constant, and I the intensity of the radiation. The rate of interaction with moist gases is found to be almost independent of the pressure, instead of proportional to the pressure, as it would be if the result of Bodenstein and Dux were correct. The results here recorded are in agreement with the theory of the reaction put forward by Chapman and his co-workers (this *J.*, 1909, 1196; 1913, 424).—E. H. R.

Silicon hydrides. VII Protosiloxane. A. Stock and K. Somieski. *Ber.*, 1919, 52, 1851–1860.

COMPOUNDS of the formula SiH_2Hal_2 react with water to form polymerides of protosiloxane. The

unimolecular form, $\text{O} \cdot \text{SiH}_2$, is obtained as a gas by the interaction of water vapour and the dichloride in a large flask under reduced pressure, but the tendency to polymerise is so great that the apparatus must be clean and smooth. On condensation, the gas polymerises at once, to benzene-like liquids, soluble in benzene, $(\text{SiOH})_n$ etc., and insoluble solids. All polymerides react with sodium hydroxide to give hydrogen and sodium silicate. (See further, *J. Chem. Soc.*, 1920, ii., 31.)—J. C. W.

Diamond; Experiments on the artificial production of —. C. A. Parsons. *Phil. Trans.*, 1919, A 220, 67–107.

BAKERIAN Lecture, 1918, containing a full account of work already described before the Institute of Metals (this *J.*, 1918, 300 A).

Zirconium ore. Powell and Schoeller. See X.

Electrochemical industry. Goldschmidt. See XI.

Phosphoric acid. Tuinzing. See XXIII.

Determination of zinc. Houben. See XXIII.

PATENTS.

Sulphuric acid; Manufacture of —. H. Royal-Dawson, Huddersfield. Eng. Pat. 135,359, 4.1.19. (Appl. 265/19.)

To facilitate the horizontal movement of gases in sulphuric acid chambers, columns of packing material are fixed in the chambers, the surfaces being kept wet by water sprays or steam jets. The chamber top may contain sunken channels or reservoirs for the feed acid, with inlets to the chambers immediately above the baffle columns.—W. J. W.

Ammonia; Oxidation of —. H. E. F. Goold-Adams, London, J. R. Partington, Lostock Grahm, and E. K. Rideal, London. Eng. Pat. 135,224, 7.7.17. (Appl. 9853/17.)

A CATALYST for the oxidation of ammonia consists of nickel or "other non-platinum metal" in the form of gauze or perforated plate, which may be covered with a layer of nickel oxide or other catalytic substance.—W. J. W.

Nitric acid; Continuous concentration of dilute —. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 297,901, 13.4.16.

A MIXTURE of concentrated sulphuric acid and dilute nitric acid flows down a tower packed with acid-resisting material, such as glass, quartz, or the like, and passes into an externally heated still or retort. An outlet siphon tube is attached to the retort at a suitable height, e.g., half way up the side. The mixture of acids is so adjusted that nitric acid of the required strength distils over, and about 70% sulphuric acid flows out of the retort.—L. A. C.

Nitrous sulphuric acid; Denitration of waste —. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 297,902, 11.7.16.

WASTE nitrous vitriol is concentrated to about 56° B. (sp. gr. 1.635); the greater part of the nitric oxide is then removed by passing a stream of air through the hot acid, and the last traces are eliminated by treating a stream of the acid with a counter-current of sulphur dioxide.—L. A. C.

Nitric acid; Manufacture of highly concentrated — from dilute nitric acid. Deutsche Gasglühlicht-A.-G. (Auerger.), Berlin. Ger. Pat. 299,681, 1.12.16.

NITRIC acid of less than 69% strength is concentrated to above that strength by distillation with

approximately 78% sulphuric acid; the partially concentrated acid is then distilled in an apparatus fitted with a fractionating column to produce an acid of high concentration. Complete separation of the dilute nitric acid from the mixed acids remaining in the still is effected in a separate apparatus, and both the dilute nitric acid thus obtained and the sulphuric acid, after concentration to about 78% strength, are returned to the vessel in which the first stage of the concentration takes place.—L. A. C.

Ammonia; Method of purifying gases intended for synthetic production of —. L'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude, Paris. Eng. Pat. 129,637, 4.10.18. (Appl. 16, 986/19.) Int. Conv., 15.2.18.

Is the purification by means of heated palladium-asbestos of mixtures of nitrogen and hydrogen intended for the synthesis of ammonia under very high pressures (500—2000 atm.; see Eng. Pat. 130,086; this J., 1919, 718A), the same "hyper-pressure" is used for eliminating the water formed in the purification process. Two temperature exchangers are employed, one of which brings the gases to the temperature of combination in the presence of the palladium-asbestos, while the other cools the gases to a temperature near the freezing point of water.—W. J. W.

Potassium chloride; Manufacture of —. E. E. and P. C. Dutt, Jubbulpore. Eng. Pat. 135,246, 18.10.18. (Appl. 17,080/18.)

A MIXTURE of potassium fluoride, silica, and carbon is heated in a rotary furnace and treated with gaseous hydrogen chloride. The mass is lixiviated and the solution evaporated. The silicon tetrafluoride evolved during the reaction passes into a chamber containing felspar heated to 500°—600° C., a stream of water vapour being introduced into the chamber. The mixture of potassium fluoride, silica, and aluminium silicate thus produced serves as the starting point for the process. (See also Eng. Pats. 124,554 and 133,474; this J., 1919, 323A, 901A.) —W. J. W.

Potassium sulphate; Manufacture of —. E. E. and P. C. Dutt, Jubbulpore, India. Eng. Pat. 135,545, 18.10.18. (Appl. 17,079/18.)

A MIXTURE of potassium fluoride and clay or silica is heated in a rotary furnace into which sulphur dioxide is introduced. The product is lixiviated and the solution evaporated to dryness. The silicon tetrafluoride evolved in the furnace passes into a chamber containing felspar heated to 500°—600° C., into which a stream of water vapour is also passed. The mixture of potassium fluoride, aluminium silicate, and silica thus produced serves as the starting-point for the process. (See also Eng. Pat. 124,554; this J., 1919, 323A.)—W. J. W.

Ammonium sulphate; Washing —. F. E. Dodge, Flushing, N.Y., Assignor to The Barrett Co. U.S. Pat. 1,321,993, 18.11.19. Appl., 19.6.19.

THE surplus liquid is removed from the crystals by whirling in a perforated centrifugal basket. By means of a three-way valve, liquid can be delivered from a storage tank to a secondary tank, and thence to the centrifugal basket, so that each batch of crystals may be washed with an equal pre-determined quantity of saturated ammonium sulphate solution. A uniform product is thus obtained. —D. F. T.

Greensand; Utilisation of —. E. Hart, Easton, Pa. U.S. Pat. 1,322,900, 25.11.19. Appl., 8.1.17.

THE glauconite fraction of greensand is separated and treated with a strong mineral acid.

—W. E. F. P.

Greensand, marl, and glauconite; Treatment of — [for extraction of potassium compounds]. E. Hart, Easton, Pa. U.S. Pat. 1,323,228, 25.11.19. Appl., 5.6.18.

THE material is treated to produce a solution containing the chlorides of all the metals present except iron. Calcium and aluminium are then precipitated and removed from the solution separately, and the filtrate is evaporated to concentrate the potassium compounds.—W. E. F. P.

Potassium and aluminium compounds; Recovery of — [from greensand]. M. Hauber, jun., West Haverstraw, N.Y. U.S. Pat. 1,323,764, 2.12.19. Appl., 15.7.19.

AN intimate mixture of greensand and ferrous sulphate is heated to convert the greater part of the aluminium and potassium present into sulphates.

—W. E. F. P.

Potassium-bearing silicates; Method of treating —. W. Glaeser, Brooklyn, Assignor to Potash Extraction Corporation, New York. U.S. Pat. 1,323,464, 2.12.19. Appl., 8.3.15.

THE silicate is heated to low redness and suddenly cooled, then powdered and mixed with powdered pyrites. The mixture is burnt in the presence of air and steam at a temperature above 900° C.

—W. E. F. P.

Hydrogen peroxide; Process of making —. D. Levin and L. A. Molin, Buffalo, N.Y., Assignors to Commercial Electrolytic Corporation, New York. U.S. Pat. 1,323,075, 25.11.19. Appl., 5.4.16. Renewed 2.5.19.

HYDROGEN peroxide is obtained from solutions of persulphates by distilling them over heated metallic surfaces resistant to sulphuric acid, a rapid rate of flow of the solution being maintained.—W. J. W.

Manganese dioxide; Purifying —. J. S. Gravely, Assignor to Winchester Repeating Arms Co., New Haven, Conn. U.S. Pat. 1,323,690, 2.12.19. Appl., 14.6.19.

MANGANESE dioxide containing lower oxides is purified by treatment with a solution of a peroxide which removes the water-soluble impurities.

—W. J. W.

Sulphides; Process of oxidation of —. G. N. Libby, Berkeley, Cal. U.S. Pat. 1,323,879, 2.2.19. Appl., 3.2.16.

INSOLUBLE sulphides are oxidised by the electrolysis of alkaline solutions in which they serve as anodes.

—W. J. W.

Sulphur; Method for the preparation of oxygen compounds of — from natural sulphates, especially calcium sulphate. Badische Anilin und Soda Fabrik, Ludwigshafen. Ger. Pat. 298,491, 28.11.16.

NATURAL sulphates are first converted into ammonium sulphate, which is then mixed with anhydrous sodium sulphate and the mixture subjected first to a moderate and finally to a dull red heat. At 400° C. ammonia is driven off and sodium pyrosulphate is formed. As the temperature rises the latter compound is decomposed into sodium sulphate and sulphur trioxide.—T. St.

Chlorine or sulphuryl chloride and sulphates; Method of preparation of —. F. Bergius, Essen. Ger. Pat. 299,031, 30.11.13.

SULPHUR dioxide and air or oxygen are caused to come in contact with a chloride, which must be in a finely divided state, on the counter-current principle, the temperature of the reaction being con-

trolled by the velocity of the gaseous mixture. The reaction is exothermic and if the gas current is properly adjusted, no external heating is required to complete the reaction. The temperature must be maintained between 500° C. and the melting-point of the chloride-sulphate mixture, the upper limit in the earlier stages being 623° C., the melting-point of the eutectic. An excess of sulphur dioxide leads to the formation of sulphuryl chloride.—T. St.

Sulphur dioxide; Purification of the gases containing — produced by calcining alkali bisulphates. Akt.-Ges. Dynamit Nobel, Vienna. Ger. Pat. 299,035, 19.9.16. Int. Conv., 13.9.16.

SULPHUR dioxide of a high degree of purity may be produced by scrubbing the gases generated in the calcination of sodium bisulphate with a saturated solution of sodium bisulphate. The solution, which gradually decreases in strength owing to absorption of water from the gases, is again brought to saturation by the addition of the solid bisulphate.

—L. A. C.

Nitric oxide; Manufacture of — from ammonia and air. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 299,643, 12.1.17.

IN the manufacture of nitric oxide the addition of 2–6 vols. of water vapour to a mixture of 8 vols. of ammonia and 92 vols. of air increases both the yield of nitric oxide and the output of the apparatus.

—L. A. C.

Aluminium silicates; Treatment of naturally occurring —. W. Borchers, Aachen. Ger. Pat. 300,092, 10.2.16.

CLAY and similar substances are mixed with reducing substances, and a quantity of sulphates of alkaline-earths, preferably of magnesium and calcium, sufficient to produce neutral silicates, and the mixture is heated to 900°–1300° C. with admission of steam if necessary. The sulphate sulphur is thus almost entirely driven off as sulphur dioxide, and an alkaline-earth silicate is formed. The alumina can be removed from the mass as sodium aluminate either by roasting with sodium carbonate or by heating with an aqueous solution of sodium hydroxide under pressure. As reducing material may be used sawdust, spent tan and dye-woods, tar and petroleum residues, water-gas, etc.—T. St.

Sodium alum; Production of crude — from clay or kaolin and sodium bisulphate. R. Linkmeyer, Barby. Ger. Pat. 301,611, 7.12.16.

CRUDE sodium alum is produced by treating clay or the like with sodium bisulphate containing approximately 1 mol. of water to 1 mol. of sodium bisulphate; under these conditions the greater part of the aluminium present in the clay combines with the sodium bisulphate, and when the reaction is complete a dry product is obtained which may be readily removed from the vessel.—L. A. C.

Sulphur compounds in gases; Process for precipitating —. Apparate-Vertriebs-Ges. m.b.H., Berlin-Wilmersdorf. Ger. Pat. 309,159, 26.8.16.

SULPHUR dioxide or hydrogen sulphide is introduced, according to whether the gas contains the one or the other of these constituents, and the reaction takes place in the presence of a liquid catalyst in an apparatus resembling a disintegrator, in which the liquid catalyst is atomised into particles of colloid-chemical dimensions. In contact with the impalpable particles of the liquid mist the reaction between hydrogen sulphide and sulphur dioxide takes place so rapidly that a gas containing only small quantities of these impurities is practically completely de-sulphurised. Suitable catalysts are oxides, hydroxides, sulphides, and salts.

—J. F. B.

Phosphates; Production of soluble — from crude phosphates. Chem. Fabr. Budenheim L. Utz m.b.H., Mainz. Ger. Pat. 313,964, 13.2.17.

CRUDE phosphates are treated with an acid, and the requisite amount of ammonia or an alkali base or carbonate is added to the product without previous filtration. The mass is then filtered and heated for the production of soluble phosphates. The preparation of primary ammonium phosphate is described as an example.—L. A. C.

Hydrogen; Method of manufacturing —. Badische Anilin und Soda Fabrik, Ludwigshafen. Ger. Pat. 300,032, 8.4.14. Addition to Ger. Pat. 279,582.

IN the manufacture of hydrogen from carbon monoxide and steam in the presence of a catalyst (Fr. Pat. 459,918 of 1913; this J., 1914, 24), where the latter consists of such forms of metals of the iron group (particularly iron, nickel, cobalt), or their oxides, as are only slightly active, hydroxides or salts of the metal in question are added to increase the activity of the catalyst. For example, iron oxide, which in its preparation was subjected to a high temperature, is formed into a contact mass with damp iron hydroxide; or fine-mesh iron wire gauze, conveniently in the form of a roll, is once or more times acted upon by a concentrated solution of iron nitrate, and then subjected to a moderate heat until the nitrate is completely decomposed. In all cases such contact masses show a considerably higher activity at, say, 500° C., than the original materials.—T. St.

Hydrogen; Regulation of water supply in apparatus for producing and compressing —. F. Lang, Frankenthal. Ger. Pat. 314,260, 24.9.16.

THE air-free cooling water from the compressors used in apparatus for compressing hydrogen is employed for cooling and scrubbing fresh supplies of the gas. Contamination of the hydrogen with the gases present in fresh water is thereby avoided and consumption of water is reduced.—L. A. C.

Nitric acid; Process for concentrating — and apparatus for use in connection therewith. E. C. R. Marks, London. From E. I. du Pont de Nemours and Co., Wilmington, Del., U.S.A. Eng. Pat. 135,535, 6.9.18. (Appl. 14,522/18.)

SEE U.S. Pat. 1,292,948 of 1919; this J., 1919, 285 A.

Sulphuric acid; Method for the concentration of —. A. Dryen, Assignor to Soc. Anon. Fabr. de Soie Artificielle de Tubize, Tubize, Belgium. U.S. Pat. 1,323,847, 2.12.19. Appl., 25.6.15.

SEE Eng. Pat. 10,386 of 1915; this J., 1916, 537.

Potassium; Recovery of — in the form of nitrate from the waste gases of cement kilns and the like. H. Fairbrother, London. From Chemical Construction Co., Los Angeles, Cal., U.S.A. Eng. Pat. 135,713, 20.2.19. (Appl. 4255/19.)

SEE U.S. Pat. 1,296,460 of 1919; this J., 1919, 362 A.

Potassium compounds; Process of recovering —. H. Fairbrother, London. From Chemical Construction Co., Los Angeles, Cal., U.S.A. Eng. Pat. 135,720, 24.2.19. (Appl. 4563/19.)

SEE U.S. Pat. 1,296,463 of 1919; this J., 1919, 362 A.

Ammonium sulphate; Method and apparatus for producing —. H. A. Carpenter, Sewickley, Assignor to Ritter-Conley Manufacturing Co., Pittsburgh, Pa. U.S. Pats. 1,322,757 and 1,322,758, 25.11.19. Appl., 15.2.16 and 18.7.18.

SEE Eng. Pat. 104,155 of 1916; this J., 1917, 548.

Strontium peroxide; Process for manufacture of —. J. B. Pierce, jun., Charleston, W. Va. U.S. Pat. 1,325,043, 16.12.19. Appl., 3.6.18

SEE Eng. Pat. 130,840 of 1918; this J., 1919, 719 A.

[*Nitrogen-potassium-carbon compounds.*] U.S. Pat. 1,322,038. See X.

Fullers' earth. Ger. Pat. 305,152. See XII.

[*Chrome*] *leather waste.* Eng. Pat. 115,421. See XV.

Purifying liquids. U.S. Pats. 1,323,239, 1,323,251, and 1,323,256. See XIXu.

Gas mixtures. Ger. Pat. 303,986. See XXIII.

VIII.—GLASS; CERAMICS.

Egyptian bricks. G. R. Thompson. Analyst, 1919, 44, 403—405.

BRICKS from the Land of Goshen of undoubted Jewish origin and probably manufactured between 1580 and 1220 B.C., contained no trace of vegetable matter, especially of the nature of straw or wood fibre. The chemical composition of the bricks agreed closely with that of dried Nile mud. The author is of the opinion that straw or stubble was not an integral part of the bricks, and that it was used simply as a non-adherent material to prevent the bricks sticking to the ground during drying or to the workers' hands during moulding. The lumps of mud or the sides of the mould would be covered with chopped straw for this purpose. (See also this J., 1919, 439 B.)—W. P. S.

Anti-dimming compositions. Carleton. See XIXu.

Anti-dimming preparations. Holmes and others. See XIXu.

PATENTS.

Glass-melting furnaces; Gas-fired —. S. G. Curd, Queenborough, Kent. Eng. Pat. 135,636, 5.5.19. (Appl. 20,306/18.)

Is a regenerative gas-heated furnace for melting glass the producer gas is enriched prior to combustion by bringing it into contact with a mixture of iron and carbon, or superheated steam may be so treated and then mixed with the gas. A reservoir may be provided adjacent to the melting furnace, separated from it by a division wall having two cylindrical holes near its base which permit the glass to flow to the reservoir. A honeycombed partition may be mounted upon the division wall to act as a radiator of heat, and a second radiator may take the form of a longitudinal partition in the tank. The upper part of this partition may be honeycombed to permit a partial cross flow of the hot gases. The bottom of the tank and reservoir may be of curved cross-section and the roof of the tank may contain a niche-shaped head in which is the charging hole.—A. B. S.

[*China*] *clay and the like; Mining or quarrying* —. The Osmosis Co., Ltd., and J. S. Highfield, London. Eng. Pat. 135,277, 19.11.18. (Appl. 18,919/18.)

A SMALL quantity (0.05—0.20 %) of an alkaline electrolyte, such as caustic soda or sodium silicate or carbonate, is added to the water used for mining the clay or in the settling pit. The amount of clay which can be retained in suspension is thereby increased from about 5 to 25—50 %, and the amount of liquid to be pumped out of the pit is correspondingly reduced.—A. B. S.

Glass; Machinery for the manufacture of sheet —. A. L. Forster, West Smethwick. Eng. Pat. 135,697, 28.1.19. (Appl. 2017/19.)

Glass cane and glass tubing; Apparatus for drawing —. C. H. Quackenbush and J. C. Smedley, Cleveland, Ohio, Assignors to General Electric Co. U.S. Pat. 1,325,265, 16.12.19. Appl., 29.4.16. SEE Eng. Pat. 105,285 of 1916; this J., 1917, 551.

Clay; Treatment of —. W. Feldenheimer, London. U.S. Pat. 1,324,958, 16.12.19. Appl., 27.11.18. SEE Eng. Pat. 121,191 of 1917; this J., 1919, 41 A.

IX.—BUILDING MATERIALS.

PATENTS.

Mortar, concrete, and like material; Mixing of —. C. Candlot, Paris. Eng. Pat. 128,536, 15.11.18. (Appl. 18,778/18.) Int. Conv., 17.6.18.

THE raw materials for concrete, mortar, and the like are discharged in suitable proportions from separate hoppers, through a common pipe, into a hydrating apparatus and thence into a rotating cylinder, wherein they are further mixed without the addition of any more water.—A. B. S.

Concrete aggregates and materials of a similar nature; Apparatus for mixing together various materials for the dry mixing of —. T. Lea, Wolverhampton. Eng. Pat. 135,511, 16.4.19. (Appl. 16,857/18.)

A MIXING apparatus for dry concrete aggregates and the like consists essentially of two cones or pyramids, one above the other, the higher one, which may be rotated mechanically, having its apex projecting upwards under a feed outlet and its base within the second or inverted hollow cone or pyramid. The materials to be mixed fall on the upper cone or pyramid and pass down its surface, then through the lower cone or pyramid, and through an outlet in the bottom of the latter. —A. B. S.

Portland cement reinforced with organic fibres; Manufacture of plates of —. N. V. Hollandseho Asbest-Cement Fabriek, Schiedam, Holland. Eng. Pat. 130,312, 12.4.19. (Appl. 9393/19.) Int. Conv., 20.7.18.

FIREPROOF and waterproof plates or blocks which can be sawn, bored, nailed, cut, and planed are made of Portland cement reinforced with vegetable fibres, such as wood cellulose, or straw cellulose, which have previously been impregnated successively with a solution or suspension of a fluosilicate and with an alkali silicate in a milling engine or autoclave and then dried.—A. B. S.

Portland cement; Apparatus for manufacture of —. *Utilisation of low-grade carboniferous material.* R. W. Lesley, Haverford, Pa. U.S. Pats. (A) 1,323,293 and (B) 1,323,294, 2.12.19. Appl., 3.9.18 and 21.1.19.

(A) A ROTARY kiln is connected to a retort heated by the waste gases from the kiln. Oil shale is distilled in the retort and the combustible gases are used as fuel for the kiln. The residue from the retort is then conveyed to the kiln and burned to form Portland cement. (B) The heat escaping from a rotary kiln in which Portland cement is burned is used to heat a retort containing low-grade carboniferous material; the gas from the latter is used to heat the kiln.—A. B. S.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron; Future development in the manufacture of puddled — J. E. Fletcher. Staffs. Iron and Steel Inst., 22.11.19. Engineering, 1919, 108, 804—806, 836—840.

HALL's process (1820), by promoting intimate contact of molten pig metal and liquid iron oxides, has been shown to be the basis of all open-hearth refining operations in the manufacture of steel and iron, and is not likely to be superseded. The small single-hearth furnace, operated by hand, remains the best means of producing a regular product, though the ordinary puddling furnace is wasteful and the labour and shingling costs are high. The coal consumption per ton of metal produced is twenty times as much in a puddling furnace as in the large open-hearth steel furnace, and this indicates the thermal advantage to be gained by increasing the size of the furnace. Possible improvements in the fire-grates are suggested by employing tubular grate-bars, perforated for air ejection, on which cast-iron discs may be rotated to remove clinker and to disturb the fire. The use of a basic brick capable of withstanding splashes of iron oxide would reduce the erosion losses. The application of gas firing to puddling furnaces has been suggested, and in a design due to Chantaine the furnace is provided with a double arch, the lower one being perforated for the admission of preheated air. Gas passes along the under side of the arch, and combustion is effected in a series of flame jets issuing from the arch and impinging on the metal in the hearth.—C. A. K.

Steels; Phenomena and experimental data in the hardening of — A. Portevin. Chim. et Ind., 1919, 2, 1139—1160.

A GENERAL review of the thermal transformations of carbon steels, together with the variation in mechanical, electrical, and structural characteristics of the metal. The composition of the steel, the quenching temperature, and the rate of cooling are the three principal factors, and the two latter are determined by a consideration of overheating and other defects, and by the mechanical properties desired in the metal after treatment. The necessity of control of all variables is emphasised, and to satisfy the different objectives the proper treatment of any particular steel must be studied independently. (See also this J., 1919, 179 A, 417 A, 418 A.)

—C. A. K.

Steel forgings; Physical defects in — A. Portevin. Publ. de la Rev. de Mét., 1918, 23 pages.

DEFECTS in steel forgings may arise during the casting of the ingot, during the forging or mechanical working stage, or during heat treatment. The different kinds of defects are considered in some detail, and the precautions generally adopted to prevent the occurrence of imperfections in the forged pieces are explained. The methods for detecting the presence of defects at different stages of the process are also indicated. The paper is illustrated with a large number of photographs and diagrams.—E. H. R.

Valve steels in internal-combustion engines; Valve failures and — L. Aitchison. Inst. Automobile Eng., 5.11.19. Engineering, 1919, 108, 799—802, 834—836.

VALVES in petrol engines are subjected in use to a temperature of 100°—600° C. (inlet) and 250°—860° C. (exhaust). The various types of failure are elongation of the stem, distortion of the head, cracks in the valve face, excessive wear of the stem or foot, burning of the head, scaling, and breaking

due to self-hardening. Among other properties steel for valves should possess great strength at relatively high temperatures, and give a satisfactory result under the notched bar test. It should have the capacity for being worked and heat-treated without developing cracks or excessive scale, and its physical properties should be retained after exposure to high temperatures. Experimental data on different classes of steel show that each class of steel, e.g., tungsten-steels, chromium-steels, is different in its properties at high temperatures, but that members of one type are similar. Steels having similar constitution, although belonging to different classes, are alike in mechanical properties. Variation in composition (e.g., in carbon) has a distinct effect on the properties of the steel when hot, though this effect is reduced by a larger proportion of alloying elements. Low-carbon high-chromium (stainless) steel is weaker than a steel with lower chromium and higher carbon content, or than 0.6% tungsten-steel, and nickel or nickel-chromium steels are quite weak at high temperatures compared with other steels. Vanadium has apparently little effect on the properties of tungsten steels when hot. Nickel-chromium steels are unsatisfactory, and high chromium steels (above 7% Cr) are the most resistant to scaling. Tungsten-steels begin to scale rapidly about 850° C. The following steels are recommended:—For inlet valves, 3% nickel steel (C 0.30—0.35, Ni 2.75—3.75, Mn 0.40—0.70%), quenched in oil from 830° C. and tempered at 625° C.; for exhaust valves with a working temperature of 600°—700° C., high chromium steel (C 0.65, Si 0.60, Mn 0.50, Cr 10.0%), hardened in air from 900° C. and tempered at 750° C.; for exhaust valves working above 760° C., tungsten-steel (C 0.65, W 16.00, Cr 3.75%), refined at 950° C., cooled in air, and tempered at 800° C.—C. A. K.

Zinc ores, residues, and ashes; Utilisation of low-grade — Wöbling. Metall u. Erz, 1919, 16, 420—422.

THE materials dealt with are poor calamine deposits, residues from ore washing, ashes and muffle fragments containing zinc. Proposed methods of treatment, including leaching and electrolytic processes, are reviewed, and it is concluded that the extraction of zinc from these low-grade materials is only possible if the cost of treatment can be considerably reduced. In laboratory experiments complete reduction has been obtained by means of charcoal.

—T. H. Bu.

Zinc retorts; Influence of the pressure in — on the output of metal. O. Mühlhæuser. Metall u. Erz, 1919, 16, 363—373.

THE author has previously shown the effect of difference of pressure between the retort and the combustion chamber on the movement of gas through the retort walls (this J., 1919, 725 A). In further tests the output of retorts kept artificially under pressure has been determined and compared with the results of ordinary practice. The permeability of muffles varies considerably. In damaged retorts the loss of metal increases with the pressure in a measure corresponding with the extent of the damage. In undamaged retorts the loss does not generally increase with the pressure. When the retort is not very permeable the metal loss may sometimes be reduced by opening the receiver. If a retort is damaged and the permeability of the muffle correspondingly high the gases of combustion will penetrate the vessel in large quantities in consequence of the excess pressure in the furnace, and correspondingly larger amounts of zinc vapour are expelled from the condenser, as indicated by the intense green colour of the condenser flame. Zinc furnaces should be always kept under the highest possible pressure. In retorts with cracks or

holes the loss of metal generally becomes excessive only if a resistance to the passage of the gases is formed in the throat of the muffle or the interior of the receiver, or if the plug sealing the throat of the receiver becomes too impervious by clogging up with zinc dust.—T. H. Bu.

Pyrophoric [zinc] alloys; Utilisation of German supplies of —. W. Guertler. *Z. Metallkunde*, 1919, 10, 209—213. *Chem.-Zeit.*, 1919, 43, Rep., 260.

The alloy consists of zinc with a few per cent. of copper and aluminium, and is available in large quantities. Different methods of separation are discussed. The alloy may be heated until it separates into solid and fluid components, pure zinc being isolated. Electrolytic refining gives pure copper and zinc, but has not yet been technically perfected. The distillation of zinc is at present too costly. The removal of aluminium and copper is possible by chemical methods; also by melting the alloy with zinc oxide to oxidise the aluminium and removing the copper by another method. The alloy is too brittle for most purposes. It might be useful for galvanising.—T. H. Bu.

Aluminium-copper-zinc; The ternary system — with special reference to the zinc corner of the equilibrium diagram. V. Jares. *Int. Zeits. Metallg.*, 1919, 10, 1—11. *Chem. Zentr.*, 1919, 90, 111., 512.

THE copper corner of this ternary system has already been worked out satisfactorily by Carpenter and Edwards (this J., 1912, 778), and the present work is confined to the zinc corner of the diagram. From the results of the thermal investigation of the alloys a space diagram has been constructed. For the examination of the mechanical properties of the alloys they were prepared from a commercial zinc containing 1% Pb. In the zinc-aluminium system the hardness of the slowly cooled samples increases very rapidly with the aluminium content up to 70 (Brinell) at 10% Al, remains constant up to about 20% Al, and then increases very slowly. In the zinc-copper system it reaches a maximum at about 5% Cu, then sinks to a minimum at 12—14% Cu, and again rises very quickly. The minimum of the Zn-Cu alloys also appears in the ternary alloys when these are cooled slowly, but with quick cooling it does not occur and the hardness increases more regularly. The tensile strength of the Zn-Al alloys tested was lower than that given by Portevin (this J., 1911, 1216) and Ewen and Turner (this J., 1910, 1163), probably owing to the lead content. The Cu-Zn alloys show a maximum tensile strength at about 10% Cu. The strength of the Zn-Al alloys increases fairly rapidly with increase of copper up to about 8—10% Cu and then falls. The fractured surface of zinc becomes finer with increase of Al, retaining its ray structure, but with addition of copper it quickly becomes fine-grained. An alloy with 90% Zn, 6% Cu, and 4% Al has a very high crushing strength, twice that of tin bearing metal. Al-CuZn alloys ("white brass") as hard and strong as brass or bronze can readily be obtained, but owing to their brittleness they are not efficient substitutes for these. For technical use white brass should not contain less than 4% nor more than 10% Cu. The aluminium content should not exceed 5%, and should be diminished as the copper content increases. The addition of certain metals (Pb, Fe, Mn, Ni) may slightly increase the hardness and strength, but the tenacity and elasticity are not improved. The Al-Cu-Zn alloys rich in zinc can be used as substitutes for bearing metals, although they are less soft and plastic. White brass is not suitable for use with steam, as it oxidises too readily, withstands only moderate pressures and is rapidly corroded

by many salt solutions, natural waters, etc. It can very well be used for mechanical and physical instruments.—E. H. R.

Detinning industry. L. Hackspill. *Chim. et Ind.*, 1919, 2, 1161—1167.

A REVIEW of the methods employed for the recovery of tin from tinned iron. Complete removal of tin should be effected so as to enable the iron to be used again, and this can never be accomplished by the process of brushing or centrifuging the molten tin coating. An interesting though not a commercial process of physical separation consists in cooling the tinned metal in liquid air, thus converting the tin into an allotropic modification which is easily detachable. In chemical methods for detinning iron an agent is used which will attack tin, and the resulting solution is treated for the recovery of the small quantities of iron compounds also formed. Thus, hydrochloric acid is used with formation of stannous chloride and small quantities of lead chloride and ferrous chloride. After precipitating lead by means of sulphuric acid metallic tin is obtained by treating the solution with zinc or aluminium or magnesium alloys. An ingenious method of reducing the tin salt by the residual iron, due to Schultze (*Eng. Pat.* 3669 of 1887, this J., 1888, 388; see also this J., 1890, 550), is very slow, and has not been employed on the large scale. Sodium hydroxide in presence of an oxidising agent, e.g., lead oxide or sodium nitrate, will dissolve tin with the formation of sodium stannate, without attacking the iron. The process applied to 75% of the metal treated depends on the reaction between chlorine and tin. This reaction is extremely rapid, and the principal difficulty lies in dissipating the heat evolved. In the recovery of tin by electrolytic means the tinned metal is employed as anode, with an insoluble cathode, in an electrolyte consisting of a solution of sodium hydroxide or sodium sulphide.—C. A. K.

Zirconium ore; Analysis of Brazilian —. A. R. Powell and W. R. Schoeller. *Analyst*, 1919, 44, 397—400.

FIVE grms. of the sample, ground to pass a 90-mesh sieve, is treated with a magnet to remove particles of iron introduced in the grinding and then dried at 100° C. for 1 hr.; the loss on ignition is determined on 1 gm. of the dry ore. A further 5 grms. of the sample is then ground under water in an agate mortar and the slime decanted from time to time into a porcelain basin; when but little sandy material is left, iron is removed with a magnet, the grinding finished, and the slime evaporated to dryness. About 0.5 gm. of the dry slime is ignited in a platinum basin to constant weight, then fused with 5 grms. of sodium carbonate for 1 hr., the melt dissolved in boiling water, and any insoluble residue collected on a filter. This insoluble matter is ignited, fused with potassium bisulphate, the melt treated with 1% sulphuric acid, and the still insoluble portion collected on a filter (filtrate A). The solution from the sodium carbonate fusion is meanwhile evaporated with the addition of hydrochloric acid and the silica separated and collected (filtrate B). The silica precipitate, together with the insoluble residue from the bisulphate fusion, is ignited, weighed, evaporated with hydrofluoric and sulphuric acids, the residue weighed, fused with bisulphate, and dissolved in 1% sulphuric acid. This solution is added to the two filtrates (A and B), the solution nearly neutralised with sodium carbonate, a slight excess of sulphuric acid is added together with a little pulped filter-paper, and the mixture is saturated with hydrogen sulphide. After 18 hrs. the platinum sulphide is collected and washed with 1% sodium chloride solution. The filtrate is nearly neutralised with sodium carbonate and boiled while a current of carbon dioxide is

passed through it to expel all hydrogen sulphide. The solution is then boiled for 1 hr. with the addition of 10 grms. of sodium thiosulphate, the precipitate collected, washed with boiling water, ignited and weighed; it consists of ZrO_2 , TiO_2 , and Al_2O_3 . To determine these constituents separately the ignited precipitate is fused for 1 hr. with 30 times its weight of sodium carbonate, the melt boiled with water and 2 grms. of sodium hydroxide, the solution filtered, and the insoluble portion washed with 2% sodium carbonate solution. The alkaline filtrate is poured into an excess of dilute nitric acid, the solution boiled to expel carbon dioxide, and the aluminium then precipitated as hydroxide by ammonia. The precipitate is ignited and weighed, then fused with bisulphate, dissolved in sulphuric acid, and tested with hydrogen peroxide; it sometimes contains a trace of titanium. The insoluble portion from the sodium carbonate fusion is ignited, fused with 5 grms. of bisulphate, the melt extracted with 1% sulphuric acid, the solution diluted to 200 c.c., and 20 c.c. is treated with 2 drops of hydrogen peroxide. The colour obtained is compared with that produced by a known amount of titanium under the same conditions. The amount of zirconia is found by difference. Iron, manganese, calcium, and magnesium are determined in the filtrate from the thiosulphate precipitate after the solution has been boiled with nitric acid and filtered to remove sulphur.

—W. P. S.

Electro-chemical industry. Goldschmidt. See XI.

Determination of zinc. Houben. See XXIII.

Separations in arsenic group. Strecker and Riedemann. See XXIII.

PATENTS.

Iron or steel articles; Hardening or like treatment of —. E. H. Jones, London. Eng. Pat. 134,864, 12.8.18. (Appl. 13,054/18.)

IRON or mild steel articles, turned to finished size, are plated with a metal having a fusion point near to or higher than that of iron, e.g., nickel, cobalt, vanadium, chromium. The plated articles, protected from oxidation, are then heated to and maintained at 1000° – 1200° C. for 8–4 hours until the metal coating penetrates the iron. It is necessary to protect a chromium or vanadium plating by a further deposit of a comparatively non-fugitive metal, e.g., nickel or iron. In many cases the articles may with advantage be subjected to a case-hardening process during the heating period.

—C. A. K.

Steel; Manufacture of —. *Casting iron or steel or their alloys.* F. E. Falvet, Paris. Eng. Pats. (A) 134,915, and (B) 134,916, 12.11.18. (Appls. 18,518 and 18,519/18.)

(A) The necessary proportions of iron, tungsten, vanadium, or other metals required for high-speed steels are heated together to about 1200° C., and 0.2–5% of ferro- or ferri-cyanide of potassium or sodium is added, together with slag-forming material. The temperature is then raised to 1800° C., and the metal cast, with or without a supplementary addition of ferro- or ferri-cyanide. (B) The addition of ferro- or ferri-cyanide as specified under (A) is applied to the production of castings of steel, iron, or their alloys.—C. A. K.

Pig-iron; Process of producing high-phosphorus — [and nitrogen-potassium-carbon compounds from greensand]. T. C. Meadows, New York. U.S. Pat. 1,322,038, 18.11.19. Appl., 4.5.18.

A MIXTURE of greensand, phosphate rock, and fuel is smelted in a blast furnace, molten high phosphorus pig-iron being tapped off, and nitrogen-

potassium-carbon compounds recovered from the furnaces gases, which are drawn off immediately, above the reaction zone.—C. A. K.

Steel; Product for carbonising —. T. R. Ferguson and E. L. Larson, Chicago, Ill., Assignors to A. O. Blach Co. U.S. Pat. 1,322,298, 18.11.19. Appl., 1.4.18.

A CASE-HARDENING compound for steel consists of charred pecan shells, free from moisture or oils.

—C. A. K.

Foundry and pig-iron; Method and shaft furnace for the synthetic production of high-grade —. Rombacher Hüttenwerk, J. I. Bronn, and W. Schemmann, Rombach. Ger. Pat. 299,662, 17.4.15.

MOLTEN refined iron of low carbon content is allowed to descend through a shaft furnace filled with coke which is maintained at a high temperature throughout by means either of a blast introduced through tuyeres or electrodes, or both, at different levels, whereby the iron is caused to take up carbon up to 3%, or more, according to the height of the coke column. By mixing small pieces of ferrosilicon or ferromanganese with the coke silicon or manganese may also be added to the iron.

—T. St.

Lead and tin; Recovery of — from their solutions. W. G. Rumbold, London. Eng. Pat. 135,052, 28.1.19. (Appl. 2132/19.)

A SOLUTION of lead (or tin) chloride in ferrous chloride solution, obtained by the known method of treating the metals with a solution of ferric chloride, is mixed with an excess of ferrous chloride in concentrated solution and treated with clean scrap iron at 60° – 80° C. The lead or tin is deposited on the iron, and the solution may be regenerated for the treatment of a further quantity of metal. A similar reaction for tin takes place from its sulphate solution in the presence of a concentrated solution of ferrous sulphate.—C. A. K.

Alloys rich in lead; Process of making —. J. P. Arend, Dommeldange, Luxembourg. Eng. Pat. 135,056, 31.1.19. (Appl. 2496/19.)

ALLOYS of copper, tin, zinc, and lead consist of a skeleton of the alloy of the higher melting metals, in which the lead is carried. Such alloys are usually not uniform in structure, particularly if such a metal as zinc is present in sufficient quantity to prevent the solidification of the skeleton alloy until considerable separation of the lead has occurred. By the present method the alloy mixture is melted in an induction furnace. The unalloyed lead particles offer a much higher electrical resistance than the particles of copper-tin or copper-zinc, and thus acquire a higher temperature and more rapid motion. The lead thus becomes disseminated and "emulsified," and during the solidification period the small particles readily adhere to the other metals and produce an alloy of more uniform structure.—C. A. K.

Electrolytic copper; Process for manufacturing pure — from cement copper. A. G. Sundberg and T. E. Thomasson, Helsingborg, Sweden. Eng. Pat. 135,125, 6.5.19. (Appl. 11,312/19.)

CEMENT copper is roasted, or oxidised by other means, and is dissolved in a quantity of acid electrolyte, withdrawn during the normal process of electrolysis with insoluble anodes, the solution then being returned to the electrolysis tank. The process is made continuous by withdrawing acid solution equivalent to the metallic copper deposited, an equal amount of copper then being dissolved by the acid solution. Impurities in the cement copper are prevented from passing into solution by the

excess of copper oxide present when the neutral stage of the extraction is reached.—C. A. K.

Crucible furnaces. W. A. Shilton and R. A. Munden, Coventry. Eng. Pat. 135,241, 17.10.18. (Appl. 16,937/18.)

THE crucible heating chamber is made in the form of a bell open at the bottom, and adapted to stand on the furnace base and enclose the crucible. It is heated either by gas or by hydrocarbons, the burners passing through lateral openings. The bell can be raised or lowered, and in the top is a central outlet for waste gases with inclined or rounded shoulders so as to deflect the flames and heat into the crucible.—J. W. D.

Crucible furnaces for melting aluminium and the like. C. F. Price, Birmingham. Eng. Pat. 135,247, 21.10.18. (Appl. 17,151/18.)

THE crucible, which can be tapped either at the top or bottom, fits into the top plate of the furnace, and is retained in an upright position by firebrick projections from the back and sides of the furnace lining, the bottom resting on an arch which spans the fire grate below. In the top plate are openings for charging fuel, and in the sides of the furnace a series of openings with removable blocks for admitting and regulating a supply of air. Below the outlet flue a baffle is provided between the crucible and the casing in order to deflect the heat round the crucible.—J. W. D.

Crucible and like furnaces. J. H. Ratson, Gateshead-on-Tyne. Eng. Pat. 135,447, 10.7.19. (Appl. 17,349/19.)

AT the bottom of the furnace a metal base-plate is arranged having bangers or depending brackets which support the fire-bars and are connected to or support perforated side plates through which air can enter the fire in addition to that entering from beneath the fire-bars. The firebrick casing or lining of the furnace is supported by this base-plate.—J. W. D.

Retort furnace for the recovery of zinc by the reduction of zinc oxide. H. Bursitzky, Laura-hütte. Ger. Pat. 314,131, 23.2.15.

ALL the retorts are connected with a common condensing chamber in the form of a shaft which extends downwards sufficiently for the temperature at the bottom to remain below the vaporising point of zinc, and is provided with an outlet for carbon monoxide at the top. The retorts are each provided with two projecting openings, one above, constituting a hopper for the continuous charging of the retort, and one in front for the periodical removal of ashes. The masonry surrounding the condensing chamber contains a sand-seal to prevent the penetration of air.—T. St.

Martin furnace; Operation of a tilting — using a mixture of low-grade and high-grade gas. Deutsch-Luxemburgische Bergwerks- und Hütten-A.-G., Abteil. Dortmunder Union, and A. Klinkenberg, Dortmund. Ger. Pat. 314,235, 4.7.18.

THE charge in a Martin furnace is first melted by the mixed gas under normal oxidising conditions; the supply of low-grade gas is then shut off, and by means of the high-grade gas the charge is heated to a higher temperature in an atmosphere as low in oxygen as possible, whereby a slug of high basicity is formed which takes up the phosphorus and sulphur from the charge. The slag is then removed by tilting the furnace. The carbon content of the charge is not lowered by the treatment, and chromium, nickel, tungsten and the like

are not taken up by the slag. By the addition of more lime and subsequent removal of the fresh slag the last traces of phosphorus and sulphur may be removed.—L. A. C.

Roasting furnace; Gas-fired —. Donnersmarckhütte, Oberschlesische Eisen- und Kohlenwerke A.-G., Hindenburg. Ger. Pat. 314,263, 17.7.18. Addition to Ger. Pat. 310,283 (this J., 1919, 641 A).

IN roasting ores which require heating to 300° C. or more to drive off water of constitution there is a danger, if ore dust has been allowed to cake on the plates, that when this temperature is reached the water vapour may escape with explosive violence, and thus interfere with the smooth working of the furnace. To prevent the formation of such caked dust on the plates in the high-temperature zone of the furnace these plates are made with perforations through which loose dust drops. This dust is received on parallel plates situated below, and is returned to the main stream of ore.—T. St.

Minerals; Flotation of —. C. L. Perkins, Pittsburgh, Pa., Assignor to Metals Recovery Co., New York. U.S. Pat. 1,322,816, 25.11.19. Appl., 2.7.19.

A SMALL amount of toluidine is added to the mineral pulp, and the mixture is subjected to a flotation operation.—S. S. A.

Casting molten metal. A. R. Earnshaw, Riverton, N.J. U.S. Pat. 1,323,583, 2.12.19. Appl., 12.6.18.

MOLTEN metal on the way from the ladle or other container to the mould is broken up into relatively small globules and subjected to a gaseous cooling medium. Both actions can be effected by means of a suitable jet of gas under pressure.—B. M. V.

Alloys; Method of separating the constituents of — by "blowing" the molten metal. J. E. Knudsen, Mühlbach. Ger. Pat. 299,631, 17.7.15. Int. Conv., 11.6.15.

COPPER alloys are melted with scrap iron and air is blown through the molten metal, the oxidation of the iron furnishing the necessary heat to keep the copper molten; whilst zinc distils and is recovered as oxide. The action is interrupted for the addition of quartz sand, which serves as a flux for the iron oxide formed. About 85% of the copper in brass is thus recoverable with a purity of 99%, and about 71% of the zinc. Bronze requires more iron than does brass, and the tin passes into the slag. Nickel bronze and German silver yield a copper-nickel alloy, and any precious metals present are concentrated in the copper or copper-nickel.—T. St.

Slags rich in iron; Producing — from lead and copper smelting. W. Buddens, Berlin. Ger. Pat. 302,537, 20.3.17.

SLAGS from lead and copper smelting are unsuitable for the blast furnace on account of the presence of zinc (10%), lead, and copper. Complete separation of the metallic impurities is effected if a small part of the iron in the slag is reduced to metallic iron, which quickly takes up the copper, the lead and zinc being driven off. The slag, with or without addition of material rich in iron, is mixed with fuel in a shaft furnace having two widely distant rows of tuyères and smelted with hot blast, so that at the level of the upper row the slag is quickly melted, and at the level of the lower row the iron

is partly reduced. The mixture of ferrous oxide slag and iron containing some copper flows into the fore-hearth or slag pot in which the iron is separated from the slag.—T. H. Bu.

Tungsten; Production of chemically pure — for incandescence bodies in electric lamps. Lohmann und Heckmann Metallfabr. Ges., Neukölln. Ger. Pat. 306,364, 31.1.15.

AMORPHOUS tungsten metal, tungstic anhydride or the like is transformed in a carbon tube resistance furnace into tungsten carbide. This is finely powdered and placed in a carbon mould which is introduced into a high temperature furnace fixed to a rotating wheel. The high temperature and the centrifugal force produce a homogeneous body of tungsten carbide. The latter is mixed with tungsten oxide or other material containing oxygen, and exposed for a long time to a temperature not reaching the melting point of tungsten. By this means the carbon is extracted from the carbide and pure workable tungsten metal free from pores is obtained.—T. H. Bu.

Fine-grained ores; Smelting — in a blast-furnace or the like. O. Baumann, Amberg. Ger. Pat. 312,935, 2.11.17.

THE fine ore is introduced into the furnace, together with finely-divided carbon and flux, by means of the air blast. To prevent premature slagging in the furnace and stoppage of the tuyères the ore is preferably reduced as far as possible before adding to the air blast, so that finely-divided iron generally containing gangue is blown into the blast-furnace. In the blast-furnace the usual charge of ore and coke is fed into the shaft and the fine dusty material is introduced through the tuyères with the blast. A considerable part of the waste gases is withdrawn from the furnace hearth, so that the direction of the blast is principally downwards and the melting zone lies below the tuyères. These gases are used in the ore reduction furnace. To obtain iron low in carbon the molten iron is withdrawn from contact with the coke and collected in a fore-hearth.—T. H. Bu.

Tin; Production of — free from iron, from waste tin-plate and the like. Chem. Fabr. Buckau, Magdeburg, and T. Silbermann, Halle. Ger. Pat. 314,592, 16.1.15.

WASTE tin-plate and the like is treated with an acid, and a stannous compound is precipitated from the solution obtained by the immediate addition of just sufficient alkali or alkaline-earth oxide, hydroxide, or carbonate to give a precipitate free from iron compounds.—L. A. C.

Metal articles; Hardening, or hardening and tempering, of needles, pins, spikes and like —. A. Imbery, Halifax. Eng. Pat. 135,640, 7.12.18. (Appl. 20,385/18.)

Iron and steel; Electrolytic method of cleaning —. Q. Marino, Hendon. U.S. Pat. 1,324,317, 9.12.19. Appl., 11.8.19.

SEE Eng. Pat. 129,489 of 1918; this J., 1919, 685 A.

Steel or ferro alloys; Manufacture of —. W. L. Turner, Atherstone. U.S. Pat. 1,325,455, 16.12.19. Appl., 1.10.18.

SEE Eng. Pat. 123,113 of 1917; this J., 1919, 224 A.

Electric metallurgical furnaces. W. E. Moore, Pittsburgh, Pa., U.S.A. Eng. Pat. 135,639, 7.12.18. (Appl. 20,378/18.)

SEE U.S. Pat. 1,293,161 of 1919; this J., 1919, 372 A.

Electro-converter furnace. P. F. Sarron and J. Simon, Lyon, France. U.S. Pat. 1,325,114, 16.12.19. Appl., 8.6.18.

SEE Eng. Pat. 11,094 of 1911; this J., 1914, 907.

Welding of iron or steel or alloys thereof; Means to be employed in the electric —. A. C. Hyde, Perivale. U.S. Pat. 1,323,768, 2.12.19. Appl., 8.7.19.

SEE Eng. Pat. 128,463 of 1918; this J., 1919, 640 A.

Precipitating materials. U.S. Pat. 1,321,985. See I.

Base-metal thermo-couple. Ger. Pat. 302,452. See XXIII.

XI.—ELECTRO-CHEMISTRY.

Electro-chemical industry [in Germany]; Development of —. H. Goldschmidt. Z. Ver. deutsch. Ing., 1919, 918. Chem.-Zeit., 1919, 43, Rep., 272. (See also this J., 1919, 951 A.)

FERRO-TUNGSTEN is now prepared containing 75—85% W and 0.5—0.8% C. A 50—60% product can be made by a continuous process, but the product of higher tungsten content is too viscous and must be withdrawn from the furnace intermittently. The world-production of tungsten rose from 72 tons in 1892 to 10,600 tons in 1913. In that year Germany imported 5000 tons of tungsten ore, and exported 2200 tons to England. During the war tungsten was recovered from tin slags, 220—230 tons being thus recovered in 1916. The price rose from M. 5.5 per kilo. in 1914 to M. 80 in 1915. Ferro-chromium has been manufactured in large quantities (7000 tons) during the war, but Germany does not control any supplies of chrome ore; 5000 tons of chromium was imported formerly from France, Switzerland, and Scandinavia. Ferro-silicon was imported (20,000 tons) from Switzerland and Scandinavia, but here also war-time developments will render this unnecessary in the future. To make 1000 kilos. of ferrosilicon (75% Si) 10,000 kilowatts is required. Phosphorus, fused quartz, carborundum, and graphite are all produced electrolytically. Artificial graphite is superior in some respects to the natural material, e.g., in electrical conductivity. Of the pre-war German consumption of copper (260,000 tons) the home production amounted to 40,000 tons. In America over 1,000,000 tons was made in 1918, i.e., more than the world's output before the war. There is also general over-production of chlorine, so that the prospects for German export trade are poor. In inorganic chemistry chlorine might replace sulphuric acid. Electrolytic hydrogen and oxygen have been produced in increased quantities, the latter being used with advantage for cutting steel. The prospects of oxygen manufacture are considered very favourable. The cheap water power so necessary for these electro-chemical industries is available in Bavaria.

—H. J. H.

Electrolysis of solutions of iron; Potential necessary for the —. W. A. Noyes. Comptes rend., 1919, 169, 971—973.

THE minimum potential necessary for the electrolysis of a solution of a ferrous salt between electrodes of electrolytic iron is 0.66 volt at 20° C. This value decreases 0.007 volt for 1° C. rise in temperature, attaining a minimum at 110° C. and then rising with further rise in temperature.

—W. G.

PATENTS.

Depolarising agent; Electrolytic higher oxide of manganese — and process of making same. Densified polarising agent and process of making same. C. Ellis, Montclair, N.J., Assignor to National Carbon Co., Inc., Cleveland, Ohio. U.S. Pats. (A) 1,322,000 and (B) 1,322,001, 18.11.19. Appl., 6.12.16.

(A) A SOLUTION of manganous sulphate and another sulphate is electrolysed, whereby a higher oxide of manganese is produced; this is removed without coming into contact with the nascent hydrogen evolved. (B) Finely-divided basic manganous material is roasted to form manganese dioxide, which is mixed with finely-divided graphite and a binding agent. The mixture is subjected to a pressure of several tons per sq. in., then pulverised and re-shaped in moulds at a lower pressure than was previously used.—J. H. J.

Insulating aluminium-covered copper conductors; Methods of —. H. M. and H. D. Abernethy, Cleveland, Ohio, Assignors to H. Fuller, East Cleveland, Ohio. U.S. Pat. 1,323,236, 2.12.19. Appl., 17.4.16.

AN electrical conductor covered with a thin coating of aluminium is electrolysed by a current of low voltage and amperage in an aqueous solution of potassium permanganate and sulphuric acid. The conductor serves as the anode, and the cathode is composed of a porous cup filled with mercury.—L. A. C.

Electric furnace. W. H. Bristol, Waterbury, and M. J. Johnson, Naugatuck, Conn., Assignors to The Bristol Co., Waterbury, Conn. U.S. Pat. 1,323,576, 2.12.19. Appl., 14.1.19.

IN a furnace of the resistance type the resistance material is placed in an opening hollowed out of the top of horizontal electrodes.—B. M. V.

Gases; Method of purifying or separating — by means of high-tension electricity. Siemens-Schuckertwerke G.m.b.H., Siemensstadt. Ger. Pat. 314,171, 27.2.18.

THE gas is made to pass over high-tension electrodes provided with points or edges through a conduit on the sides of which are placed plate-like collecting electrodes. The composition of the gas stream over its cross-section thus alters, the impurities tending to pass towards the walls of the conduit. Separation of the gas into parts containing lesser and greater amounts of impurity is then effected by means of wedge-shaped separating walls placed in the conduit beyond the electrodes.—T. St.

Oxidation of sulphides. U.S. Pat. 1,323,879. See VII.

XII.—FATS; OILS; WAXES.

Tobacco seed oil. K. Preissecker and H. Brezina. Fachl. Mitt. Oesterr. Tabakregie, 1917, No. 4. Chem.-Zeit, 1919, 43, Rep., 288.

EXPERIMENTS on a large scale were made in 1915 on the extraction of Dalmatian tobacco seeds with trichloroethylene. The average yield was 35.4% of oil having the following characters:—Sp. gr. 0.9250 at 15° C.; acid value, 9.5; saponif. value, 196.4; and iodine value, 131.6. The oil-cake contained a relatively high proportion of nitrogen and fat; it would probably be suitable for fodder.—C. A. M.

Cnicus Benedictus oil. A. Ferencz. Arch. Pharm., 1919, 257, 180—190.

THE oil of *Cnicus Benedictus* seeds is odourless and tasteless, that pressed cold being pale yellow and

that pressed hot dark brown; both oils exhibit semi-drying properties. The physical and chemical characters are as follows:—Sp. gr. at 15° C., 0.9262; acid value, 1.2; refractive index, 1.47178; saponification value, 191; iodine value, 141; Hehner value, 95.75; Reichert-Meißl value, 2.53; Polenske value 0.60; unsaponifiable matter, 0.66; iodine value of unsaturated fatty acids, 146. The unsaturated fatty acids consist of about 71% of oleic and 26% of linolic acids, and the saturated fatty acids of about 40% of stearic and 60% of palmitic acids. According to Pater (Természettudományi Közlöny, 1917, 675—676) the seeds contain 24.1—28.3% of fatty oil, 14.83% being obtainable by pressing in the cold.—T. H. P.

Beeswax; Influence of cholesterol esters on the saponification of —. P. Böhrisch. Pharm. Zentralh., 1919, 60, 473—478.

A SAMPLE of East African beeswax examined by the author contained 0.61% of cholesterol esters and a sample of East Indian beeswax 0.22%. Since ordinary (German) beeswax contains 0.6—0.7% of these esters, the difficulty with which the foreign waxes saponify cannot be due to their presence.—W. P. S.

Colloidal electrolytes: Soap solutions as a type. J. W. McBain, M. E. Laing, and A. F. Tittley. Chem. Soc. Trans., 1919, 115, 1279—1300.

THE study of the electrical conductivity and other properties of soap solutions has led one of the authors to define a new class of compounds, colloidal electrolytes, of which soap solutions are a type (McBain, this J., 1918, 219 r). Conductivity and freezing point data are given for a number of sodium and potassium salts of fatty acids, including acetic, octoic, decoic, lauric, oleic, and stearic acids. The striking point about the conductivity curves is that they represent moderately good conductivities even in concentrated solutions. In many cases the conductivity curve, after passing through a minimum, rises on further concentration to a maximum, an effect which is more pronounced at lower than at higher temperatures, and more for potassium than for sodium salts. The soap solutions also show an abnormally high temperature coefficient of conductivity. The osmotic properties of soap solutions were best studied by the freezing point method. From the data obtained for the lowering of the freezing point it was possible to calculate the concentration of crystalloid in the solutions. It was found that, in all but the most dilute solutions, the soap exists almost entirely as colloid. In high dilution and especially at higher temperatures, e.g., 90° C., the soap is almost entirely crystalloid. The octoate shows properties intermediate between those of an acetate and a true soap. The phenomena are well explained by the hypothesis of ionic micelle, according to which the negative ions of the soap are aggregated to form the nucleus of a colloidal particle, termed the ionic micelle, whilst retaining their equivalent electric charges. Thus the ionic micelle exhibits conductivity as well as mobility even greater than the ions contained in it. The micelle probably combines with non-ionised molecules and with water of hydration. The change in hydration with increasing temperature explains the abnormal temperature coefficient of conductivity.—E. H. R.

Soap curd; Degree of hydration of the particles which form the structural basis of — determined in experiments on salting out. J. W. McBain and M. Taylor. Chem. Soc. Trans., 1919, 115, 1300—1308.

WHEN a soap, for instance, sodium palmitate, is salted out by sodium hydroxide, marked apparent negative sorption occurs, since the concentration of alkali in the lye becomes much higher than before.

By analysis of the soap curd and of the lye it is shown that the curd is a mechanical mixture of hydrate (or sorption compound) and enmeshed lye. The degree of hydration in the case of sodium palmitate varies with the concentration of the lye. Thus, when the concentration is 3*N* the sorption compound has the composition $\text{NaP} \cdot 3\text{H}_2\text{O}$, and for 1.5*N* the composition is $\text{NaP} \cdot 6.5\text{H}_2\text{O}$, where *P* represents the palmitic acid radical. Under extremely high pressures the degree of hydration is lowered considerably.—E. H. R.

Oiliness and lubrication. Deeley. See I.

Althaea root. Von Friedrichs. See XX.

PATENTS.

Oils and fats and the like; Deodorising and purifying —. G. Calvert, Twickenham Park. Eng. Pat. 135,295, 21.11.18. (Appl. 19,126/18.)

OILS, fats, etc., are heated in contact with a finely divided substance or substances and a non-oxidising non-flavouring gas, such as hydrogen, nitrogen, or carbon dioxide, or a non-aqueous vapour, such as petroleum spirit, ethyl acetate, or a mixture of these. The contact material may either be of natural occurrence, e.g. kieselguhr, or it may be prepared artificially, e.g., hydrated aluminium silicate deposited on kieselguhr, and be capable of acting as a carrier for objectionable substances not removed by the gas or vapour, whilst being itself left in a condition suitable for separation from the oil or fat. In one modification of the process the fatty acids or glycerides are sprayed into a chamber, where they are mechanically agitated in contact with the material and in the current of gas. Various forms of apparatus for carrying out the process are described.—C. A. M.

Fuller's earth [for oil refining]; Process for the production of —. J. Kohndorfer, Landshut. Ger. Pat. 305,452, 3.6.14.

FINELY powdered silicate rocks or silicious earths are treated with dilute sulphuric acid at 80°–100° C. The hot mixture is neutralised with ammonia, and the precipitate is separated from the liquid which can be further worked up for ammonium sulphate.—J. H. J.

Oils and fats containing phosphoric acid; Process for the production of —. Chem. Fab. Flörsheim H. Noerdlinger, Flörsheim. Ger. Pat. 313,617, 23.11.17.

PHOSPHORIC anhydride is dissolved in the cold in castor oil or other hydroxy-acid oil and after a short time a tough glassy mass forms, from which by repeated washing and heating with water an oil is obtained soluble in ammonia and soda. The oil derived from castor oil has the following characters: sp. gr. at 15° C. 1.0133, acid value 192.3, saponification value 278.2; P_2O_5 content, 8.81%. The products are soluble in ether and petroleum spirit and insoluble in water and benzene. On heating with hydrochloric acid the phosphoric acid is not completely separated. If the oils are mixed with less than the quantity of alkali required for saponification and heated to the boiling point a gelatinous or solid soap is obtained, neutral in reaction and giving a clear solution in water.—J. H. J.

Pieric acid stains on the skin; Preparation of a reagent for the removal of —. J. Klemenz, Dresden. Ger. Pat. 312,772, 1.6.18.

A SUITABLE detergent, such as water glass, Glauber's salt, or soap, is mixed with bleaching powder.

—J. H. J.

Fatty acids from wood tar. Ger. Pat. 314,358. See III.

Detergents. Eng. Pat. 135,240. See V.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Illumination from paint. H. A. Gardner. Circ. 76, Paint Manufacturers' Assoc., U.S.A., Nov., 1919. 8 pp.

The light-reflecting power of a number of paints and pigments was determined by applying three coats of the paint under test to pieces of sheet tin about 6 in. square, primed with flat lithopone paint. The reflection factor was determined in an integrating sphere in which a beam of light was thrown on the samples at an angle of 15° to the normal. A freshly surfaced block of magnesium carbonate having a reflection factor of 88 was taken as the standard. Examination of some commercial paints showed that flat paints gave higher reflection values than gloss paints, most probably on account of the higher pigment content. Aluminium enamel gave a reflection factor of 52, as against 76 for flat interior wall paint, a surprising result in view of the generally accepted view as to the high light-reflecting power of the former. Tests on various paints compounded on both a weight and a volume relationship showed conclusively the higher reflecting values obtained by increasing proportions of pigment. Paints of high reflecting power can be made by using a relatively small proportion of highly viscous binding medium thinned down with volatile solvent.—A. de W.

Resins; Determination of the saponification value of dark —. H. Salvaterra. Chem.-Zeit., 1919, 43, 765–766.

FOUR grms. of the resin is boiled for 30 mins. beneath a reflux condenser, with 50 c.c. of *N*/2 alcoholic potassium hydroxide solution, then treated with 32 c.c. of *N*/2 barium chloride solution and 300 c.c. of recently boiled water, and allowed to stand on the water-bath for three-quarters to one hour. The liquid is then cooled, diluted to a definite volume (500 c.c.), and filtered, and an aliquot portion of the filtrate (100 c.c.) titrated with *N*/2 hydrochloric acid (phenolphthalein as indicator). In some cases the insoluble barium resinate coagulates at the bottom, leaving a clear supernatant liquid which can be titrated without filtration. Simultaneously, a blank test is made and titrated under the same conditions, to make allowance for the possible action of atmospheric carbon dioxide upon the barium hydroxide during the filtration.—C. A. M.

Turpentine; Halogen absorption of —. E. M. Taylor. Analyst, 1919, 44, 401–402.

WHEN iodine (Wijs') solution is used absorption of halogen by turpentine is never complete (see also this J., 1902, 1439; 1904, 302). The author has studied the reaction, using a solution of iodine tribromide in chloroform. In one set of experiments the turpentine was treated with an excess of the reagent for varying lengths of time, in another set varying amounts of reagent were used and the excess of iodine titrated after one minute's contact. The results obtained indicate that the reaction between turpentine and halogen is according to the equation $3\text{C}_{10}\text{H}_{16} + 4\text{IBr}_3 = 3\text{C}_{10}\text{H}_{16}\text{Br}_4 + 2\text{I}_2$, and that there is no intermediate reaction. This reaction can be realised with practically no secondary reaction by adding 10 c.c. of *N*/1 IBr₃ solution in chloroform to 0.1–0.15 gm. of turpentine dissolved in 10 c.c. of chloroform, adding potassium iodide solution within 1 min., and titrating the excess of iodine immediately with thiosulphate solution.

—W. P. S.

Use of inks in histology. Bugnon. See XXIII.

PATENTS.

Resins; Method of hardening synthetic — R. P. Jackson, Edgewood Park, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,323,284, 2.12.19. Appl., 3.2.16.

A PHENOL-ALDEHYDE condensation product is applied to a porous article and partially hardened by heating; the product is then dried at a lower temperature, and, after applying a further quantity of the condensation product, the hardening process is completed by further heating at a higher temperature.—L. A. C.

Coating with bakelite. Ger. Pats. 301,319, 305,179, and 307,699. See I.

Acids from wood tar. Ger. Pat. 314,358. See III.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Hevea latex; Natural coagulation of — in the absence of air. W. Spoon. Comm. Central Rubber Stat., Buitenzorg, 1919, No. 16, 313—337.

Is a comparison of the product of natural coagulation of undiluted latex in the absence of air with that obtained by coagulation of 15% latex with acetic acid, no practical difference was observable in the inner qualities of the rubber, although the former exhibited somewhat less variation in rate of cure. Factors which affect the rate of cure of rubber prepared by the ordinary method of coagulation, *e.g.*, prolongation of the coagulation period, alteration of the coagulation temperature, and resting of the trees, similarly influence the product of spontaneous coagulation. The addition of small quantities of sugar to the latex to facilitate natural coagulation has no appreciable effect on the rubber. A temperature of 40° C. gives a more satisfactory coagulation process than the 50°—55° C. mentioned by Barrowcliff (this J., 1918, 50 r). The porosity of the natural coagulum somewhat aids crêping, but the crêped material dries more slowly than crêpe from acetic acid coagulum.—D. F. T.

Rubber; Effect of soaking the rolled or unrolled coagulum in water on the properties of the — O. de Vries. Comm. Centr. Rubber Stat., Buitenzorg, 1919, No. 17, 339—373.

THE general effect of soaking the coagulum is an extraction of serum substances causing a loss in weight of 0.2—0.4% for crêpe, 0.5—3% for sheet, and 0.2—2% for unrolled coagulum; this involves a decrease in the rate of vulcanisation. Further soaking causes a "maturing" effect with continued loss in weight, but with increase in rate of vulcanisation, so that the original retardation is diminished or changed into an acceleration. The accelerators formed during "maturing" are only partly extracted by water. Soaking freshly-rolled sheet rubber does not prevent subsequent development of "rustiness," but is of considerable effect in preventing greasiness (due to hygroscopic substances on the surface of the sheet) and in decreasing the liability to develop mouldiness.—D. F. T.

Rubber; Cause of rustiness in sheet — H. J. Hellendoorn. Comm. Central Rubber Stat., Buitenzorg, 1919, No. 18, 119—136.

"RUSTINESS" on the surface of sheet rubber is not caused by the drying of a film of serum substances but is due to the decomposition of serum substances by an aerobic micro-organism; it may be induced by keeping freshly rolled sheets in a moist atmosphere for one or two days, the most favourable temperature being about 40° C. Smoking reduces the tendency to "rust," and the trouble may be inhibited

by treating the surface of the freshly rolled sheet with a disinfectant such as a solution of formalin or bisulphite; the simplest method of prevention, however, is to ensure rapid drying of the surface of the rubber.—D. F. T.

Caoutchouc; Bromination and constitution of — W. C. Schmitz. Gummi-Zeit., 1919, 34, 167—169, 193—195.

THE various and somewhat contradictory results which have been recorded as to the behaviour of rubber towards bromine are probably to be attributed to the interference of colloidal phenomena with the purely chemical stoichiometrical process. In order to avoid this disturbing influence the author has studied the bromination of rubber after a preliminary "depolymerisation" by heat. After the removal of the resinous constituents by prolonged extraction with acetone, the rubber was heated under 15 atm. pressure in an autoclave for 10 hours with xylene of b. pt. 130°—160° C.; the resulting mobile solution readily yielded its insoluble protein and other constituents when centrifuged; the xylene was then removed by distillation and after dissolving in carbon tetrachloride the rubber was submitted to the action of a solution of bromine in the same liquid. The consumption of bromine was in the proportion 5Br:C₂₀H₁₂; in view, therefore, of the addition of a molecule of bromine at each double bond, the molecular weight of the depolymerised hydrocarbon must be at least C₂₀H₁₂, whilst that of the original rubber is presumably much greater. In the course of several hours the reaction liquid developed hydrogen bromide due to elimination of this substance from the soluble primary additive compound, C₂₀H₁₂Br₁₀, with formation of a micro-crystalline insoluble compound, C₂₀H₁₂Br₄; the former is probably an open-chain compound, whilst the latter is possibly cyclic, but with smaller rings than the original rubber, and is quite distinct from the ordinary "caoutchouc tetrabromide." Whether the opening of the cyclic caoutchouc molecule occurs during bromination or is effected already in the depolymerisation process it is at present not possible to decide. Depolymerised rubber is therefore regarded as undergoing bromination as a simple chemical process without any complications due to colloidal phenomena. The results of the investigation are in accord with Harries' more recent views of the constitution of the rubber molecule (this J., 1915, 670).—D. F. T.

Vulcanisation coefficient; Effect of organic accelerators on the — D. F. Cranor. India-rubber J., 1919, 58, 1199—1205.

A MIXTURE of smoked sheet rubber (100), sulphur (6), and zinc oxide (1), when vulcanised over a range of periods between 85 minutes and 235 minutes at 292° F. (144° C.) or 45 lb. steam pressure in a platen press gave a maximum tensile strength with the 190 minutes period, the corresponding coefficient of vulcanisation being 4.76; after an accelerated "ageing" test, however, the product of 130 minutes vulcanisation and coefficient 2.85 had the maximum strength. The latter product is therefore regarded as the "best cured" sample. Other mixtures were then tested, derived from the above by the addition of 0.5 to 1% of an organic accelerator such as hexamethylenetetramine and dimethylammonium dimethyldithiocarbamate. With the former catalyst a vulcanisation coefficient of 2.83 was attained in 50 minutes, whilst the corresponding tensile strength and resistance to stretch were much greater than in the catalyst-free mixing. With the latter accelerator satisfactory physical properties, much superior to those of the "blank" mixing, were obtained after 3, 4, and 5 minutes, the corresponding vulcanisation co-

efficients being 1.09, 1.21, and 1.45. With the unaccelerated mixture, to obtain a product showing 700% elongation at a load of 1000 lb., the presence of about 4.9% of combined sulphur was necessary; with the hexamethylenetetramine and dimethylammonium dimethyldithiocarbamate mixings the corresponding coefficients necessary were 1.8% and 0.9%. Indeed, distinct signs of vulcanisation were visible after the last-mentioned mixture had been merely stored in the dark for a month at the ordinary temperature. The results suggest that the shorter the necessary period of heating for vulcanisation the lower the proportion of combined sulphur required to produce the desired physical condition. Comparable indications were obtained with similar mixings prepared with an inferior brown plantation crêpe, which indeed could not be vulcanised satisfactorily without an accelerator.—D. F. T.

PATENTS.

Rubber; Process for devulcanising —. Devulcanising process and product thereof. Process for devulcanising vulcanised rubber. C. F. Willard, San Diego, Cal. U.S. Pat. (a) 1,322,077, (b) 1,322,151, and (c) 1,322,152, 18.11.19. Appl., (a) 31.5.17, (b) 18.3.18, and (c) 5.12.18.

(a) VULCANISED rubber is boiled with a liquid consisting largely of a viscid, gummy hydrocarbon; this forms a froth on the surface of the liquid and on account of its greater affinity for sulphur under the prevailing conditions, removes combined sulphur from the rubber. (b) Vulcanised rubber is boiled with a viscous mixture of a tar and a flux of a liquid hydrocarbon in the presence of water. (c) More or less of the combined sulphur of vulcanised rubber is liberated by boiling the rubber with an emulsoid colloid solution and a detergent solution.—D. F. T.

XV.—LEATHER; BONE; HORN; GLUE.

Viscosity. Rothlin. See XXIII.

PATENTS.

[Chrome] leather waste; Process for the utilisation of —. C. Bennert, Grünau, Germany. Eng. Pat. 115,421, 9.4.18. (Appl. 6050/18.) Int. Conv., 30.4.17.

CHROME-LEATHER cuttings are freed from materials and colours used in finishing the leather by treatment at 35° C. with a dilute alkaline solution, agitation with sand, ground pumice or the like, and repeated boiling with water, containing if necessary oxidising or reducing bleaching agents, and are then heated to 75°–80° C. for 2–3 hours with a solution containing 15 parts of sodium hydroxide in 500 parts of water for each 100 parts of the cuttings. After cooling and standing for some time, the mixture is filtered and the liquor is made neutral to phenolphthalein by the addition of acetic or formic acid, and is concentrated to a syrupy consistence. The product contains protalbinic acid, lysalbinic acid, and other similar substances formed by hydrolysis of the leather which possess the property of yielding silver salts capable of keeping silver, silver oxide or hydroxide in colloidal solution, and may be used in cleaning, fulling, and dyeing textile fabrics. Chromium compounds may be recovered from the residue obtained on filtration of the liquor.—L. A. C.

Tanning leather for belts. F. Gilardini, Turin, Italy. U.S. Pat. 1,321,730, 11.11.19. Appl., 21.5.18. (See also Eng. Pat. 114,631; this J., 1919, 297 A.)

PORTIONS of the unprepared hide are subjected to heated compressing elements, and the hide is then treated with a tanning solution.—E. W. L.

Tanning compound. B. Levin, Hale. U.S. Pat. 1,328,878, 2.12.19. Appl., 28.5.18.

SEE Eng. Pat. 117,693 of 1917; this J., 1918, 579 A.

Treating coal gas etc. Eng. Pat. 135,577. See IIA.

Willow or poplar bark. Ger. Pat. 300,644. See V.

Food from scrap leather. Ger. Pat. 314,323. See XIXA.

XVI.—SOILS; FERTILISERS.

Soil analysis. F. Münster. Landw. Versuchs-Stat., 1919, 94, 181–189.

THE following method of extraction is recommended:—300 grms. of the soil is mixed with 900 c.c. of concentrated hydrochloric acid, and the mixture is shaken every hour for 48 hours; the clear liquid is then decanted, filtered, and 300 c.c. of the filtrate is evaporated, with the addition of ammonium chloride. The residue is moistened with nitric acid, hydrochloric acid is added, and the mixture is again evaporated; this operation is repeated, the residue then dissolved in hydrochloric acid and water, the silica collected on a filter, and the filtrate used for the determination of iron, alumina, calcium, magnesium, potassium, and phosphoric acid.—W. P. S.

Soil-sorption. E. Ramann and A. Spengel. Landw. Versuchs-Stat., 1918, 92, 127–146. (See also J. Chem. Soc., 1919, i., 615.)

THE results of experiments in which an artificial zeolite (permutite, hydrated aluminium alkali silicate) was treated with solutions of neutral potassium, ammonium, calcium, and sodium salts, showed that the interchange of bases which takes place has the character of a chemical exchange; no signs of physical adsorption could be detected. Potassium and ammonium are mutually replaceable, and displace sodium and calcium completely from the zeolite, whereas the displacement of potassium and ammonium by sodium and calcium is incomplete. The ratios between the bases in the solutions and in the zeolite are different; bases present in small proportions in the solution are combined by the zeolite in relatively greater amounts. Within wide limits the absolute concentrations of the salts in the solution are without appreciable influence on the composition of the zeolite, this being the case even with mixtures of calcium salts with those of univalent metals.

—T. H. P.

Copper; Content of — in cultivated soils. L. Maquenne and E. Demoussy. Comptes rend., 1919, 169, 937–942.

THE amount of copper brought into solution by boiling 6 grms. of the soil for half an hour with 50 c.c. of 10% sulphuric acid has been estimated in a number of ordinary arable soils and a number of vineyard soils. With the arable soils the copper content varied from 1 to 50 mgrms. per kilo. of fine soil, but in some of the vineyard soils it rose to 250 mgrms. Despite these high figures, there has not been any indication of a toxic effect of the copper on the vines. Most of the copper is in the surface layer of the soil, the penetration being very slow.

—W. G.

Calcium carbide content of crude calcium cyanamide. V. Maly. Z. Landw. Versuchsw. in Oesterreich, 1916, 19, 445. Bied Zentr., 1919, 48, 369–370.

ALL the samples of crude calcium cyanamide intended for agricultural use which were examined showed a calcium carbide content of under 0.24%.

The carbide content of crude calcium cyanamide filled into sacks was at first 2.7%; it fell after one day to 2.1%, and on the third day was 0.2%. Pipes of varying width and length, closed at one end, were filled with crude calcium cyanamide containing 2.7% of carbide. On the 29th day the carbide content at the closed end of a 15 cm. pipe had sunk to 0.66%, and in a 45 cm. pipe to 1.67%. In both cases the pipes were 3.5 cm. wide, and at the open ends there was found 0.014% of carbide. In layers of material which are not easily accessible to air, the decomposition goes on only very slowly.

—J. H. J.

Calcium cyanamide; Influence of storage on the nitrogen losses and nitrogen changes occurring in crude —. D. Meyer and R. Gorkow. *Illustr. Landw. Zeit.*, 1919, 39, 27—28. *Bied. Zentr.*, 1919, 48, 370—373.

CRUDE calcium cyanamide was spread in a layer 1 cm. thick on glass plates, some of which were kept in a room the temperature of which varied from 7° to 24° C. from winter to summer, and the remainder in a cellar at 6°—18° C. Increases in weight took place after 18—25 weeks' storage of 21—26% during summer and 22% during winter in the room and of 63—65% during summer and 69% during winter in the cellar. Hardly any loss in total nitrogen took place during storage, but there was a considerable decrease in cyanamide nitrogen and an increase in dicyanodiamide nitrogen and in other forms of nitrogen. This was especially marked in the samples stored in the cellar during summer, when about 50% of the cyanamide was converted into dicyanodiamide. It is concluded that calcium cyanamide should be stored in a dry, closed room, and if loose it should be covered with sacking and straw. Paper bags of the material should be stored close together, the spaces being filled with more material and the whole covered with straw.

—J. H. J.

Calcium cyanamide and ammonium sulphate; Influence of iron oxide and common salt upon the fertilising action of —. O. Lemmermann and A. Einecke. *Mitt. Deut. Landw.-Ges.*, 1918, 33, 574. *Bied. Zentr.*, 1919, 48, 290—292.

FIELD experiments were made during two years with the usual fertilisers, to which were added calcium cyanamide and iron oxide, but the addition of the iron oxide failed to produce any better crop than the calcium cyanamide alone. Sodium chloride caused no increased crop when used with calcium cyanamide and ammonium sulphate respectively. Calcium cyanamide containing 10% of soda produced no better results than the ordinary variety, and both were inferior to saltpetre. The crops used were rye, mangolds, and turnips.

—J. H. J.

Peat; Experiments on the action of various fertilisers made from —. B. Tacke. *Mitt. Ver. Förderung der Moor-Kultur im Deut. Reiche*, 1918, 36, 369—373. *Bied. Zentr.*, 1919, 48, 289—290.

For these experiments a special fertiliser was used made from peat and containing ferric salts. Its analysis was: mineral matter 10%, nitrogen 1.5%, lime 12%, potash 0.6%, phosphate 0.5%. Another fertiliser used was humus silicic acid, which had the following composition: mineral matter 30%, nitrogen 0.9%, lime 1.5%, potash 0.3%, phosphate 0.2%. Pot cultures of oats in sand were made, to which were added potassium phosphate, calcium carbonate, and one of the special fertilisers. The crops obtained were poor, as also were those obtained in peat cultures. The peat fertilisers did not appear to have provided any nitrogen for the use of the plants.—J. H. J.

Phosphoric acid; Rate of solution in water containing carbon dioxide of the — present in basic slag and other phosphates. J. G. Maschhaupt Versl. Landbouwk. Onderzoek. Rijkslandbouwproefstat., 1919, 23, Sept. *Chem. Zentr.*, 1919, 90, IV., 866.

THE rate of solution of various phosphates was determined by continuous extraction at 30° C. with water containing carbon dioxide, with a view to testing the theory that the value of a phosphate as a manure depends primarily upon the rapidity with which the compounds taken up by the plants from the moisture of the soil are replaced in solution. The results obtained were somewhat irregular, for example, the rate of solution of Florida phosphate was much greater than that expected, and Algerian phosphate dissolved more rapidly than basic slag.—L. A. C.

Ammonium polysulphide wash. Eyre and others. See XIXb.

PATENTS.

Organic material; Process of reinforcing, bacterising, and composting — for use as inoculators, improvers, and fertiliser of soils. T. F. Manns, Newark, Del. U.S. Pat. 1,320,701, 4.11.19. Appl., 13.10.15.

PROTEIN, carbohydrate, and phosphate are mixed with peaty material, and sufficient of a base is added to neutralise the mass. The product is inoculated with azotobacter, *B. radicola*, nitrobacter, and *B. rossica*, and is then made into a compost.—J. H. J.

Fertiliser. L. Phillip, Redlands, Cal. U.S. Pat. 1,322,817, 25.11.19. Appl., 17.6.19.

A FERTILISER comprising ashes of almond rinds. —S. S. A.

XVII.—SUGARS; STARCHES; GUMS.

Frothing of boiling liquids, foam separators, and juice-traps in sugar factories. H. Claassen. *Zentr. Zuckerind.*, 1919, 27, 500—502. *Chem. Zentr.*, 1919, 90, IV., 871—872.

THE particles of liquid escaping from the evaporating pans may exist either as small or large droplets suspended in the vapours or as froth, and the type of trap required to catch them must be differently designed according to their nature. According to whether the bubbles at the surface of the boiling liquid immediately disappear or persist for some time, a distinction is made between non-frothing and frothing liquids. In the case of non-frothing liquids the drops can be trapped simply by providing in the upright evaporators a vapour space 3—4 m. in height, the efficiency of which is increased by one or two baffle plates suitably placed about half-way up. The physical properties of liquids which are the cause of foaming are not well known. Bundles of suitably disposed steam-heated tubes, through which the stream of vapour is led, may constitute a very effective foam destroyer. Less effective is the use of a steam jet in the upper part of the evaporator. A special kind of frothing takes place at the beginning of the boiling; this may be largely prevented by destroying the initial froth by the addition of some fat and then increasing the evaporation by the admission of more heating steam, whereby a stronger circulation of the de-aerated and no longer superheated liquor is induced. Froth separators are apt to be ineffective in cases where frothing is caused by a sudden increase in the vacuum, and it is better in such cases to endeavour to arrive at an arrangement which will ensure more uniform conditions of pressure in

the evaporator plant. Effective froth separators will cause a very considerable loss of heat efficiency (up to 14%), and they should only be installed in cases where they are really necessary.—J. F. B.

Cane sugar; Construction of an autocalculus [nomon] or chart for the indication of commercial —. W. E. Appleby. Chem. Eng. and Min. Rev., 1919, 11, 173–178; 12, 33–38.

THE author anticipated Deming (this J., 1918, 217A) by several years in the principle of the nomon. In 1898 he elaborated a "sucrograph" for the rapid calculation of the results of sucrose determinations by the double polarisation method, whilst, later, he devised graphs for a number of other routine calculations in the factory and laboratory. Instructions are given for the construction of "autocalculus" charts relating to the determination of the "commercial cane sugar" in cane. This value serves for the purchase of cane in Queensland, the method recently prescribed by the Government of that country being as follows:—

$$\begin{aligned} \text{Total soluble solids} \times \frac{100 - (3 + \text{fibre})}{100} &= \text{total soluble solids in the cane.} \\ \text{Sucrose in the juice} \times \frac{100 - (5 + \text{fibre})}{100} &= \text{sucrose in the cane-juice.} \\ \text{Total soluble solids} - \text{sucrose in the cane} &= \text{impurities in the cane.} \\ \text{Sucrose in the cane} - \text{impurities} &= \text{"commercial cane sugar."} \end{aligned}$$

Total soluble solids are found by the Brix hydrometer reading; sucrose by direct polarisation; and the fibre by the diffusion method. The graphic indication of results according to the nomon or autocalculus principle cannot compare in minute accuracy with correct arithmetical or algebraic computation, and the results are neither so exact nor obtained as rapidly as those given by a modern calculating machine. The method is nevertheless of sufficient accuracy for a number of routine calculations.—J. P. O.

Mannitol; Oxidation of — by nitrous fumes. E. Votocék and C. Kranz. Z. Zuckerind. Böhm., 1919, 43, 577–580.

AQUEOUS solutions of mannitol are slowly oxidised by nitrous fumes, yielding a mixture of mannose and hexulose; reaction can be accelerated by the addition of small quantities of a ferrous salt. Nitrous fumes in this case behave like other oxidising agents, yielding a mixture of ketonic and aldehydic products.—H. W.

Starch; Systematic ageing experiments with solutions of various kinds of — under exact time conditions: Time law of the ageing of starch solutions. H. Sallinger. Kolloid-Zeits., 1919, 25, 111–115.

THE ageing of starch solutions at 7° C. has been followed by precipitating the starch gel at various times by means of ptyalin and weighing it after drying at 110° C. If G is the weight of dry gel and t the time of ageing in hours, $G = g t^a$, g and a being characteristic constants for the different kinds of starch. Some values obtained for the constants are shown in the following table:—

	g	a
Soluble potato starch I.	4.06	0.445
" " " " II.	3.00	0.455
Soluble wheat starch	5.37	0.165
Soluble barley starch	11.17	0.214
Amylodextrin from soluble potato starch	3.51	0.521

—J. F. S.

Viscosity. Rothlin. See XXIII.

PATENTS.

Sugar beet; New edible product [syrup] and process of obtaining same. P. Kestner, Paris. Eng. Pat. 135,235, 14.9.18. (Appl. 14,969/18.)

A SYRUP free from any unpleasant odour or flavour is obtained from beet juice by treatment in an evaporator (preferably of the climbing film type) in which the temperature is raised by setting up a counter-pressure. If, for example, the desired concentration of the syrup be such that its boiling-point under atmospheric pressure would be 120° C., a counter-pressure is provided sufficient to raise the boiling point to 130° C., but it is essential that the syrup shall not be maintained at this degree for more than a few seconds.—J. P. O.

Sugar solutions; Process for continuous defecation of —. G. B. Williamson, Gramercy, La., Assignor to Industrial Apparatus Corporation, Brooklyn, N.Y. U.S. Pat. 1,317,607, 30.9.19. Appl., 30.4.18.

SUGAR solutions are treated with an acid, neutralised with lime, drained off, aerated, heated to cause the impurities to rise to the surface, and then drawn off from the bottom. (See also Coates, this J., 1919, 872A.)—J. H. L.

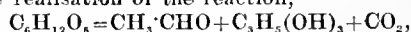
[Beet] diffusion battery. J. Masin, Prague. Ger. Pat. 314,200, 19.4.18. Addition to Ger. Pat. 313,433 (this J., 1919, 958A).

IN the middle of each of the vessels of the battery described in the chief patent, in place of the partition, there is a vertical tube, the lower end of which reaches the bottom, while the upper part bends over into the next vessel. Each of these tubes is provided with an injector by means of which the beet slices are forced from one vessel to the next, while the extract is made to pass in the contrary direction from vessel to vessel through suitable overflow tubes. To prevent an excess of juice being carried over with the slices, the bend of each tube is perforated and is surrounded by a jacket connected with the vessel by a return tube. —J. P. O.

XVIII.—FERMENTATION INDUSTRIES.

Alcoholic fermentation; Correlative formation of acetaldehyde and glycerol by the scission of sugar, and new contributions to the theory of —. C. Neuberg and E. Reinfurth. Ber., 1919, 52, 1677–1703.

IN outline, Neuberg's theory is as follows. Sugar is converted into methylglyoxal, which may be hydrated and reduced to glycerol or oxidised to pyruvic acid, this breaking down into carbon dioxide and acetaldehyde, which is reduced to alcohol. In the presence of sulphites, the best being calcium sulphite, as is now shown, the acetaldehyde is fixed, and the available hydrogen works in the direction of the production of glycerol. In fact, practically equimolecular proportions of aldehyde and glycerol are produced, and in the more concentrated solutions the quantitative realisation of the reaction,



has been achieved as well as might be expected of such a biochemical process. This evidence, therefore, strongly supports the theory. (See further, J. Chem. Soc., 1920, i., 124.)—J. C. W.

Alcoholic fermentation; Course of — in presence of calcium carbonate. J. Kerb. Ber., 1919, 52, 1795–1800.

ALCOHOLIC fermentation by pure cultures of yeast proceeds quite normally in the presence of calcium carbonate. The yield of alcohol is practically the

same, and no calcium pyruvate is formed, whereas Fernbach and Schön (this J., 1914, 97, 707) reported the production of a very large quantity of this salt. Probably they used a very impure ferment, and were, therefore, not really studying alcoholic fermentation at all.—J. C. W.

Yeast; Utilisation of amides by —. P. Thomas. Ann. Inst. Pasteur, 1919, 33, 777—806.

UNDER suitable conditions, yeasts are capable of utilising urea and, to a lesser extent, acetamide and its immediate homologues as sources of nitrogen. (See further, J. Chem. Soc., 1920, i, 126.) —W. G.

Diastatic properties of formaldehyde. G. Woker and H. Maggi. Ber., 1919, 52, 1594—1604.

A RECAPITULATION of the evidence on which the authors base their view that formaldehyde resembles diastase in its action on starch (this J., 1916, 1268). Recent criticisms (this J., 1919, 433 A, 508 A) are met by the assumption that the opponents of the theory kept their mixtures too long, for the authors believe that the hydrolytic products are in time rebuilt into non-hydrolysable and non-reducing substances, such as the so-called "reversion dextrin," just as when egg-albumen is digested with papain there is a reversion of the process after a time.—J. C. W.

[Yeast] *Saccharomyces thermantilonum*. H. Euler and I. Laurin. Biochem. Zeits., 1919, 97, 156—170.

THE inversion capacity, the catalase activity, the rate of fermentation at 35°—40° C., and the growth of *S. thermantilonum* have been studied. After the tests the culture showed certain deviations in its behaviour at the characteristic temperatures from the original culture. It is suggested that adaptation may be responsible for this.

—S. S. Z.

Glycerol; Influence of the presence of trimethyleneglycol on the estimation of [fermentation] — by Zeisel's isopropyl iodide method. C. A. Rojahn. Ber., 1919, 52, 1454—1460.

ZEISEL'S method (this J., 1902, 992) is not applicable to the estimation of fermentation glycerol (the German "protol"-glycerol), for this contains trimethyleneglycol, which develops di-iodopropane. This passes over more or less completely with the isopropyl iodide, although its boiling-point is much above the reaction temperature (120°—125° C.).

—J. C. W.

Tyrosinase of potatoes; Separation of — into components. H. Huehn. Ber., 1919, 52, 2029—2041.

POTATO tyrosinase can be separated by ultra-filtration into a thermolabile residue (α -tyrosinase) and a filtrate (activator) which is stable towards heat; when separately tested, neither component shows the tyrosine reaction, but the mixture of the two is again active. The inactive residue can be activated by the addition of the solution obtained by boiling expressed potato juice in dilute acetic acid solution and subsequent filtration, as also by addition of an aqueous solution of the ash obtained by igniting the residue left on the evaporation of such a solution. Toluene appears to act as an oxygen carrier, since the preservation of solutions beneath toluene does not inhibit the enzymic process of tyrosinase as would be expected in consequence of the assumed exclusion of air. Alkaline solutions of pyrogallol become brown when placed beneath a layer of toluene 8 cm. high, and the result is not due to hydroclastic activity of toluene, since the

pyrogallol solutions remain unchanged when the experiment is performed in an atmosphere of hydrogen.—H. W.

PATENTS.

Alcohols; Process and apparatus for continuous rectification of —. E. Barbet et Fils et Cie., Paris. Eng. Pat. 107,975, 4.7.17. (Appl. 9659/17.) Int. Conv., 19.10.11.

SEE Fr. Pat. 478,946 of 1911; this J., 1916, 1173.

Alcohol-reduced beverages; Manufacture of —. H. Wade, London. From H. Heuser, Evanston, Ill., U.S.A. Eng. Pat. 135,785, 4.6.19. (Appl. 14,218/19.)

SEE U.S. Pat. 1,302,551 of 1919; this J., 1919, 551 A.

Peroxydase preparations; Process for manufacture of high-class —. A. Stoll, Assignor to Chemical Works formerly Sandoz, Basle, Switzerland. U.S. Pat. 1,324,752, 9.12.19. Appl., 7.11.18.

SEE Eng. Pat. 120,571 of 1918; this J., 1919, 551 A.

XIXA.—FOODS.

Rice; Chemical composition of natural and polished Italian —. I. G. Issoglio, Atti R. Accad. Sci. Torino, 1917-1918, 53, 423—436.

POLISHED rice is very poor in ash and also in total phosphoric anhydride, the phytin phosphoric anhydride being reduced to a minimum.—T. H. P.

Rice; Chemical composition of residues from the treatment of —. II. G. Issoglio, Atti R. Accad. Sci. Torino, 1918—1919, 54, 440—451.

RICE residues derived from the husk are very poor in nutritive substances, whilst those obtained by removal of the outer cortical layers of the corns are very rich in organic and inorganic phosphorus compounds, fats, and proteins, and contain phytin and vitamins. (See J. Chem. Soc., 1920, i, 132.)

—T. H. P.

Moisture content of feeding stuffs; Change in the — by grinding, a source of error in analysis. H. Neubauer. Landw. Versuchs Stat., 1919, 94, 1—8.

EXPERIMENTS made with potatoes and barley showed that when the moisture was determined on the un-ground sample, and on the same sample after grinding to 1 mm. fineness, the moisture was always less in the ground sample, the decrease varying from 1 to 5%. This difference was nearly the same as the loss of weight which occurred when the sample was exposed in a warm room for 24 hours. If the sample was partially dried before grinding, a further loss of moisture equal to the above difference still took place during grinding. The moisture content was determined also at different stages during the grinding process, and it was found that the loss of moisture occurred gradually in each stage. As the other constituents of the feeding stuff are usually determined on the ground sample, it is necessary to make allowance for the change in moisture content.—J. H. J.

Straw fodder; Treatment of — by Beckmann's [alkali] method. G. Fingerling and K. Schmidt. Landw. Versuchs Stat., 1919, 94, 115—152.

IN Beckmann's process of making fodder (see this J., 1919, 384 A, 789 A) straw is simply treated with alkali solution in the cold, and the authors have carried out numerous experiments with the object of ascertaining how the product thus obtained compares with that produced by methods in which the

straw is heated under pressure with alkali. They find that there is no appreciable difference in the digestibility of the two products. The results obtained show that three hours' treatment with 1.5% sodium hydroxide solution yields the best product.

—W. P. S.

Tyrosinase of potatoes. Haebl. See XVIII.

PATENTS.

Chocolate, cocoa butter, or other materials which coagulate when cold; Apparatus for cooling —. E. L. A. Savy, Paris. Eng. Pat. 124,196, 18.2.19. (Appl. 4047/19.) Int. Conv., 13.3.18.

A COOLING chamber is provided with two or more conveyors side by side and with horizontal fans supported on standards both above and below the conveyor. The conveyors may be worked at different speeds. A temperature-adjusting chamber is situated below the conveyors, in which the cooled material is placed and into which air at ordinary temperature is drawn by horizontal fans and caused to pass with a swirling motion over the material, so that its temperature is rapidly raised to that of the atmosphere.—J. H. J.

Hay, straw, and similar forage; Method of treating —. C. J. Coleman and H. G. Jones, London. Eng. Pat. 134,944, 15.11.18. (Appl. 18,787/18.)

THE material to be treated is placed in a revolving drum and heated to 300°–400° F. (about 150°–200° C.) at atmospheric pressure for 20–30 mins., whereby assimilable substances are produced from the starch and cellulose.—J. H. J.

Baking-powder and self-raising flour; Manufacture of — and materials to be used therein. A. E. Berry, Wanstead, and A. Boake, Roberts, and Co., Ltd., Stratford. Eng. Pat. 134,987, 30.11.18. (Appl. 19,815/18.)

PHOSPHORIC acid or a solution thereof is sprayed into agitated flour or other absorbent material, whereby a product which can be used as the acid ingredient of a baking-powder is produced. This product may be mixed with sodium bicarbonate to form a baking-powder, and with flour in addition to form a self-raising flour.—J. H. J.

Proteid substances; Process of manufacturing vegetable —. S. Satow, Sendai, Japan. U.S. Pats. (A) 1,321,479 and (B) 1,321,480, 11.11.19. Appl., 25.11.16.

(A) PROTEIN-CONTAINING material is treated to destroy its cellular structure and to remove oil, after which it is extracted by a liquid without heat, and the protein precipitated from the solution by ferment action. (B) In the preceding process the protein is precipitated by sulphurous acid.

—J. H. J.

Cereal food and process of production. J. L. Kellogg, Battle Creek, Mich. U.S. Pats. (A) 1,321,753 and (B) 1,321,754, 11.11.19. Appl., 10.6.19.

(A) GRAIN is heated at a temperature sufficient to dextrinise starch partially, after which it is slightly dried and formed into separate particles, which are afterwards dried or toasted. (B) In the preceding process the separate particles, which may be in the form of shreds, are subjected to a high temperature, about 400° F. (about 200° C.), for some minutes so as to puff the particles and cook them completely. The preliminary heating may be effected with live steam. The food produced has the characteristic of retaining its puffed form when added to milk.

—J. H. J.

Food product; Process for the production of a — rich in proteins from cereal grains. A. Backhaus. Ger. Pat., 301,365, 22.12.16.

AFTER removing the oil as completely as possible, the grains are heated with a small amount of alkali, dried, and ground. A product of good keeping quality and improved taste, containing about 40% of proteins, is thus obtained. It may be added to other foods, or used as a coffee substitute.

—J. P. O.

Food condiments; Process for the preparation of —. A. Backhaus, Berlin. Ger. Pat. 303,994, 20.4.17.

THE germ of grain, from which the fat and bitter principle have been removed, is treated with acid and submitted to the combined action of proteolytic enzymes and yeast. By this treatment the albuminous matter is dissolved, the starch and sugar fermented, and flavouring substances are formed. The product is filtered and evaporated to any desired consistence.—J. H. J.

Meat-juice preserves; Manufacture of —. Nobel und Co., Hamburg. Ger. Pat. 307,135, 13.5.16.

RAW meat is chopped, partly fine and partly in cubes or irregular pieces, suitable quantities of salt and spices are added, and the meat is then mixed with readily soluble dried milk. The mixture is placed in the containers in which it is to be packed, and exposed to the air at 10°–12° C. for about 24 hours, in order to obtain a uniform distribution of the dried milk throughout the mass and a change of the alkaline reaction to acid, with the liberation of small quantities of gas, after which the containers are sealed and sterilised.—J. F. B.

Bran; Process for the removal of — from whole grain for the purpose of making meal. E. W. Wippermann, Hamburg. Ger. Pat. 313,798, 30.1.16.

WHOLE grain is moistened with water or with 0.5 salt solution and is immediately afterwards roasted, the quantity of water or salt solution used being such that only the outer bran coat is acted upon, whilst the grain proper remains unchanged.

—J. H. J.

Food or fodder from scrap leather; Process for the production of a —. J. G. Bader, Mühlhausen. Ger. Pat. 311,323, 23.3.15.

LEATHER scrap is washed, soaked, treated with milk of lime, again washed and soaked, treated in an acid bath, once more washed, pressed, ground, and dried, and if necessary reduced to a meal.

—J. H. J.

Milk; Device for skimming or creaming —. R. Clavel, Basle, Switzerland. U.S. Pat. 1,323,015, 25.11.19. Appl., 26.11.18.

SEE Ger. Pat. 314,090 of 1918; this J., 1920, 40 A.

XIXB.—WATER PURIFICATION; SANITATION.

Waters; Effect of sea-salt on the pressure of carbon dioxide and alkalinity of natural —. E. B. R. Prideaux. Chem. Soc. Trans., 1919, 115, 1223–1230.

TO determine the effect of carbon dioxide on the alkalinity of sea water, experiments were made in which standard carbonate and bicarbonate solutions were mixed with artificial brine solutions having approximately the composition of sea water, and the alkalinity was determined by comparison with

standard solutions, using phenolphthalein or α -naphtholphthalein as indicator. When the value of R , the ratio of equivalents of alkali to molecules of carbon dioxide, was between 1.08 and 1.12, the alkalinity in the case of fresh water was $p_H=8.9$ and in salt water $p_H=8.1$. All waters of higher alkalinity than $p_H=8.1$ will gain carbon dioxide from the air, and this is true of nearly all surface sea waters. The alkalinity of surface waters cannot fall much below 8.1, for then the pressure of carbon dioxide in the sea becomes greater than that in the atmosphere. The alkalinity of bicarbonated fresh waters is kept at about the same value, $p_H=8$, but in these cases the ratio R is about equal to 1.0. Solutions having the higher ratios, 1.06 to 1.16, found in sea water would contain an appreciable amount of carbonate and could not long exist.

—E. H. R.

Drinking water; Purification of — with *potassium permanganate*. H. Drunk. Veröff. Gebiete d. Milit. Sanitätswesen [66], 1. Chem.-Zeit., 1919, 43, Rep., 270.

THE use of an excess of hydrogen peroxide in the permanganate process of purification is not detrimental, as any remaining over is decomposed by the manganese oxides. Tests on the Berlin Nordkanal water and a well water are reported. The observed increase in the total solids left on evaporation is accounted for by the residue of the purifying material. Iron was completely removed and nitrous acid oxidised to nitric acid. The carbonate hardness was only slightly reduced, and no reduction in lime content was observed. The manganese content was usually considerable. Copper sulphate did not behave as a catalyst, but fixed the potassium ions derived from the permanganate, and thus prevented the water from becoming alkaline. The reaction between copper sulphate, permanganate, and hydrogen peroxide was studied, but a regular course of the reaction was not observed. The manganese content of the water varied. On reducing the proportion of copper sulphate to permanganate, the manganese content of the purified water diminished and the minimum was found when the reagents were in equivalent proportions. When calcium carbonate was added before or during the process, the water was freed from manganese and copper. In general, the process yields clear, sterile, colourless drinking water of undiminished palatability and hygienically satisfactory.—H. J. H.

Eusol; Method for the accurate preparation of —. E. J. Hart. Pharm. J., 1919, 103, 535—536.

THE antiseptic hypochlorite solution for the treatment of wounds usually known as eusol (Rettie, this J., 1918, 237) should not contain an excess of boric acid beyond that required to liberate the hypochlorous acid, otherwise its keeping qualities are impaired and it is more irritating to the wound. A solution of suitable strength containing, when fresh, 0.45% of hypochlorous acid is made by dissolving separately 9.636 grms. of boric acid and 6.132 grms. of calcium hypochlorite (in practice an equivalent quantity of bleaching powder is used and the solution is filtered), each in a sufficient quantity of water to make 500 c.c. of solution, and when cold the two solutions are mixed. The above quantities include the amount of boric acid required to neutralise the calcium hydroxide passing into solution from the bleaching powder.

—G. F. M.

Hydrocyanic acid; Disinfection with —. H. Fühner. Pharm. Zentrbl., 1919, 60, 487—492.

HYDROCYANIC acid vapour has been used for destroying clothes moths, Indian meal moth, etc.,

with satisfactory results. To determine the risk of poisoning when rooms are so treated, the author carried out a disinfection experiment in a sealed room containing upholstered furniture, cushions, clothes, etc., 670 grms. of hydrocyanic acid (from sodium cyanide and sulphuric acid) being liberated in the 425 cb. m. of room space. After 15 hours the room was well ventilated. All moths, flies, etc., in the room were killed, as were also mice and ants placed in cages in the room. All the poison was removed by 10 hours' ventilation, except in the case of the cushions, which retained some of the gas for a considerable time longer.—W. P. S.

Anti-dimming compositions for use in the gas mask. P. W. Carleton. J. Ind. Eng. Chem., 1919, 11, 1105—1111.

EXPERIMENTS with various compositions to prevent the glass or celluloid eyepiece of gas masks from being rendered dim by condensed moisture showed that those made from sulphonated oils gave by far the best results. For use with unventilated gas masks of the English and American type sticks and paste were made in accordance with the following formula:—Turkey-red oil (85%), 100; caustic soda, 15; water-glass, 5; and paraffin oil, 5 parts, but subsequently sodium carbonate was used in place of caustic soda owing to the action of the latter on the hands of the workpeople. The product contained from 10% to 20% of moisture. In the case of the French ventilated "Tissot" masks an addition of glycerin was necessary, and the composition was made according to the following formula:—Turkey-red oil (85%), 100; caustic soda, 15 (subsequently replaced by sodium carbonate); water-glass, 5; paraffin oil, 5; and glycerin, 23 parts. In each case a little of the composition was applied to the glass or celluloid, and the latter then polished gently with a rag.—C. A. M.

Anti-dimming preparations for gas masks. H. N. Holmes, F. F. Jewett, G. Leavell, D. Bailey, and E. Shaver. J. Ind. Eng. Chem., 1919, 11, 1111—1116.

COMPARATIVE tests for anti-dimming preparations are described. The requirements of a good preparation are that it should lower the surface tension of water to a pronounced extent, that it should be soluble, but not too soluble, or it will not be retained by the glass, and that it be sufficiently deliquescent to prevent drying. Alkali soaps of unsaturated fatty acids have a much greater power of forming films than those of unsaturated fatty acids. The action of soaps in general is improved by the addition of a small percentage of glycerin, 10 to 15% of sodium hydroxide, and a small amount of "marine oil," with the addition of a small quantity of water-glass as a binding agent. The addition of paraffin oil recommended by other workers is not advantageous. Sulphonated oils made from castor, cottonseed, rape, maize, linseed, and olive oils and from olein all gave excellent results, which proved that the hydroxy group (present only in castor oil) is not a main factor in the process. The alkali salts of sulphonated saturated fatty acids are also effective. There is little difference in the effects of different alkali soaps of sulphonated oils, although the potassium soap of sulphonated cottonseed oil gave better results than the sodium soap. The preparations made in accordance with the following formulae are stated to be decidedly better than the official (U.S.A.) products:—*Sulphonated rape oil*: Sodium salt of 65% sulphonated rape oil (35% H_2O), 100; sodium hydroxide, 20; water-glass, 5; glycerin, 3; and "marine oil," 3 parts. *Sulphonated cottonseed oil*: Potassium salt of 62% sulphonated cottonseed oil (38% H_2O), 100; sodium hydroxide, 15; water-glass, 3; glycerin, 3; and "marine oil," 3 parts.

In both cases these are dried to form solid sticks when compressed. The "mariae oil" (Standard Oil Co.) was probably a mixture of blown rape oil and heavy mineral oil; it had fairly good film-forming properties by itself.—C. A. M.

Ammonium polysulphide wash. J. V. Eyre, E. S. Salmon, and L. K. Wormald. J. Bd. Agric., 1919, 26, 821—822. (See also this J., 1919, 335 A.)

EXPERIMENTS showed that a wash containing 0.11% of polysulphide sulphur was fungicidal to the conical stage of hop mildew (*Sphaerotheca humuli*). A new method of preparing a concentrated ammonium polysulphide wash is as follows: A current of hydrogen sulphide is passed through a mixture of 1 litre of ammonia solution (sp. gr. 0.880), 300 c.c. of distilled water, and 400 grms. of flowers of sulphur until all the sulphur is dissolved. This takes 46—47 hrs., and the mixture should be shaken occasionally. To the dark red liquid 250 c.c. more of ammonia solution is added. This liquid forms the stock solution. It contains 6.5% of sulphide sulphur and 21.9% of polysulphide sulphur. For use, $\frac{1}{2}$ gal. of the stock solution is poured into a solution of 5 lb. of soft soap in 99 $\frac{1}{2}$ gals. of water. This mixture forms the dilute wash and contains 0.5% of soap and 0.11% of polysulphide sulphur. Two applications of the wash should be given at intervals of 7 or 10 days.—J. H. J.

Chloropicrin; Influence of temperature and other physical agents on the insecticidal power of —. G. Bertrand, Brocq-Roussau, and Dassonville. Comptes rend., 1919, 169, 1059—1061.

In the use of chloropicrin against insects, light and the moisture content of the air have no influence on the result, but the temperature plays an important part. Over the temperature range 10°—30° C. the temperature coefficient of the velocity of action is 2—2.5.—W. G.

PATENTS.

Gas; Apparatus for measuring out charges of — [e.g. for sterilising water]. S. H. Menzies and H. J. Magrath, London. Eng. Pat. 134,615, 5.11.18. (Appl. 18,114/18.) (See also Eng. Pat. 121,521; this J., 1919, 63 A.)

A CYLINDRICAL measuring cylinder communicates at the top through a diaphragm with a pressure gauge and is closed at the bottom by a cap. The cap may vary in size so as to vary the capacity of the cylinder, and may carry bars to project into the cylinder and thus reduce its capacity. The cylinder communicates by narrow passages on each side with a paired valve apparatus, acting as inlet and outlet valves. The inlet valve is operated by a push plunger which impinges on a diaphragm connected by rods to a needle valve, which admits gas from a pressure reservoir. The gas first passes through a porous plug so as to equalise the rate of flow. The measuring cylinder is allowed to fill with the gas to the pressure desired, as indicated on the pressure gauge above the cylinder. The push plunger is then released and the one on the exit valve operated, which allows the gas to leave the measuring cylinder and to pass by a pipe to the liquid to be charged. The apparatus is specially suited for measuring a noxious gas, such as chlorine, for the treatment of water for sterilising purposes.—J. H. J.

Sterilising and drying animal and vegetable matter, including slaughter-house offal; Process for —. J. C. Marshall, Manchester. Eng. Pat. 133,983, 23.8.18. (Appl. 13,774/18.)

PIPES, arranged horizontally in series and spaced apart, are supported above the floor of a drying-room. Several such horizontal layers of pipes are arranged one above the other. All the layers are

supplied with valves and are jointly connected to a steam supply or a hot-air supply. The material to be dried is spread in a layer three or more inches deep on the pipes. As the material dries it falls through the spaces between the pipes.—J. H. J.

Liquids; Treatment of —. W. S. Elliott, Pittsburgh, Pa. U.S. Pat. 1,321,999, 18.11.19. Appl., 31.3.15.

LIQUID to be treated is heated and introduced into a chamber in which a partial vacuum is maintained, so that the entering liquid is caused to boil violently, whereby all dissolved gases are evolved and also scale-forming substances are precipitated.—J. H. J.

Liquids [containing phenolic substances]; Purification of —. (A) C. A. Basore, (B) H. S. Davis and S. D. Semenow, and (C) E. A. Dieterle and S. D. Semenow, Assignors to The Koppers Co., Pittsburgh, Pa. U.S. Pats. (A) 1,323,239, (B) 1,323,251, and (C) 1,323,256, 2.12.19. Appl., 15.2.19.

LIQUIDS containing phenolic bodies, such as waste ammonia still liquors, are clarified and filtered through a bed of (A) animal charcoal, (B) a phenol-absorbing humus material, such as ground lignite, or (C) peat. Animal charcoal after use may be re-charred and used for the treatment of more liquid.—L. A. C.

Germicide solution and method of making the same. C. A. Weeks, Philadelphia, Pa., U.S.A. Eng. Pat. 134,880, 11.10.18. (Appl. 16,606/18.)

CHLORINE gas is bubbled upwards through a tall column of eucalyptus oil or eucalyptol kept at 100°—125° F. (38°—52° C.). Hydrochloric acid is formed, and its escape from the oil is facilitated by passing in air or ozone. The treatment is continued until the oil contains 3% Cl, corresponding to the formula $C_{10}H_{17}OCl_2$. Hydrochloric and other acids are also present. The product is an oily liquid of sp. gr. 1.2, soluble in paraffin, alcohol, and ether, and miscible with water. For use, the oil is mixed with water in the proportion of $\frac{1}{2}$ —2 drachms of oil to 1 gal. of water.—J. H. J.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Chelidonium alkaloids. J. Gadamer. Arch. Pharm., 1919, 257, 298—303.

ON the basis of the results obtained by the author, structural formulae are proposed for chelidonine and α - and β -homochelidonines. (See also J. Chem. Soc., 1920, i., 75.)—T. H. P.

Cervine and sabadinine. K. Hess and H. Mohr. Ber., 1919, 52, 1984—1988.

INVESTIGATION of the alkaloids themselves, of their hydrogen sulphates, potassium salts, aurichlorides, hydrochlorides, and monobenzoyl-derivatives, shows them to be identical. The formula is $C_{22}H_{43}O_8N$. Certain corrections of the data recorded in the literature are given. The alkaloid crystallises with 3.5 mols. H_2O , m. pt. 110° C. The hydrogen sulphate (+2.5 H_2O) has m. pt. 250° C. (decomp.) after becoming discoloured at 210° C. The hydrochloride forms anhydrous needles, m. pt. 247° C.—H. W.

Ormosine and ormosinine, two new alkaloids from Ormosia dasycarpa. K. Hess and F. Merck. Ber., 1919, 52, 1976—1983.

ORMOSINE has been isolated previously by Merck (Wiss. Abhandl. Gebiete Pharm., 1888, No. 22, 346) from *Ormosia dasycarpa*, Jack., a leguminous

plant growing in Venezuela. Its physiological relationship to the morphia alkaloids has been demonstrated by Harnack (Kobert's Jahresber. Fortschr. Pharm., 1881, 1, 59). The authors find that it is not related chemically to these substances, and, indeed, occupies a unique position among alkaloids. In addition, they have isolated ormosinine from the same source, the yield of the former amounting to 0.15%, and of the latter to 0.023%, of the dried seeds. These substances, however, only represent a fraction of the total alkaloid content. The alkaloids are isolated by extracting the crushed seeds with alcohol, evaporating the solvent in a vacuum, and extracting the residue with water; the aqueous solution is made alkaline with ammonia, and shaken with ether. The ethereal solution is agitated with powdered sodium carbonate, after which the solvent is removed, leaving a crystalline residue, from which ormosinine is isolated by taking advantage of its sparing solubility in alcohol, whilst ormosine separates from the alcoholic solution on addition of water. Ormosine, $C_{26}H_{41}N_3$, crystallises (with 3–4 H_2O) in long needles, m. pt. 85° – 87° C., and is readily soluble in alcohol or chloroform. When preserved over sulphuric acid, it loses its water of crystallisation, and forms a colourless gummy mass, which again becomes crystalline when brought into contact with water. At 100° C. it is converted into a viscous oil which does not recombine with water. Ormosinine, $C_{26}H_{41}N_3$, crystallises in well-defined anhydrous cubes and short prisms, m. pt. 203° – 205° C. It is probably the anhydrous form of ormosine.—H. W.

Aloes; Chemistry and pharmacology of —. Products of the oxidation of the constituents of aloes by alkali persulphate, Caro's acid, and hydrated sodium peroxide. E. Seel. Arch. Pharm., 1919, 257, 212–228, 229–254, 254–259.

THE products obtained on oxidising aloin, aloëtin, and the crude resin of aloes have been separated, and the physiological actions of certain of them investigated. (See also J. Chem. Soc., 1920, i., 67, 68, 69.)—T. H. P.

Athaea [marsh mallow] root; Some components of —. O. von Friedrichs. Arch. Pharm., 1919, 257, 288–298.

MARSH mallow root contains 1.7% of an oil, composed of palmitin and olein; butyric acid; a phytosterol, apparently identical with sitosterol; a hydroxy-acid of high molecular weight; an odorous substance of unknown composition; a lecithin containing palmitic and oleic acids and with a basis of choline; 10.2% of sucrose and 0.78% of invert sugar; a mucilage, $(C_6H_{10}O_5)_n$, consisting of glucosan (64%) and xylan; and another saccharo-colloid giving *d*-galactose on hydrolysis.—T. H. P.

Xanthosterol, a crystalline compound from the bark of Xanthoxylum Budrunga. H. Dieterle. Arch. Pharm., 1919, 257, 260–263.

THE bark of *Xanthoxylum Budrunga* contains about 0.25% of an alcoholic compound, $C_{27}H_{48}O$, related to lupeol and alstol, and named xanthosterol by the author. Its reactions and certain of its derivatives are described. (See also J. Chem. Soc., 1920, i., 42.)—T. H. P.

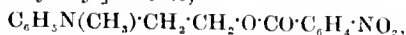
Cholesterol; Transformation of — into cholic acid. A. Windaus and K. Neukirchen. Ber., 1919, 52, 1915–1919.

THE saturated hydrocarbon, cholestane, $C_{27}H_{48}$, obtained by the chemical reduction of cholesterol, is oxidised by chromic acid to an isomeride of cholic acid, $C_{27}H_{46}O_2$, which differs from cholic acid, $C_{27}H_{46}O_2$, in having three hydrogen atoms for the three hydroxyl groups. ψ -Cholestane, which is the saturated hydrocarbon of coprosterol, the

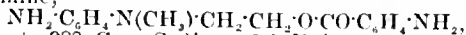
bacterial reduction product of cholesterol, gives true cholic acid on oxidation. This shows that cholic acid has the same carbon skeleton as cholesterol, except that this has an extra isopropyl group, since it yields acetone on oxidation whilst the cholic acid derivatives do not. (See further, J. Chem. Soc., 1920, i., 11.)—J. C. W.

Novocaine; Aromatic analogues of —. J. von Braun and G. Kirschbaum. Ber., 1919, 52, 2011–2015.

IT has been recently shown by Fränkel and Cornelius (Ber., 1918, 51, 1654) that derivatives of primary β -aminoethyl alcohol, in contrast to the tertiary *N*-dialkylated alkamine derivatives (stovaine, novocaine) do not possess anaesthetic action. The compounds which they have examined, however, contain an acyl residue attached to the nitrogen atom in place of the alkyl radical of novocaine, and thus do not belong to quite the same type. Closer analogy is shown by a series of substances which are now described in which a phenyl or substituted phenyl group is introduced in place of one of the alkyl groups. It is found that the aromatic substitution at the nitrogen atom of novocaine is without influence on its anaesthetic power if the basicity of the molecule is also suitably increased. *N*-methyl-*N*- β -benzoyloxyethyl-*p*-phenylenediamine, $NH_2 \cdot C_6H_4 \cdot N(CH_3) \cdot CH_2 \cdot CH_2 \cdot O \cdot CO \cdot C_6H_5$, long slender needles, m. pt. 56° C., is obtained by reduction of the corresponding *p*-nitroso compound, pale green powder, m. pt. 90° C. Sodium *p*-nitrobenzoate reacts readily with β -chloro- or β -bromoethylmethylaniline yielding *N*-methyl-*N*-[β -*p*-nitrobenzoyloxyethyl]-aniline,



yellow needles, m. pt. 70° C., which is reduced to *N*-methyl-*N*-*p*-aminobenzoyloxyethylmethylaniline, colourless leaflets, m. pt. 112° C. When the solution of the latter in dilute hydrochloric acid is treated with sodium nitrite, it yields the corresponding *p*-nitroso-derivative, m. pt. 105° – 106° C., which is almost quantitatively reduced by stannous chloride to *N*-methyl- β -*p*-aminobenzoyloxyethyl-*p*-phenylenediamine,



m. pt. 98° C. Sodium 3,5-dinitrobenzoate and chloroethylmethylaniline yield *N*- β -3,5-dinitrobenzoyloxyethylmethylaniline, m. pt. 121° C., which is normally reduced to the corresponding diamino compound, m. pt. 80° C.—H. W.

Anaesthetics Local —. L. Launoy and Y. Fujimori. Comptes rend. Soc. Biol., 1919, 82, 732–736. Chem. Zentr., 1919, 90, III., 831.

THE benzoyl derivatives of a number of amino-alcohols of the formulae, $(CH_3)_2N \cdot CH_2 \cdot CR(OH) \cdot CH_3$, and $(CH_3)_2N \cdot CH_2 \cdot CH_2 \cdot CH(OH)R$, were examined in respect to their toxicity, their haemolytic action on red blood corpuscles, and their anaesthetic properties. Of the first series, to which stovaine belongs, the benzoyl derivatives of 2-methyl-, 2-ethyl-, 2-amyl-, 2-phenyl-, and 2-benzyl-propanol were investigated, and of the second series the derivatives of 3-ethyl- and 3-amyl-propanol. The derivatives of the second series were less toxic than those of the first, whilst those of the alcohol containing five carbon atoms were the most poisonous. Of the aromatic derivatives the benzyl-propanol derivatives were more toxic than the corresponding phenyl compounds. The haemolytic action on red blood corpuscles increased with increasing molecular weight; it appeared also to increase with increasing separation of the hydroxyl and amino groups. The anaesthetic action of the tertiary alcohols is superior to that of the secondary, and the maximum is attained with the C_5 alcohols.

—G. F. M.

Paracymene. III. Preparation of 2-chloro-5,6-dinitrocymene. H. A. Lubs and R. C. Young. *J. Ind. Eng. Chem.*, 1919, 11, 1130—1133.

THE nitration of 2-chloro-*p*-cymene does not result in the simple formation of dinitrocymene; the principal product is 2-chloro-5,6-dinitro-*p*-cymene, whilst an isomeric compound, not yet isolated, also appears to be formed. A small amount of another dinitrochloro compound (m. pt. 90°—91° C.), containing less carbon than dinitrochlorocymene, is also produced, together with a small quantity of another compound, possibly a chloroterephthalic acid.—C. A. M.

Tetrachlorodinitroethane. W. L. Argo, E. M. James, and J. L. Donnelly. *J. Phys. Chem.*, 1919, 23, 578—585.

ALTHOUGH wrought iron satisfactorily withstands the action of a mixture of dry liquid nitrogen tetroxide and tetrachloroethylene, it is in practice impossible completely to exclude moisture. A porcelain-lined autoclave is therefore more suitable for the production of tetrachlorodinitroethane, the mixture being heated at 80° C. for three to six hours. It is important that the nitrogen tetroxide be dry and be present in excess in the liquid form. (See further, *J. Chem. Soc.*, Feb., 1920.)—J. K.

Nitro-compounds; Influence of the position of substituents on the behaviour of aromatic — in the Kjeldahl estimation of nitrogen. B. M. Margosches and E. Vogel. *Ber.*, 1919, 52, 1992—1998.

THE behaviour of the isomeric nitrophenols, nitrobenzoic acids, and nitrobenzaldehydes when treated with sulphuric acid and potassium sulphate has been investigated. The nitrogen content of the *ortho*-derivatives can be estimated accurately in this manner, but the results for the *meta*- and *para*-compounds are much too low. The favourable influence of the hydroxy- or alkoxy-group in the *ortho*-position to the nitro-group is further shown by the fact that the process gives exact results with 2-nitroresorcinol, 3-nitro-*p*-cresol, and *o*-nitrophenetol, but low results with *p*-nitrophenetol. The presence of *o*-nitrophenol or of *o*-nitrobenzoic acid has a favourable influence on the behaviour of the corresponding *meta*- and *para*-derivatives, and this action cannot be ascribed solely to their content of phenol or benzoic acid. The use of salicylic acid appears particularly advantageous in the case of *meta*-compounds. It is somewhat remarkable that the three nitrocinnamic acids give accurate results. A further series of estimations is recorded with 2,4-dinitrophenol, 2,4-dinitro-*a*-naphthol, 2,4-dinitrobenzoic acid, 2,4-dinitrotoluene, 3,5-dinitrobenzoic acid, 2,6-dinitrotoluene, and 2,4,6-trinitrophenol; the results are considerably low in every case, although concordant amongst themselves. The facts, however, that the 2,4-dinitro-compounds give higher values than the corresponding 3,5- or 2,6-dinitro-derivatives and that 2,4-dinitrophenol gives results almost identical with those obtained with 2,4-dinitrotoluene are in accordance with the observations made with the mononitro-compounds.

—H. W.

Halogen attached to a ring carbon atom; Replacement of — by other substituents. I. Replacement by the carbonyl group. K. W. Rosenmund and E. Struck. *Ber.*, 1919, 52, 1749—1756.

HALOGEN atoms in cyclic compounds can be directly replaced by carboxyl groups by heating the compounds with aqueous or dilute alcoholic potassium cyanide and a little euprous cyanide, for some hours at about 200° C. in strong tubes. Good results have been obtained with halogen derivatives of benzene (mono- and di-), toluene, aniline, nitro-

benzene, phenol, carboxylic acids, naphthalene, and thiophen. (See further, *J. Chem. Soc.*, 1920, i., 44.)
J. C. W.

Aldehydes; New method of converting carboxylic acids into —. A. Sonn and E. Müller. *Ber.*, 1919, 52, 1927—1934.

THE replacement of the chlorine atom of anilide-imido-chlorides by hydrogen is readily effected by the action of an ethereal solution of pure stannous chloride containing the requisite quantity of hydrogen chloride; the tin double salts of the corresponding Schiff's bases appear to be formed as intermediate products, and these pass readily into aldehydes and aniline salts when heated with dilute acids or, in certain cases, when simply treated with steam. The preparation of benzaldehyde, cinnamaldehyde, *p*-hydroxybenzaldehyde, and 3,4,5-trimethoxybenzaldehyde in this manner from the anilide-imido-chlorides of the corresponding carboxylic acids is described, the yields being very satisfactory. The method does not appear to be applicable to the preparation of aliphatic aldehydes. (See also *J. Chem. Soc.*, 1920, i., 58.)

—H. W.

Acrolein; Stabilisation of —. IV. Compounds acting as stabilisers against the formation of desacryl (insoluble resin). C. Moureu, C. Dufrasse, and P. Robin. *Comptes rend.*, 1919, 169, 1068—1072. (See this *J.*, 1919, 923 A.)

Benzoic acid is present in certain of the fractions obtained in the fractional distillation of acrolein and exerts a marked but variable stabilising action. Phenol is also present, but is only a feeble stabilising agent.—W. G.

Arsenic acid; Action of — on polyhydric phenols. A. Sonn. *Ber.*, 1919, 52, 1704.

POLYHYDRIC phenols suffer oxidation when heated with arsenic acid in the usual way, but if pyrogallol and dilute arsenic acid solutions are concentrated in a vacuum and left in the cold, a crystalline acid of the formula $O:As[O:C_6H_3(OH)_3]$ is deposited.

—J. C. W.

Aromatic selenium compounds; Simple and convenient way to prepare —. A. Schoeller. *Ber.*, 1919, 52, 1517—1518.

DIAZONIUM salt solutions react with alkali polyselenides to form organic selenides and sometimes diselenides. The reagent is obtained by fusing black selenium with potassium hydroxide and dissolving the product in ice-cold water. The use of hydrogen selenide is thus unnecessary. (See further, *J. Chem. Soc.*, 1920, i., 41.)—J. C. W.

Thallium; Use of — in organic chemistry. K. Freudenberg and G. Uthemann. *Ber.*, 1919, 52, 1509—1513.

THALLIUM hydroxide or carbonate is useful in the isolation of acids of the gallie acid series. Uric acid, phthalimide, and lactulose also give well-defined thallium salts. Glycine may be prepared from the hydrochloride of its ester by boiling with thallium carbonate and water, filtering the precipitate of thallium chloride, saturating with hydrogen sulphide, and filtering again. (See further *J. Chem. Soc.*, 1920, i., 17.)—J. C. W.

Fluorine; Action of — on organic compounds. B. Humiston. *J. Phys. Chem.*, 1919, 83, 572—577.

THE action of fluorine on organic compounds is usually violent, leading to decomposition. Saturated hydrocarbons are ignited with incandescence and formation of free carbon, fluorides of carbon, and hydrofluoric acid. The behaviour of charcoal ("filthchar"), selenium, acetone, chloroform, phos-

gene, carbon monoxide, carbon tetrachloride, and tetrachloroethylene was also investigated. (See further J. Chem. Soc., Feb., 1920).—J. K.

Ichthyol oils. Scheibler. See IIa.

PATENTS.

Ethyl acetate; Manufacture of — from acetaldehyde. Konsortium für elektrochemische Industrie, Munich. Ger. Pat. 314,210, 27.10.14.

THE catalytic action of aluminium alkyl-oxide (alcoholate) on the conversion of acetaldehyde into ethyl acetate (see Eng. Pats. 26,825-6 of 1913; this J., 1914, 666) is aided by the presence of small quantities of substances which, by themselves, effect the conversion of the aldehyde into paraldehyde or into alcohol. When present to the extent of 5% calculated on the acetaldehyde, aluminium alkyl-oxide gives rise to a 15% yield of ethyl acetate; in the additional presence of small quantities of the following substances the yields are as bracketed:—2% of aluminium chloride (80%); 1% of benzenesulphonic acid (61%); 1.5% silicon chloride (63%); 40% anhydrous copper sulphate (50%); 0.4% sodium hydroxide (56%); 0.4% calcium hydroxide (55%); 0.1% sodium acetate (47%); 0.4% sodium cyanide (77%).—D. F. T.

Erythrene; Purification of —. I. Ostromisslensky, Moscow. Ger. Pat. 314,364, 13.5.14.

CRUDE erythrene, e.g., that obtained by dehydration of 1,3-butyleneglycol, can be purified by treatment with a neutral oxidising agent such as benzoyl peroxide or barium peroxide, or with an alkaline oxidising agent consisting of a mixture of barium peroxide and sodium.—D. F. T.

Monochloroacetic acid; Continuous process for preparation of — from trichloroethylene. A. Guyot, Salindres, Assignor to Comp. des Prod. Chim. d'Alais et de la Camargue, Paris. U.S. Pat. 1,322,898, 25.11.19. Appl., 21.9.18.

SEE Eng. Pat. 132,042 of 1918; this J., 1919, 846 A.

Acids from wood tar. Ger. Pat. 314,358. See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Papers; Measuring the gloss of photographic —. K. Kieser. Z. angew. Chem., 1919, 32, 357-359.

THE method proposed is based upon the fact that the light reflected from non-metallic surfaces is plane-polarised in proportion to the degree of gloss of the reflecting surface. A suitable instrument is the Martens' polarisation-photometer which constitutes the upper portion of the Schmidt and Hensch densimeter. If the two slits through which the light enters for the estimation of the density or degree of blackening be replaced by a single central opening, the Martens' photometer is converted into a polarimeter, resembling the old Cornu polarimeter. The rotation of the analyser Nicol is the measure of the polarisation, and the percentage of polarised light in the total light is proportional to the tangent of the angle of rotation. The light must fall on the surface of the paper at an angle of 56° and the polarimeter must be directed towards the paper also at that angle; the line of demarcation of the two halves of the field of observation of the polarimeter will then lie in the same plane as the incidence of the light. Complete polarisation will correspond to a rotation of the analyser through 45°. The best source of light is an electric incandescence lamp controlled by a variable resistance and provided with a depolarising matt

screen; a dark-room is unnecessary. Papers are always examined undeveloped, washed, and dried; it is very important that they should not be "fogged," since a slight greyness considerably increases the degree of polarisation. A slight coloration does not interfere, but with pronounced colours values which are much too high are obtained and the two halves of the observation field are differently coloured. Matt gelatin photographic papers give a value of about 10°, highly glazed ones about 30°, which is about the same as that of white enamelled glass. The method is capable of being used for the determination of the gloss of ordinary white papers which show values between 3° and 12°; moreover, it is capable of showing much finer differences than can be detected by an experienced eye.—J. F. B.

PATENT.

Coloured motion-pictures; Method and apparatus for producing —. W. V. D. Kelley, Brooklyn, N.Y., Assignor to Prizma Inc. U.S. Pat. 1,322,794, 25.11.19. Appl., 12.7.15.

A PART of each of a series of motion-pictures is obtained by open exposure, i.e., without the interposition of the colour-filter which is used for the rest of the picture.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Trinitrotoluene; Oxidation in the manufacture of —. A. S. Eastman. J. Ind. Eng. Chem., 1919, 11, 1124.

IN the nitration of toluene the high temperature at which the process is carried out causes a considerable oxidation of the nitrotoluenes, with a corresponding decrease in the yield of TNT. The spent acid contains about 18% HNO_2 , whilst the nitrator contains a mixture of the various isomeric di- and trinitrotoluenes, and, in one experiment, 2,4-dinitrobenzoic acid was isolated. The following method of determining the total organic acids formed by oxidation of the methyl group has been devised: Nitric acid is removed by evaporation, and sulphuric acid by neutralisation with barium hydroxide and separation of the barium sulphate, the filtrate then containing the soluble barium salts of the organic acids, carboxylic acids formed by oxidation of the methyl group of the toluene, and sulphonic acids produced during the nitration. The barium salts of the organic acids are determined by evaporating an aliquot part of the filtrate and weighing the dry residue of barium salts. The residue is then ignited and the resulting mixture of barium carbonate and sulphate weighed, treated with hydrochloric acid to remove the carbonate, and the residue of barium sulphate weighed. From these data the loss of toluene due to sulphonation and oxidation to carboxylic acids can be calculated, whilst the loss on igniting the dry barium organic salts represents the total loss of toluene (e.g., 1.86%). This would only account for a small proportion of the HNO_2 present. In the nitration of dinitrotoluene ("bi-oil") to TNT, carbon dioxide, carbon monoxide, and oxygen are evolved, the two former in a proportion sufficient to lower the yield of TNT by about 5%, or more than a third of the total loss in the whole process. The composition of the gas may vary considerably, even in the course of a single nitration, and under certain conditions the proportion of carbon monoxide may be so high that the gas mixture becomes explosive. This may account for various explosions in TNT nitrators in which the top of the vessel has been blown off without exploding the charge of TNT.—C. A. M.

Nitrotoluenes; Studies on the —. III. Binary systems of the components p-nitrotoluene, 1,2,4-dinitrotoluene, and 1,2,4,6-trinitrotoluene. J. M. Bell and C. H. Herty, jun. J. Ind. Eng. Chem., 1919, 11, 1124–1127.

SYNTHETIC mixtures of pure p-nitrotoluene (MNT), 1,2,4-dinitrotoluene (DNT), and 1,2,4,6-trinitrotoluene (TNT) were prepared, and the m. pts. of two-component systems of these were determined by the melting point method, and plotted in curves. The most probable value for the m. pt. of MNT was found to be $51.5^{\circ} \pm 0.3^{\circ}$ C., for DNT $69.6^{\circ} \pm 0.3^{\circ}$ C., and for TNT $80.35^{\circ} \pm 0.3^{\circ}$ C. In the binary system MNT–TNT the eutectic points were 33.85° – 33.95° C. (with MNT as solid phase) and 33.7° – 34.2° C. (with TNT as solid phase); in the system DNT–TNT the eutectic points were 45.4° – 45.6° C., and in the system MNT–DNT, 26.44° – 26.54° C. The freezing points of the respective mixtures of from 0 to 100% TNT are given in tables and graphically. (See also J. Chem. Soc., Feb., 1920.) —C. A. M.

Nitrotoluenes; Studies on the —. IV. The three-component system: p-nitrotoluene, 1,2,4-dinitrotoluene, 1,2,4,6-trinitrotoluene. J. M. Bell and C. H. Herty, jun. J. Ind. Eng. Chem., 1919, 11, 1128–1130.

IN a ternary mixture there are three freezing points; the first that at which the first solid appears, the second at which the second solid appears, and the third eutectic point, at which all three solids are in equilibrium with the melted mass or eutectic mixture. In the case of the primary and secondary freezing points in the above-mentioned ternary system, there was often great supercooling when DNT was the solid to be separated, and seeding with small crystals was then required. Curves have been constructed from the data obtained with different mixtures, each containing 10% DNT, with the percentages of TNT as abscissae, and these have been plotted on the conventional triangular diagram. By a method of interpolation both the composition and temperature of points on the boundary curves may be found. By this means it is possible to identify an unknown mixture of these three components, the primary and secondary freezing points, and then the identity of the solid phases being determined, and reference made to the diagrams to locate the composition point. (See also J. Chem. Soc., Feb., 1920.)—C. A. M.

Petroleum spirit; Preparation of trinitrotoluene from —. E. Berl and F. Ziffer. Petroleum, 1919, 14, 1213–1217. Chem. Zentr., 1919, 90, IV., 797.

A PETROLEUM spirit of Galician origin yielding by distillation about 90% between 85° C. and 125° C. gave on nitration about 9% of pure mononitrotoluene, corresponding to 6% of toluene in the petroleum spirit. As nitration of the petroleum spirit on the large scale was impracticable, extraction with liquid sulphur dioxide was tried. The extract obtained amounted to 18.5%, had sp. gr. 0.810, and could be separated by distillation on the large scale into fractions of higher and lower density. In this manner a toluene fraction, composed of 60% of toluene and 40% of petroleum spirit, was obtained, which, after being washed with sulphuric acid to remove olefines, was nitrated to mononitrotoluene. The yield of toluene from the crude oil amounted to 0.162%.—T. St.

Vessels for liquid air. Moser. See I.

Nitro-compounds. Margosches and Vogel. See XX.

PATENTS.

Explosives from smokeless powders; Manufacture of —. E. H. Blyth, Vienna. Eng. Pat. 135,813, 28.8.19. (Appl. 21,217/19.)

SMOKELESS powders may be converted into explosives suitable for blasting purposes by grinding them finely under water. Gelatinised nitroglycerin may be added.—W. J. W.

Cellulose preparations. Ger. Pat. 305,118. See V.

Removing picric acid stains. Ger. Pat. 312,772. See XII.

XXIII.—ANALYSIS.

Distillation in a high vacuum [; Laboratory apparatus for —]. J. Houben. Ber., 1919, 52, 1460–1462.

THE apparatus, which has a suitable attachment immersed in liquid air, is filled with carbon dioxide and evacuated by a water pump, and during the distillation the gas is allowed to enter through a fine capillary below the surface of the liquid to ensure steady boiling. A pressure of 0.12 mm. is easily maintained.—J. C. W.

Homogeneous liquid mixtures; Apparatus for rapid preparation of —. P. Jolibois. Comptes rend., 1919, 169, 1095–1098.

A SIMPLE apparatus is described and illustrated by means of which a homogeneous mixture may be obtained with two miscible liquids in 1/100th of a second. It consists essentially of two containers, one for each liquid, arranged so as to deliver the liquids at a uniform rate and in the requisite proportions, one to each of the inclined limbs of a Y-tube. The diameter of the Y-tube is 6.2 mm., and the lower vertical limb has a constricted portion 1.5 mm. in diameter extending for a distance of 2 cm. from the junction with the inclined limbs. The device is useful in analytical operations in which it is desired to keep constant the relative proportions of two interacting solutions.—W. G.

Gas volumes; Chart for the reduction of —. W. Ostwald. Z. angew. Chem., 1919, 32, 359–360.

A GRAPHICAL method for calculating the corrected gas volumes at 0° C. and 760 mm. pressure, with allowance for the vapour tension of water, is illustrated. The chart consists of a logarithmic scale for barometric pressure and a logarithmic scale for the correction factors. By laying a straight-edge between the respective points on these two scales, it is possible to plot a third scale for the corresponding temperatures, which takes the form of a straight line parallel to the other two. The temperature scale thus empirically determined includes the necessary correction for the tension of aqueous vapour. By another series of logarithmic scales the multiplication of the observed volume by the correction factor to give the corrected volume may be provided for on the same diagram, so that it is only necessary to shift the straight-edge from the point of the correction-factor scale, obtained by joining up the barometer and temperature values, across to the point on the observed volume scale in order to read off directly the corrected volume.—J. F. B.

Viscosity of organic colloids; Technique of the estimation of the —. E. Rothlin. Biochem. Zeits., 1919, 98, 34–92.

THE superiority of the "capillary" methods over the "damping" methods in the estimation of the

viscosity of fluids is discussed. The viscosity of various emulsoids under different pressures were determined by means of Hesse's viscometer (capillary principle). Emulsoids like gum arabic, soluble starch, β -gelatin, and casein have been found to obey Poiseuille's law. Others, like gelatin, egg albumin, insoluble starch, agar, and soap, have shown a deviation from this law as the pressure was diminished. The importance of the pressure factor and the dimensions of the capillaries in the determination of the viscosity of fluids is pointed out. Hesse's viscometer fulfils requirements in these respects.—S. S. Z.

Phosphoric acid; Grete's volumetric method [for the determination of] —. R. W. Tuinzing. Landw. Versuchs-Stat., 1919, 94, 191—195.

This method, in which the phosphoric acid is titrated with ammonium molybdate solution containing gelatin (this J., 1888, 771), was found to be trustworthy. Directions are given for the recovery of the ammonium molybdate from the collected titration solutions.—W. P. S.

Ferrous sulphide as an indicator in acidimetry, and a new volumetric method for the estimation of zinc. J. Houben. Ber., 1919, 52, 1613—1621.

MINERAL acids can be titrated with precision by adding a crystal or two of ferrous ammonium sulphate to the solution, passing in a little well-washed hydrogen sulphide, and running in alkali until the mixture just begins to darken. The end-point is somewhat indistinct in the titration of sulphuric acid by borax, but in this case it is possible to titrate back until the dark colour disappears. Zinc is completely precipitated by hydrogen sulphide from zinc sulphate if the solution is less than 0.2N, and from the chloride if the concentration is below 0.067N. Titration of the acid set free would be a means of estimating zinc, and for this purpose the above method is useful. The solution is saturated with hydrogen sulphide, a crystal of ferrous ammonium sulphate is added, and alkali is run in until the white turbidity becomes brownish. Examples of the application of the method to metallic zinc, zinc salts, zinc ash, and blends show that the process is superior to any other volumetric method, the results agreeing very well with those of gravimetric analyses. Of course, metals of group II. are first removed, and cobalt and nickel must be absent. (Compare J. Chem. Soc., 1920, ii., 53.)—J. C. W.

Iron; Volumetric estimation of —. J. Houben. Ber., 1919, 52, 2072—2076. (See preceding abstract.)

The method permits the estimation of ferrous and ferric iron and free mineral acid in a solution. A portion of the solution is completely reduced by hydrogen sulphide and titrated with N/1 alkali hydroxide solution until the dark coloration due to the incipient precipitation of ferrous sulphide is observed and does not disappear after vigorous shaking; the free acid plus one-third of the acid originally combined in the ferric salt is thereby estimated. A second portion of the solution is reduced by sulphur dioxide with complete exclusion of air; most of the excess of the reducing agent is expelled by boiling the solution, and the latter is titrated with N/1 alkali as before, after being cooled and treated with hydrogen sulphide, which completes the removal of sulphur dioxide and also serves as indicator. The free acid, plus two-thirds of the acid originally combined with the ferric iron is thus estimated. The difference between the two titrations multiplied by three gives the number of c.c. of N/1 ferric chloride solution originally

present. If this number is subtracted from the smaller of the two burette readings, the difference is the number of c.c. of N/1 acid which was uncombined in the solution. The ferrous iron is estimated in the usual manner. Test analyses show the method to have a high degree of accuracy.

—H. W.

Arsenic group; Separations in the —. W. Strecker and A. Riedemann. Ber., 1919, 52, 1935—1947.

THE following modification of the distillation method for the estimation of arsenic is recommended. The arsenic solution is placed in a flask provided with a dropping funnel and connected through an efficient worm-condenser to a vessel containing water; the flask is half filled with concentrated hydrochloric acid and potassium bromide (1.5 grms.) and a few pieces of porous pot are added. The contents of the flask are heated to gentle boiling, and 10 c.c. of thionyl chloride is gradually run in during the course of half an hour. The distillate is diluted with water to 700—800 c.c., boiled under a reflux condenser in a brisk current of carbon dioxide until sulphur dioxide is expelled and the sulphur has collected (this should be the case in half an hour at most), and filtered. Arsenic is estimated in the filtrate as the trisulphide. Phosphorus trichloride (25 c.c.) may replace the thionyl chloride, in which case the subsequent boiling of the filtrate is unnecessary, but, on the other hand, the presence of phosphorous acid in the residue often causes complications in subsequent estimations. The method gives accurate results and can be applied to the separation of arsenic from antimony, tin, copper, lead, mercury, and iron, and for the estimation of arsenic and iron in iron arsenide. The separation of antimony from tin can be effected by taking advantage of the fact that antimony chloride is volatile at 155°—165° C. from solutions in which the tin can be retained by the presence of phosphoric acid; the tin can be volatilised subsequently at a somewhat higher temperature if the restraining influence of the phosphoric acid is destroyed by the addition of hydrobromic acid. The authors have attempted to shorten the time required for this separation by utilising the catalysing action of hydrobromic acid from the commencement, but the results are unsatisfactory since tin distils with the antimony, the catalysing influence of hydrogen bromide outbalancing the restraining effect of phosphoric acid. On the other hand, the use of hydrobromic acid is very advantageous in the separation of antimony or tin alone from other elements and does not require the absence of nitric acid. Thus, in the separation of antimony from copper, the solution of the metals in hydrochloric acid is added to concentrated sulphuric acid (6 c.c.) and phosphoric acid (sp. gr. 1.78, 7 c.c.) contained in a distillation flask connected to a receiver containing concentrated hydrochloric acid; the contents of the flask are heated to 160° C., and a mixture of concentrated hydrochloric acid (10 vols.) and hydrobromic acid (sp. gr. 1.78, 1 vol.) is added at such a rate that the temperature remains constant. The antimony is completely removed in 30 mins. The separation of antimony from lead is similarly accomplished. Tin is quantitatively separated from copper and lead in an analogous manner; the solution of the metals in concentrated hydrochloric acid is treated with sulphuric acid (12 c.c.) and distilled at 160° C. with the regulated addition of the hydrochloric-hydrobromic acid mixture (20 c.c.). The process is complete in half an hour; the tin is precipitated from the distillate as the sulphide and weighed as oxide. The method can be applied to the estimation of tin in brass. The separation of antimony and tin from mercury cannot be effected in this

manner, since the latter is also partly volatilised. A novel type of filter tube is described, which is constructed by drawing out the end of a piece of wide glass tubing so as to form a stem; a filter disc covered with asbestos rests on the shoulder of the wider tube, which is further provided with a ground-in cap in which a capillary opening is made.

—H. W.

Molybdenum; Detection of — by means of xanthic acid. J. Koppel. Chem.-Zeit., 1919, 43, 777—778.

THE characteristic reaction between molybdenum compounds and xanthates, first described by Siewert (Z. ges. Naturw., 1864, 23, 5; see Malowan, this J., 1920, 27 A) is capable of detecting 0.00064 mgrm. of molybdenum in l.e.c. when applied in the following manner:—The cold, practically neutral, solution is mixed with a few particles of potassium xanthate and a few drops of a strong acid added. The yellow coloration due to the xanthic acid changes to red, and then to plum colour, in the presence of sufficient molybdenum. If a large amount of molybdenum is present black oily drops separate and adhere to the sides of the flask. The reaction product is stable for hours, but is rapidly destroyed by heat. A somewhat weaker reaction is produced when phosphoric, formic, or acetic acid is used for the acidification, whilst with salicylic acid it is very faint. With oxalic, citric, or tartaric acid little, if any, coloration is produced. The colouring matter may be readily extracted with an organic solvent, preferably chloroform, and the sensitiveness of the reaction thus increased. The compounds formed by metals such as copper, cobalt, nickel, and iron, do not interfere with the test for molybdenum, but vanadates, uranates, and tungstates may possibly interfere. Uric acid gives a pale yellow coloration and vanadic acid a brownish coloration, the coloured compounds in each case being soluble in ether, but the colour is not deep enough to mask the molybdenum reaction. Tungstic acid does give any definite coloration with xanthic acid, and does not interfere with the molybdenum test unless it is present in a very large preponderance, which, however, is frequently the case in technical work. In such cases the solution may be diluted so that the tungstic acid is not immediately precipitated on adding a slight amount of acid, or the tungstic acid may be precipitated by boiling the solution with acid, and the filtrate used for the xanthic acid test. An addition of sufficient phosphate or tartrate to keep the tungsten in solution before acidification will obviate the difficulty caused by the precipitation of the tungstic acid. (See also J. Chem. Soc., 1920, ii., 58.)—C. A. M.

Metals; Electrolytic determination of — without the use of an external current. M. François. Ann. Chim., 1919, 12, 178—192.

A MORE detailed account of work already published (see this J., 1918, 784 A).—W. G.

Inks; Use of commercial — in plant histology. P. Bugnon. Comptes rend., 1919, 169, 1051—1051.

TWO staining mixtures recommended for plant histological work are prepared by adding to a saturated solution of Soudan III. and Light-Green in 70% alcohol one-tenth of its volume of ink and by adding to 100 c.c. of a saturated solution of Soudan III. in 90% alcohol 0.5 gm. of Iodine Green and 5 c.c. of ink. With such mixtures a triple coloration is obtained, the pectocellulose membranes being stained violet by the ink, the cutinised and suberised membranes orange-red by the Soudan III., and the lignified membranes stained

green by either of the greens. The ink used is a commercial iron-tannin or iron-logwood black ink.

—W. G.

Dehydrogenation of hydrocarbons. Tausz and von Putuoky. See IIA.

Ignition temperatures. White and Price. See IIA.

Constituents of wood which give colour reactions. Wichelhaus. See V.

Ferri- and ferro-cyanides. Kolthoff. See VII.

Zirconium ore. Powell and Schoeller. See X.

Resins. Salvaterra. See XIII.

Turpentine. Taylor. See XIII.

Soil analysis. Münter. See XVI.

[*Fermentation*] glycerol. Rojahn. See XVIII.

Feeding stuffs. Neubauer. See XIXa.

Nitro-compounds. Margosches and Vogel. See XX.

Photographic papers. Kieser. See XXI.

PATENTS.

Pyrometric method and apparatus. E. F. Northrup, Princeton, Assignor to Pyroelectric Instrument Co., Trenton, N.J. U.S. Pat. 1,323,309, 2.12.19. Appl., 28.2.19.

IN a pyrometric apparatus, one end of a rod of "light-transmitting material" is subjected to the temperature to be measured, and the other end extends through a tube into, and is observable in, a light-excluding chamber. The temperature of the heated end is determined from the intensity of the light transmitted through the rod.—W. E. F. P.

Thermo-couple; Base-metal —. W. C. Heraeus G.m.b.H., Hanau. Ger. Pat. 302,452, 8.5.17.

A THERMO-COUPLE has its two arms constructed of alloys of nickel with 2—6% and 5—12% of chromium respectively. If alloys of nickel with 3.19% and 7.41% of chromium are employed, using the purest metals obtainable, the indications of the instrument diverge by a maximum of -15° from those of the Pt—Pt Rh couple between 0° and 900° C., and of $+15^{\circ}$ between 950° and 1150° C., while at 950° C. the two couples agree exactly.—L. A. C.

Gas and vapour mixtures; Determination of ingredients which react exothermally in — [e.g., oxygen in hydrogen]. Badische Anilin und Soda Fabrik, Ludwigshafen. Ger. Pat. 303,986, 24.3.17.

THE gas current is divided and passes through two equally heated tubes containing similar thermo-elements which are capable of acting as catalysts for the ingredients of the gas mixture. The thermo-element in one tube is exposed to the gas current, whilst that in the other tube is protected by quartz or other suitable material. The rise in temperature of the exposed thermo-element is measured electrically. The apparatus is suitable for the estimation of traces of oxygen in electrolytically prepared hydrogen.—T. St.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

- Bennett. Apparatus for diffusing fluids for evaporating, cooling, mixing, etc. 715. Jan. 9.
 Blagden and others. 32,567. *See* XII.
 Bowden and McNaught. Means for raising liquids or mixtures of liquids and solids. 856. Jan. 10.
 Breakell. Grinding and crushing mills. 32,628. Dec. 30.
 Brinjes and Goodwin, and Seaman. Roller grinding mills. 32,121. Dec. 22.
 Brown and Urquhart. Centrifugal filters, strainers, separators, etc. 551. Jan. 7.
 Bucci. Cooking-ovens or drying apparatus. 27. Jan. 2.
 Buckley. 32,332. *See* XIX.
 Butters and Co., and Memmell. Apparatus for indicating density of liquids and automatically grading same accordingly. 32,416. Dec. 24.
 Candlot. Furnaces, ovens, etc. 32,573. Dec. 29. (Fr., 10.7.17.)
 Delaygue. Solution for use with apparatus for refrigerating by liquefaction. 367. Jan. 5.
 Diver. Process of ground distillation. 32,165. Dec. 23.
 Ellis. Distillation apparatus. 698. Jan. 8.
 Frazer and Sealione. Catalysts, and processes of making them. 575. Jan. 7.
 Henning. Solvents for extraction purposes. 32,433. Dec. 24.
 Imray (Ruggles-Coles Engineering Co.). Drier. 32,852. Dec. 31.
 Jones and McKean. Liquid-fuel furnaces. 32,461. Dec. 29.
 Kirk. Heat-treating furnace. 32,429. Dec. 24.
 Lovelace, Miller, and Patrick. Separating gases. 32,408. Dec. 24. (U.S., 28.12.18.)
 Manlove, Allott and Co., and Mason. Oil-fired furnaces. 711. Jan. 8.
 Mitchell. Apparatus for transferring material and for separating mixtures etc. 594. Jan. 7.
 Paget. Grinding, refining, or reducing apparatus. 169. Jan. 2.
 Pirrie. 74. *See* V.
 Raworth (Soc. Anon. des Appareils de Manutention et Fours Stein). Oil-burning furnaces. 32,796. Dec. 31.
 Rosanoff Process Co. Fractionating apparatus. 32,582. Dec. 29. (U.S., 23.12.18.)
 Soc. Anon. d'Exploit de Proc. Evaporatoires Système Prache et Bouillon. Evaporating apparatus. 32,140. Dec. 22. (Fr., 24.12.18.)
 Thoens. Refrigerating. 32,291. Dec. 21.
 Wilton. Centrifugal machines. 905. Jan. 10.
 Wright. Means for supplying liquid, powdered, etc. substances to furnaces etc. Jan. 2.

COMPLETE SPECIFICATIONS ACCEPTED.

- 8529 (1918). Gallot et Cie. *See* XI.
 14,617 (1918). Ely. Lining of furnaces. (136,860.) Jan. 7.
 16,758 (1918). Steiger and Steiger. Gas-fired shaft kilns for burning and roasting non-sintering substances. (136,588.) Dec. 31.

- 21,060 (1918). Harris. Drying-apparatus. (136,631.) Dec. 31.
 21,085 (1918). Odum. Atomising materials in a melted state. (122,103.) Dec. 31.
 21,286 (1918). Shaw. Apparatus for evaporating or concentrating liquids. (136,651.) Dec. 31.
 21,452 (1918). Hughes. Methods and apparatus for carrying out certain chemical reactions. (136,896.) Jan. 7.
 3065 (1919). Taylor. Vacuum drying-ovens. (136,703.) Dec. 31.
 5220 (1919). Chaudière. Shaft-furnaces and gas-producers. (137,168.) Jan. 11.
 12,588 (1919). Coles, and Allen and Co. Apparatus for mixing, circulating, and agitating materials in a liquid or semi-liquid state. (136,758.) Dec. 31.
 14,219 (1919). Corbett. Grinding-mills of the rotating disc type. (137,231.) Jan. 14.
 21,228 (1919). Gerlach. Drums for drying materials. (133,327.) Jan. 7.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

- André. Recuperation of combustible material contained in ashes from furnaces etc. 32,709. Dec. 30.
 Barber. Apparatus for drying and filtering illuminating gases. 747. Jan. 9.
 Bateman. Continuous carbonisation of coal etc. by inclined retorts. 32,438. Dec. 29.
 Bates. Fuel. 32,048. Dec. 22. (U.S., 5.8.19.)
 Berg. Distilling and refining volatile substances and carbonising and coking the same. 32,096. Dec. 22. (Fr., 21.12.18.)
 Crowther. Spent-oxide burner. 728. Jan. 9.
 Gardner, and Woodall, Duckham, and Jones. Vertical retorts for carbonising coal. 32,220. Dec. 23.
 Green and Leek. Methods of revivifying material in gas-purifiers. 32,073. Dec. 22.
 McComb. 32,309. *See* III.
 Meade. Distillation of coal etc. 288. Jan. 5.
 Paris. Cleaning and refining distillates of petroleum. 70. Jan. 2.
 Pasley. Gas-producers. 32,602. Dec. 30.
 Robinson. Retort setting for complete gasification of coal. 526. Jan. 7.
 Rütgerswerke A.-G. Production of soot-carbon, retort-graphite, etc. from natural gas. 32,380. Dec. 24. (Ger., 12.6.18.)
 Smith and Tulloch. Gas-producers. 32,071. Dec. 22.
 Smith. Distillation of coal etc. 288. Jan. 5.
 Stephens. Preparation of carbonaceous materials in the form of briquettes. 303. Jan. 5.
 Teague. Production of pure carbon. 32,236. Dec. 23.

COMPLETE SPECIFICATIONS ACCEPTED.

- 1159 (1918). Comp. pour la Fabr. des Compteurs et Matériel d'Usines à Gaz. Manufacture of water gas. (113,271.) Jan. 7.
 13,414 (1918). Hughes, and Hill-Jones, Ltd. Manufacture of artificial fuel. (136,584.) Dec. 31.
 14,176 and 19,177 (1918). Burney. Apparatus for distilling solid carbonaceous material. (136,585.) Dec. 31.
 19,083 (1918). Merz and McLellan, Michie, and Weeks. Low-temperature distillation of fuel. (136,868.) Jan. 7.
 20,971 (1918). Eastick and Eastick. *See* XVII.
 21,239 (1918). Davies. Carbonisation of fuel by vertical coke-ovens etc. (136,880.) Jan. 7.

- 21,637 (1918). Whiteher, Hiller, and Hewitt Construction Syndicate. Appliances for steaming gas retorts. (136,910.) Jan. 7.
 5220 (1919). Chaudière. *See I.*
 14,105 (1919). Thompson (Fours et Procédés Mathy Soc. Anon.). Coke ovens. (137,005.) Jan. 7.
 22,433 (1919). Eyre. Fuel. (136,790.) Dec. 31.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

- Adam and Legg. Reduction of nitro-compounds. 32,351. Dec. 24.
 Barker. Tar-distilling plant. 32,059. Dec. 22.
 British Dyestuffs Corporation, Green, and Porter. Manufacture of phthalic acid and anhydride. 862. Jan. 10.
 Commin. Treating pitch. 79. Jan. 2.
 McComb. Process of converting liquid hydrocarbons. 32,309. Dec. 24.

IV.—COLOURING MATTERS AND DYES.

COMPLETE SPECIFICATION ACCEPTED.

- 10,797 (1919). Thompson (Poulenc Frères, and Meyer). Manufacture of 3,6-diaminoacridine. (137,214.) Jan. 14.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

- Brydon, Forster, and Turner. Treatment of paper etc. for electrical, chemical, etc. purposes. 60. Jan. 2.
 Colebrook, Langlois, Moncorget, and Tebbitt. Retting textile plants. 32,473. Dec. 29.
 Distillates, Ltd., and Jarman. Treatment of wool or piece scouring liquors or effluents. 32,486. Dec. 29.
 Great Northern Paper Co. Paper-making machines. 32,349. Dec. 24. (U.S., 31.1.17.)
 Great Northern Paper Co. Making paper. 706. Jan. 8. (U.S., 26.3.18.)
 Howden. Apparatus for drying coated or enamelled paper, waterproofed cloth, etc. 32,787. Dec. 31.
 Langwell and others. 694. *See XVIII.*
 Pirrie. Drying fibrous, granular, pulpy, etc. materials. 74. Jan. 2.

COMPLETE SPECIFICATIONS ACCEPTED.

- 12,188 (1917). Korselt. Increasing the strength and elasticity and preventing or decreasing subsequent weakening of the fibres of loaded silk. (136,578.) Dec. 31.
 21,790 (1918). Skinner. Preparation of pulp or fibrous material for making paper, papier-mâché, etc. (137,105.) Jan. 14.
 9576 (1919). De Cew and Marx. Treatment of paper pulp. (137,207.) Jan. 14.
 19,298 (1919). Clayton. Apparatus for making artificial silk. (136,784.) Dec. 31.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

- Baldwin and Partners, and McIntosh. Bleaching wool etc. 645. Jan. 8.

- Burgess, Ledward, and Co., and Harrison. Dyeing artificial silk. 722 and 723. Jan. 9.
 Cole and Kershaw. Machines for dyeing, mordanting, bleaching, scouring, etc. wool, yarn, etc. 32,733. Dec. 31.
 Heap. Rotary perforated beam apparatus for dyeing etc. yarn. 511. Jan. 7.

COMPLETE SPECIFICATIONS ACCEPTED.

- 21,864 (1918). Lord and Lord. Machines for dyeing, washing, scouring, sizing, mercerising, and bleaching yarn in the hank form. (136,666.) Dec. 31.
 4229 (1919). Morley, and Bleachers' Assoc. Machines for bleaching and similarly treating textile fabrics and yarns in rope form. (136,713.) Dec. 31.
 5788 (1919). Driver and Sunderland. Apparatus for testing colour fastness in dyed fabrics. (136,971.) Jan. 7.
 6926 (1919). Samuel. Machines for printing fabrics or the like. (126,948.) Jan. 7.
 9108 (1919). Davis and Davis. Dyeing-machines. (136,991.) Jan. 7.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

- Ashcroft. Nitrogen fixation and manufacture of nitrogen compounds. 122. Jan. 2.
 Craig. Recovering potash etc. salts from blast-furnace gases. 839. Jan. 10.
 Datta. Manufacture of alkali bichromates and production of sulphates. 309. Jan. 5.
 Douglas. Manufacture of ammonia. 32,756. Dec. 31.
 Fabr. de Prod. Chim. de Thann et de Mulhouse. Manufacture of potassium sulphate. 32,568 and 32,672. Dec. 29 and 30. (Fr., 28 and 30.12.18.)
 Fredriksstad Elektroemiske Fabriker Aktieselskabet. 32,886. *See XI.*
 Fredriksstad Elektrokemiske Fabriker Aktieselskabet. Manufacture of perborates. 489. Jan. 6. (Norway, 4.3.19.)
 Golding, and United Alkali Co. Manufacture of ferrous sulphide and sulphuretted hydrogen. 32,112. Dec. 22.
 Heberlein. Method for electrolysing a solution of nickel salt. 32,853. Dec. 31.
 Jones and Kelly. Preparation of sodium pentaborate etc. direct from boron ores. 648. Jan. 8.
 Kelly. Manufacture of acid sodium pyrophosphate. 353. Jan. 5.
 Loring. Electrodes for oxidising nitrogen. 32,763. Dec. 31.
 Mackay. Obtaining strontium and barium sulphates from their native sulphates or carbonates. 822. Jan. 9.
 Mackay. Obtaining copper sulphate direct from metallic copper. 823. Jan. 9.
 Napp (Tschudi). Manufacture of hydrobromine from hydrogen and bromine. 433. Jan. 6.
 Norsk Hydro-Elektrisk Kvaestofaktieselskab. Production of concentrated nitrous gases. 32,397. Dec. 21. (Norway, 21.10.16.)
 Officine Elettrochimiche Dr. Rossi, and Toniolo. Production of hydrogen and mixtures of hydrogen and nitrogen. 32,414. Dec. 24. (Italy, 21.10.19.)
 Officine Elettrochimiche Dr. Rossi, and Toniolo. Production of hydrogen. 366. Jan. 5. (Italy, 29.10.19.)
 Shah. Manufacture of zinc chloride. 32,717. Dec. 30.
 Sutcliffe. Apparatus for hydrating lime etc. 32,144. Dec. 22.
 Thibault. Conversion of metallic lead into its oxides. 32,539. Dec. 29. (Fr., 30.12.18.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 4562 (1919). Fairbrother (Chemical Construction Co.). Apparatus for recovering potassium compounds. (136,718.) Dec. 31.
 6928 (1919). Golding, Raschen, and United Alkali Co. Purification of calcium citrate and manufacture of citric acid. (136,979.) Jan. 7.
 10,188 (1919). Jackson (Air Reduction Co.). Manufacture of cyanides. (136,750.) Dec. 31.
 14,439 (1919). Hedstrom. Production of compounds of radium. (136,768.) Dec. 31.
 15,560 (1919). Thorssell and Lundén. Production of cyanides. (136,772.) Dec. 31.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

- British Thomson-Houston Co. (General Electric Co.). Substances for coating glass surfaces. 674. Jan. 8.
 Kent. Manufacture of glass tubing having more than one bore. 50. Jan. 2.
 Kent. Manufacture of thermometer tubing. 51. Jan. 2.

IX.—BUILDING MATERIALS.

APPLICATIONS.

- Knapp and Slade. Fuming oak etc. 185. Jan. 2.
 Smidth & Co. Building material for concrete structures. 189. Jan. 2. (Denmark, 1.5.18.)
 Smidth & Co. Method of making concrete. 641. Jan. 8. (Denmark, 8.1.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 4771 (1918). Jones (White Heat Products Co.). Manufacture of artificial stones. (136,580.) Dec. 31.
 185 (1919). Gladman. Manufacture of wood substitutes. (137,115.) Jan. 14.
 5358 (1919). Frydenland. Production of porous building and insulating bodies. (123,992.) Jan. 7.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

- Brettschneider and Schäfer. Case-hardening mixtures for iron and steel. 32,093. Dec. 22.
 Craig. 839. *See* VII.
 Edser and others. 32,376. *See* XII.
 Johanson and Stönen. Treating weakly magnetic iron ore containing sulphides as secondary constituents. 32,571. Dec. 29.
 Kirk. 32,429. *See* I.
 Loring. Method of altering electrical conductivity of metals. 32,764. Dec. 31.
 Mackay. Obtaining iron and tin from tin scrap. 821. Jan. 9.
 Metallindustrie Schiele und Bruchsalder. Production of aluminium castings. 65. Jan. 2. (Ger., 18.2.18.)
 Quinoneri. Magnetic separators for treating ferrous ores. 32,409. Dec. 24.
 Ramen. Roasting sulphur ores containing low percentage of sulphur. 589. Jan. 7.
 Ramen. Roasting furnaces. 691. Jan. 8.
 Redfern (Guthridge, Ltd.). Ore-concentrating tables. 32,133. Dec. 22.
 Strasser. Deoxidising aluminium and its alloy. 32,363. Dec. 24. (Switz., 9.4.19.)
 Strasser. Deoxidising copper and its alloy. 32,364. Dec. 24. (Switz., 26.4.19.)
 Wright. Smelting or reduction of metallic ores etc. 22. Jan. 2.

COMPLETE SPECIFICATIONS ACCEPTED.

- 9215 (1917). Head. Separating fusible tungstates from the gangue resulting from the decomposition of the ore. (136,577.) Dec. 31.
 10,819 and 19,318 (1918). Sulman and Ballantine. Manufacture of alloys. (137,080.) Jan. 14.
 3066 (1919). White (Byers Co.). Puddling iron. (136,704.) Dec. 31.
 3915 (1919). Mackay. Drying blast-furnace air. (136,957.) Jan. 7.
 8890 (1919). Wellman, Seaver, and Head, and Gray. Steel-making and like furnaces or mixers. (137,200.) Jan. 14.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

- Allison (Narabayashi). Filling for storage batteries. 638. Jan. 8.
 Böcker and Eichhoff. Galvanic elements. 32,700 and 32,701. Dec. 3.
 Brydon and others. 60. *See* V.
 Bültemann. Manufacture of electric insulating-material. 66. Jan. 2. (Ger., 29.1.18.)
 Cayrol and Odion. Electric battery. 64. Jan. 2. (Fr., 25.4.14.)
 Cuthbertson. Insulating material. 25. Jan. 2.
 Fredriksstad Elektrokemiske Fabriker Aktiefelskabet. Apparatus for electrolysis of metal salt. 32,886. Dec. 31. (Norway, 13.2.19.)
 Green. Electric accumulators. 467. Jan. 6.
 Heberlein. 32,853. *See* VII.
 Jackson (Allen Electrolytic Cell Corporation). Electrolytic cells. 363. Jan. 5.
 Levin. Electrolytic cells. 32,789—32,792. Dec. 31.
 Levin. Electrodes. 32,793. Dec. 31.
 Lovin. Electrolytic apparatus. 32,794. Dec. 31.
 Loring. 32,763. *See* VII.
 Wade (Booth Electric Furnace Co.). Electric furnaces. 811. Jan. 9.

COMPLETE SPECIFICATIONS ACCEPTED.

- 8529 (1918). Gallot et Cie., and Poussin, Rondeaux, et Cie. Apparatus for electrical separation of substances in suspension in gases. (116,101.) Jan. 14.
 10,454 (1918). Elektro-Osmose A.-G. Electrodes for electro-osmotic processes. (135,816.) Jan. 14.
 10,457 (1918). Elektro-Osmose A.-G. Apparatus for electro-osmotically removing liquids from materials. (135,818.) Jan. 7.
 364 (1919). Armour Fertilizer Works. Electric furnaces. (122,828.) Jan. 7.
 4320 (1919). Watson and Co., Greaves, and EtcHELLS. Electric furnaces. (136,960.) Jan. 7.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

- Blagden, and Howards and Sons. Manufacture of catalysts for hydrogenation, and application of such catalysts. 32,567. Dec. 29.
 Edser, and Minerals Separation, Ltd. Treatment of froths containing fatty matter etc. 32,376. Dec. 24.
 Ellis (Codal). Decomposition of fatty acid glycerides by sulpho-aromatic decomposing agents. 902. Jan. 10.
 Green. 791. *See* XIX.
 Mitchell. Apparatus for making emulsions. 32,639. Dec. 30.
 Townsend. Soap. 599. Jan. 7.

COMPLETE SPECIFICATIONS ACCEPTED.

- 20,425 (1918). MacIlwaine and Holdcroft. Extraction of oil by volatile solvents. (136,870.) Jan. 7.

19,246 (1919). Danin. Reducing solid fats to liquid. (137,252.) Jan. 14.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Baker. Process of making pigments. 469. Jan. 6.
Koppers Co. Paints or varnishes, and methods of making same. 32,718. Dec. 30. (U.S., 18.1.19.)
Melamid. Manufacture of resin-like bodies. 32,553, 32,551, and 32,559. Dec. 29. (Ger., 28 and 29.12.18.)
Melamid. Manufacture of resin-like substances. 890. Jan. 10. (Ger., 12.5.19.)
Thibault. 32,539. *See* VII.

COMPLETE SPECIFICATION ACCEPTED.

467 (1919). Titanine, Ltd., and Bowles. Manufacture of varnishes or dopes. (136,927.) Jan. 7.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Farrel Foundry and Machine Co. Mixing or masticating rubber etc. 799. Jan. 9. (U.S., 13.1.19.)
Fuller. Ebonite. 32,781. Dec. 31.
Gare. Heat treatment of rubber, ebonite, etc. 748. Jan. 9.
Smith. Devulcanising rubber. 32,259. Dec. 24.
Stevens. Vulcanising rubber and treating vulcanised rubber. 7. Jan. 2.

COMPLETE SPECIFICATION ACCEPTED.

4330 (1919). Peachey. Accelerating the vulcanisation of caoutchouc or caoutchouc-like substances. (136,716.) Dec. 31.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Adachi and Nakayama. Utilisation of waste leather. 537. Jan. 7.
Cross and Ingle. Finishing leather. 32,128. Dec. 22.
Handke. Waterproof coatings for leather etc. 32,837. Dec. 31.
Melamid. Manufacture of artificial tanning-substances. 63. Jan. 2. (Ger., 24.12.18.)

XVI.—SOILS; FERTILISERS.

COMPLETE SPECIFICATION ACCEPTED.

5477 (1919). Radmann. Fertiliser and method of producing same. (137,171.) Jan. 14.

XVII.—SUGARS; STARCHES; GUMS.

COMPLETE SPECIFICATION ACCEPTED.

28,971 (1918). Eastick and Eastick. Manufacture of decolorising vegetable carbon. (136,873.) Jan. 7.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Langwell, and Power Gas Corporation. Fermentation of cellulose. 694. Jan. 8.
Townsend. Improving fermentation and flavouring of fermented products. 32,245. Dec. 23.

COMPLETE SPECIFICATION ACCEPTED.

1845 (1919). Robinson. Preservation of yeast. (137,131.) Jan. 14.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Bollmann. Manufacture of foodstuffs. 32,839. Dec. 31.
Buckley. 32,332. *See* I.
Campbell. Margarine etc. 583. Jan. 7.
Candy. Filtration of water. 843. Jan. 10.
Evans and Ormes. Manufacture of casein, albumin, etc. from vegetable etc. products. 349. Jan. 5.
Faulkner. Dietetic material. 32,250. Dec. 23.
Forget-Me-Not Flours, Ltd., and Hutchinson. Manufacture of alimentary products containing water-soluble vitamins. 32,239. Dec. 23.
Green. Manufacture of margarine. 791. Jan. 9.
Howles and McDougall. Sheep-dips etc. 570. Jan. 7.
Jeacock. Baking-powder, self-raising flour, etc. 32,623. Dec. 30.

COMPLETE SPECIFICATIONS ACCEPTED.

16,735 and 16,736 (1917). Marks (Hildebrandt). Treatment of nitrogenous animal waste. (136,853 and 136,854.) Jan. 7.
15,904 (1919). Yamamoto and others. Deodorising and decoloring bean flour. (136,775.) Dec. 31.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Blagden, and Howards and Sons. 32,567. *See* XII.
Dupont, and Darrasse Frères et Cie. Manufacture of valerianic acid and alkaline valerianates. 32,368. Dec. 24. (Fr., 3.12.18.)
Leysmith and Mackintosh. Treatment of tobacco. 321. Jan. 5.
Schrader's Son. Ethyl chloride tubes. 175. Jan. 2. (U.S., 2.1.19.)
Weiss. Manufacture of oxyaldehydes and their ethers. 443. Jan. 6. (Fr., 18.2.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

10,797 (1919). Thompson (Poulenc Frères). *See* IV.
16,489 (1919). Marks (Union Carbide Co.). Producing chlorination products of propylene. (137,247.) Jan. 14.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATION.

Cox. Photographic plates. 608. Jan. 8.

XXII.—EXPLOSIVES; MATCHES.

APPLICATIONS.

Bielouss. Conversion of high explosives. 475. Jan. 6. (U.S., 6.1.19.)
Hedberg. Manufacture of explosives. 32,690. Dec. 30.
Lowndes. Explosives. 465. Jan. 6.

XXIII.—ANALYSIS.

APPLICATION.

Davis and Rosling. Apparatus for analysing gases. 32,697. Dec. 30.

COMPLETE SPECIFICATION ACCEPTED.

9136 (1919). Boulton. Absorption apparatus for analysing gases. (136,992.) Jan. 7.

I.—GENERAL; PLANT; MACHINERY.

Contact catalysis; Further problems in —. H. S. Taylor. Trans. Amer. Electrochem. Soc., 1919, 167—173. [Advance proof.]

SEVERAL features of the phenomenon of contact catalysis in cases where carrier action or formation of intermediate compounds does not take place, are discussed by the author. Most contact agents have a porous or finely divided structure, e.g., bog iron ore, firebrick, platinum deposited on asbestos, etc., and platinum gauze, which acquires activity in use, is found to have developed a corrugated, pitted surface. Although these porous materials are also adsorbent, yet the two phenomena of catalysis and adsorption are quite distinct. Thus, although charcoal shows marked adsorptive action towards carbon monoxide, oxygen, and carbon dioxide, its activity in promoting the oxidation process is practically nil compared with metallic oxides. On the other hand, ethylene and oxygen react at ordinary temperatures in presence of charcoal. The differentiations may be due to the relative adsorptive capacities of charcoal and metallic oxides in one case, and preferential adsorption of particular gases by charcoal in the other. Data in regard to the amounts of gases which may be adsorbed separately or from a mixture are still scanty. The effect of poisons on catalytic agents may be explained by their interference with the adsorptive properties of the catalyst, but the time factor or velocity of adsorption must be taken into consideration, and since the normal time of contact is extremely short, the effect of the poison may be due to a reduction of the velocity with which adsorption occurs. Charcoal used for gas masks gave very different results in the "clean" and "unclean" condition. The adsorption curves indicate that although the ultimate amount adsorbed would be the same, yet the clean charcoal has a much higher velocity of adsorption in the early stages. The nature of the adsorbent is important, and an alteration of its chemical reactivity influences its catalytic activity. Copper oxide wire is reduced by hydrogen at 300° C., but if the copper is oxidised at a lower temperature, subsequent reduction will occur at a temperature as low as 100° C., and such copper oxide will also catalyse hydrogen and oxygen at 100° C. The increase of catalysis by promoters, such as small quantities of chromium oxide added to iron oxide, has not been fully accounted for.—W. J. W.

Heterogeneous catalysis; Reactivity and adsorption in —. E. K. Rideal. Trans. Amer. Electrochem. Soc., 1919, 203—211. [Advance proof.]

THE mechanism of heterogeneous catalysis involves investigations as to the way in which the medium in juxtaposition to the catalyst differs from that in the free space and of the effect on reaction velocity caused by alteration in the state of the medium. Adsorption is now regarded as the primary action in catalysis, and surface adsorption is frequently accompanied by intra-solid diffusion, resulting in formation of solid solutions within the body of the solid material, but since catalytic action is proportional to the area of the catalytic material, intra-diffusion plays a small part. Divergent opinions prevail in regard to the surface film in which catalytic reactions take place. Three theories have been expounded to explain why quasi-chemical combination between reacting substances and catalytic material accelerates a reaction which proceeds but slowly when chemical combination alone occurs. Fink and Bodenstein assume that every molecule penetrating the diffusion layer to the catalyst surface reacts, the rate of catalysis being governed by

the rate of diffusion of the reacting substances or of the products. According to the single layer theory, only those molecules of the reacting substances undergo chemical combination which strike the catalyst in juxtaposition to one another, the rate of catalysis being usually governed by the rate of evaporation of the products from the surface. The hypothesis of the critical energy increment assumes that molecules in juxtaposition only react when they attain a certain critical energy.

—W. J. W.

Steam; Effect of the oil present in exhaust — upon the efficiency of evaporators in sugar factories. H. Claassen. Z. Ver. deut. Zuckerind., 1919, 128—135.

EXAMINATION of the vapour space of an evaporator heated by exhaust steam, which after leaving the engines had been passed through an oil separator, showed that at the end of the season only those tubes immediately opposite and contiguous to the steam inlet had become contaminated with oil, and that the average thickness of the layer did not exceed 0.005 mm. Owing to the small proportion of the total heating surface of the evaporator thus affected, the efficiency of the apparatus had not been diminished appreciably under the conditions obtaining in the test.

—J. P. O.

Colours; Heat-reflecting properties of — applied to oil and gas storage tanks. H. A. Gardner. Paint Manufacturers' Assoc., U.S.A., Circ. No. 44. Jan., 1917. 2 pages.

THE effect produced by the calorific rays on surfaces painted in various colours was determined with a view to application of the results to storage tanks containing volatile distillates. Determination of the rise in temperature of the contents of small cylindrical metal tanks containing benzine (petroleum spirit), the exterior surfaces of which were painted in various colours, were made after 15 minutes' subjection to the rays of a powerful arc. Gloss paints were selected on account of their lesser absorption of thermal rays than those presenting a matt surface. A container of tin plate (unpainted) and one painted with aluminium paint gave the lowest recorded rises in temperature, i.e., 19.8° F. and 20.5° F. (11° C. and 11.4° C.) respectively, but white and light-coloured paints giving rises of 22.5° F. and 23°—26.6° F. (12.5° C. and 12.8°—14.8° C.) are to be preferred from the point of view of their serviceability and the protection they afford. With a black paint a rise of 51° F. (30° C.) was recorded, whilst red oxide paint showed a rise of 29.7° F. (16.5° C.).—A. de W.

Thermo-couple installation. Williamson and Roberts. See VIII.

PATENTS.

Refrigerating apparatus. Apeldoornsche Machinefabriek en Metaalgieterij voorheen Loog Landaal, Apeldoorn, Netherlands. Eng. Pat. 125,358, 21.11.18. (Appl. 19,106/18.) Int. Conv., 8.4.18. Addition to 120,923 (this J., 1920, 51 A).

THE compression cylinder is surrounded by a jacket from which the high-pressure vapour enters the cylinder through a ring of small openings formed so that their cross-section grows wider in the direction of the jacket, and which are unclosed by the piston at the end of the suction stroke. The high-pressure vapours enter the jacket from a main through an opening whose cross-section also grows wider in the direction of the jacket. The intermediate receivers and the liquid separator are provided with inlet and outlet openings of a similar conical form.—W. H. C.

Refrigerating apparatus. G. A. Wegner, Rochester, N.Y., Assignor to Wegner Machine Co., Buffalo, N.Y. U.S. Pat. 1,325,128, 16.12.19. Appl. 13.5.18.

In a refrigerator employing a volatile liquid, the supply of liquid is maintained at a predetermined pressure, and the discharge of vapour is controlled by a valve governed by the temperature of the issuing gas, a temperature above the saturation point being used.—B. M. V.

Distillation; Apparatus for fractional —. E. A. R. Chenard, Cognac, France. Eng. Pat. 130,992, 5.8.18. (Appl. 19,318/19.) Int. Conv., 5.8.18.

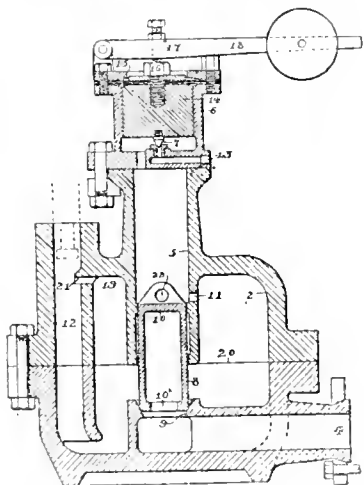
The vapours from the still pass first through a series of concentric pipes, where heat interchange takes place between the ascending vapours and the returning condensed liquid. The vapours next pass through a bent pipe into a series of doubly conical vessels, each provided near the outlet end with a scrubber or filter-box charged with finely-divided material to help the separation of the condensed liquid, which is returned to the bent pipe by tubes provided with siphons and taps to enable part of the liquid to be withdrawn. The vapour passes from the last of the conical vessels to an ordinary condenser.—W. H. C.

Air filters; Bag cleaning devices for —. W. F. L. Beth, Lübeck, Germany. Eng. Pat. 132,513, 9.9.19. (Appl. 22,128/19.) Int. Conv., 9.9.18.

TUBULAR filter bags used for filtering air are cleaned by repeatedly slackening and tightening them, the bags being held taut during the filtering period. A device consisting of levers and two cams effects this: one slowly rotating cam determines the periods of cleaning and stationary filtering, and the other cam, rotating faster, provides the slackening and tightening effect.—B. M. V.

Raising liquids by direct fluid pressure; Apparatus for —. J. E. L. Ogden, Liscard. Eng. Pat. 135,900, 26.11.18. (Appl. 19,416/18.)

THE liquid to be raised flows into the vessel, 2, from a tank placed at a higher level, passes through the pipe, 4, lifts the valve, 8, off its seat, 9, and fills



the vessel, 2, driving out the air through the opening, 19, into the rising main, 12. When the liquid rises to the level of the opening, 11, in the chamber, 5, it flows through and traps the air in 5, and at the same time compresses it slightly. This causes the dead weight, 14, fastened at the upper end to the diaphragm, 15, which closes the box, 6, to lift

until the projection, 16, comes into contact with the stud, 17. The air-inlet valve, 7, lifts slightly and compressed air enters by the pipe, 13, and the pressure beneath the weight increases until it overcomes the resistance of the lever and weight, 18, when the valve, 7, opens wide and allows the compressed air to enter freely. The pressure of the air forces down the valve, 8, on to its seat and drives the liquid up the rising main, 12. The air vent, 19, is provided with a projection, 21, against which the liquid impinges, and this helps to seal the opening, 19, until all the liquid is forced out of the vessel, 2; when this has taken place the air escapes up the rising main, the pressure in the vessel falls, the valve, 7, closes and 8 is opened, and the operation recommences.—W. H. C.

Grinders; Impact —. W. S. and G. S. Barron, Gloucester. Eng. Pat. 135,955, 6.12.18. (Appl. 20,253/18.)

THE grinding chamber is divided into two compartments by a rotating disc which carries impact studs on both sides. The material is fed into the first compartment, where it receives a preliminary grinding and then passes through a passage to the second compartment on the other side of the disc where it receives a final grinding, the finely-ground material being discharged through a peripheral screen. Any particles too coarse to pass through the screen are returned by way of the conduit between the compartments to the grinding portion of the second compartment, and are not ground against the screen, so that the life of the latter is prolonged.—W. H. C.

Filter-presses. R. W. Webster, London. Eng. Pat. 135,976, 17.12.18. (Appl. 21,057/18.)

In filter-presses of the type in which the liquid to be filtered passes through layers of filtering material, supported by a series of frames, connected with common inlet and discharge pipes, one or more preliminary filter surfaces are provided through which the liquid is passed first so that it may receive a preliminary filtration before it reaches the main filter surface. At first the liquid is fed through the preliminary filters to the main filter, but when these become clogged the flow is diverted by means of suitable valves directly to the main surface. In this way the frequency with which the press has to be cleaned is lessened.—W. H. C.

Liquids; Apparatus for separating solid particles of matter from —. W. Acton, Paisley. Eng. Pat. 135,992, 30.12.18. (Appl. 21,756/18.)

AN endless band of filter cloth is arranged to pass round a drum, which has its surface divided by transverse partitions into separate suction chambers each connected separately to the hollow shaft, then over a breaking roller to discharge the deposit, and back to the drum, against the surface of which it is pressed by a spring guide roller. The drum is rotated, partly immersed, in a tank which contains the liquid to be filtered, and the hollow shaft is connected to a vacuum pump, and is also provided with a valve by which the suction is cut off from that suction chamber the surface of which is exposed whilst the cloth is passing over the breaking roller. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 19,600 of 1890, 2257 of 1891, 1591 of 1892, 9956 and 22,871 of 1898, 19,237 and 22,852 of 1901, 10,071 of 1904 and 120,055; this J., 1899, 120, 498; 1901, 1194; 1902, 243; 1919, 24 A.)—W. H. C.

Gas furnaces. South Metropolitan Gas Co., and D. Chandler, London. Eng. Pat. 136,356, 28.1.19. (Appl. 2097/19.)

In gas furnaces of the type in which the flame

passes tangentially into the furnace, part of the air for combustion is admitted tangentially to the wall of the furnace at about the point where the flame would otherwise impinge, with the result that noise and local overheating are reduced.—B. M. V.

Dehydrating apparatus. C. Shorman, Chicago, Ill. U.S. Pat. 1,324,073, 9.12.19. Appl., 18.6.15.

THE material to be dried is supported on trays in a vertical cylindrical chamber, and air is supplied to a central hollow shaft provided with discharge pipes, in the form of arms which rotate in proximity to the material.—B. M. V.

Heating drum. H. C. Janes, Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,324,157, 9.12.19. Appl., 12.11.17.

A HORIZONTAL rotary cylinder is provided with an annular jacket, and is supported by coaxial horizontal hollow trunnions projecting outwards and carried by bearings. Fluid is supplied to the interior of the cylinder through one of the trunnions and exhausted through the other. The annular jacket is connected by radial pipes at each end with concentric hollow sleeves surrounding the trunnions and supported by independent bearings. Fluid is supplied to and exhausted from the jacket through the hollow sleeves.—W. F. F.

Reactions between gases; Apparatus for bringing about and controlling —. C. Conover, Philadelphia, Pa. U.S. Pat. 1,324,443, 9.12.19. Appl., 30.4.19.

THE gases pass from a supply chamber to a number of reaction chambers, being equally distributed among the latter by suitably restricted passages. The temperature of reaction is controlled by the circulation and boiling of a liquid in jackets surrounding the reaction chambers.—B. M. V.

Centrifugal extractor. F. M. Watkins, Cincinnati, Ohio. U.S. Pat. 1,325,064, 16.12.19. Appl., 9.8.16. Renewed 26.10.17.

THE side wall of the centrifugal basket is inclined upwards and outwards, so that material within the basket tends to be discharged outwards over the rim during rotation. Means are provided for preventing such discharge of solids while permitting discharge of liquid, such means having radial partitions projecting inwards into the basket and rotating with it. The solids may be discharged by an axial movement of the restraining means.—W. F. F.

Precipitating matter from gases; [Electrical] process and apparatus for —. W. W. Strong, Mechanicsburg, Pa. U.S. Pat. 1,325,124, 16.12.19. Appl., 23.10.18.

THE apparatus comprises an active electrode in contact with the gas and having a relatively great surface curvature, with a passive electrode out of contact with the gas and having a comparatively small surface curvature.—B. N.

Electrical treatment of gas; Apparatus for —. L. Bradley, East Orange, N.J., Assignor to Research Corporation, New York. U.S. Pat. 1,325,136, 16.12.19. Appl., 18.2.16.

AN apparatus for the electrical treatment of gases is provided with a number of flues of polygonal cross-section having walls forming an electrode, and with discharge electrodes extending longitudinally through the flues.—B. N.

Measuring quantities of different materials in varying proportions; Apparatus for —. T. Lea, Wolverhampton. Eng. Pat. 135,888, 16.4.19. (Appl. 16,854/18.)

Electro-osmotically removing water; Apparatus for —. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. Eng. Pat. 135,820, 25.6.18. (Appl. 10,459/18.) Int. Conv., 20.10.17.

SEE Ger. Pat. 311,663 of 1917; this J., 1919, 544 A.

Heat-changes; Method and apparatus for inducing —. R. Vuilleumier, New Rochelle, N.Y., U.S.A. Eng. Pat. 136,195, 4.6.18. (Appl. 9215/18.)

SEE U.S. Pat. 1,275,507 of 1918; this J., 1919, 210 A.

Gas producing [and steam generating] plant. Eng. Pat. 135,959. See IIa.

Electro-osmotic apparatus. Eng. Pats. 135,815 and 135,819. See XI.

Dehydrating. U.S. Pat. 1,324,072. See XIXa.

Mixing materials. U.S. Pat. 1,324,771. See XXII.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Extraction of — under pressure with solvents other than benzene. F. Fischer. Ges. Abhandl. zur Kennt. der Kohle, 1919, 3, 246—247. Chem. Zentr., 1919, 90, IV., 632.

THE oil soluble in light petroleum spirit which was previously obtained by extracting coal under pressure with benzene may also be obtained by extracting soft coal directly with petroleum spirit (b. pt. 60°—100° C.) under pressure at 250° C. The quantity of the yellowish-red, viscous product, which apparently contains paraffin scale, amounts to 1% of the coal. Extraction in a similar manner with ethyl acetate yields 2% of a yellowish-red, translucent oily resin. Alcohol extracts a thick golden-yellow oil, also some sodium chloride.—J. F. B.

Coal; Manufacture of a dense, semi-coke by the low-temperature carbonisation of —. F. Fischer. Ges. Abhandl. zur Kennt. der Kohle, 1918, 3, 102—104. Chem. Zentr., 1919, 90, IV., 633—634.

IN rotating apparatus the simple insertion of a sufficiently heavy roller when using powdered coal brings about such a considerable compacting of the semi-coke that it may be utilised as a smokeless fuel, and is suitable for transportation. The recovery of low temperature tar and gases is not interfered with by the roller. For example, from 15 kilos. of gas coal there were obtained 11.3 kilos. of compact semi-coke, apparent sp. gr. 0.68, and 1.5 kilos. of tar oils. The arrangement also permits the introduction of steam or the application of vacuum.—J. F. B.

Gas industry; Refractory problems of the —. W. H. Fulweiler and J. H. Taussig. J. Ind. Eng. Chem., 1919, 11, 1153—1157.

BOTH fireclay and silica products are used as refractory materials, and this gives rise to complex problems in the design and construction of the retort benches, on account of the difference in expansion. In the combustion chamber, retorts, and connecting blocks exposed to 1550° C., a high-grade silica material with over 96% of silica and about 1.5% of lime is used. In recuperators, with a temperature range of 850°—1100° C., fireclay material containing 55% of silica is used. At the top and bottom of the retorts fireclay is used on account of its small coefficient of expansion and resistance to spalling. The outer walls are made of a lower-grade firebrick. The whole bench should be insulated with kieselguhr. In water-gas apparatus the effect of high temperatures, abrasion, slagging, and spalling have.

to be considered. In the generator the lining is subjected to abrasion by the fuel particles whirled about by the blast, and by the pokers and other tools. In the combustion zone the lining is subjected to high temperature, slagging action from the fuel-ash, some abrasion by the fuel, and more or less serious abrasion by the clinker. In the lower portion of the generator the lining is subjected to the action of steam, hot gases, and the tools used for removing the clinker. In the connecting ducts there are high temperature, erosion by particles of fuel and slagging action of the ash. In the carburettor the chequer-bricks and crown are subjected to high temperature and sudden cooling, causing spalling. The usual lining for generators is fireclay brick; silica and magnesite bricks are not successful owing to the changes in temperature. Chromite, zirconia, and carborundum bricks seem good, but are about 30 times as costly as fireclay bricks; they reduce the adhesion of slag to the lining, and therefore the amount of cleaning required. For chequer-work flint-clay bricks are best. The cements used should be free from plastic clay or other material added to give smooth working; they should be of ground grog with a minimum quantity of rather more fusible material.—A. B. S.

Carbonising variations effected by temperature changes in the top of the coking chamber. A. H. Middleton. Northern Sect., Coke-Oven Managers' Assoc., Dec., 1919. Gas World, 1920, 72, Coking Sect., 13—17.

AN investigation into the cause of the presence of large quantities of paraffin hydrocarbons in the crude benzol from a set of coke-ovens at Langley Park is described. In the rectified products 8–9% of paraffins was found in the benzol, 2–4.5% in the "pure" toluene, and as much as 26% in the solvent naphtha. In the installation there is an exceptionally great distance between the top of the heating flues of the coke-ovens and the crown of the oven arch, and hence only comparatively low temperatures are obtained at the top of the retort when fully charged. The paraffin content of the crude benzol could be decreased by reducing the coal charge so as not to fill the top of the oven, or by fully charging with fine coal, all of the coal or its distillation products being then subjected to a high temperature. The same result could be obtained by fully charging with large coal, and working with a higher gas pressure in the flue and lengthening the flame. Charging the ovens with fine coal reduced the output of the ovens, but gave an improved coke. The high yield of paraffins corresponded with a decreased yield of benzol and naphthalene, but an increased yield of ammonia. A gas of excellent quality was also obtained.—W. P.

Ethylene present in coke-oven gas; Fixation of — as alcohol and its derivatives. E. Bury. Cleveland Inst. Eng. Gas J., 1919, 148, 718—721.

PEAT coke has been used successfully at the Skinninggrove Ironworks during the war for the separation of ethylene and other gases from the gaseous products of carbonisation processes (this J., 1919, 857 A). Though this method is advantageous in some cases, sulphuric acid (95%) is preferable as an absorbent when liquid derivatives of ethylene are to be prepared. The hot gas from the retorts is cooled and purified in the usual manner, and then passed through a drying scrubber in which 80% sulphuric acid is circulated. The gas is then heated to 60°–80° C., the hot retort gases being utilised for this purpose, and is then passed into the ethylene scrubbers, which contain 95% sulphuric acid also maintained at 60°–80° C. The acid flows in the opposite direction to the gas, the acidity being reduced by about 10% on leaving the scrubbers. The alcohol is distilled off by means of superheated steam, or acetic acid may be obtained by electro-

lysis or by the use of ozonised air. Any sulphur dioxide formed by the reduction of the sulphuric acid is recovered by interaction with hydrogen sulphide in the crude coke-oven gas. A plant dealing with 5800 tons of coal per week should yield 9280 galls. of absolute alcohol, taking a 50% conversion of ethylene to alcohol as actually obtained. The reduction in calorific value of the extracted gas is small.—W. P.

Carbon, hydrogen, and methane; Equilibrium between —. H. F. Coward and S. P. Wilson. Chem. Soc. Trans., 1919, 115, 1380—1387.

THE proportion of methane in equilibrium with carbon and hydrogen diminishes with increasing temperature, and hence when equilibrium is once established in the zone of reaction the cooling of the gaseous mixture on its removal from the carbon is not accompanied by an alteration in composition. The series of experiments described were carried out in the same manner as those of Bone and Coward (this J., 1908, 1143; 1910, 744) except that the gaseous products of reaction were passed backwards and forwards over the carbon used until analysis showed the attainment of constancy of composition. The percentage compositions of the equilibrium mixtures of methane and hydrogen in contact with amorphous carbon at atmospheric pressure were found to be: at 1100° C., CH₄ 0.6, H₂ 99.4%; at 1000° C., CH₄ 1.1, H₂ 98.9%; at 850° C., CH₄ 2.5, H₂ 97.5%. The attainment of equilibrium was found to be extremely slow with highly-purified charcoal (ash 0.06%) at temperatures below 1100° C.; a series of experiments was therefore carried out using a mixture of equal weights of the charcoal and nickel oxide. Experiments at 650° C. indicated that equilibrium could not be attained within any reasonable period of time. Carbon monoxide was found to be present in the equilibrium gas mixtures; its formation was due partly to a slight oxidising action by the glaze of the heated tube, but mainly to a side reaction between carbon and water vapour.—W. P.

Flame; Propagation of — in complex gaseous mixtures. Part I. Limit mixtures and the uniform movement of flame in such mixtures. W. Payman. Chem. Soc. Trans., 1919, 115, 1436—1445.

FROM a consideration of the heat balance existing during the propagation of flame in limit mixtures a "limits generalisation" is advanced, from which simple formulæ are deduced which enable the limits of inflammability of complex gaseous mixtures to be calculated from the known values for its simple constituents. The generalisation states that all mixtures, in any proportions, of limit mixtures of the same kind are themselves limit mixtures, the limiting percentage being that of the mixed inflammable gas. The expression deduced from this for air mixtures is the same as the empirical formula advanced by Le Chatelier, which has been experimentally verified (this J., 1919, 126 A). Where the combustible gas remains the same and the atmosphere of oxygen and nitrogen varies in composition, the percentage of combustible gas will be directly proportional to the oxygen present in the different limit mixtures. This is shown to be true over a wide range of upper and lower limits of inflammability of methane in different atmospheres, a slight deviation being noted in mixtures containing a large excess of nitrogen. The experiments described in this and the following papers were carried out in a glass tube 2.5 cm. in diameter. The limits of inflammability of methane in pure oxygen were found to be 5.7% and 59.2%. Carbon was deposited by the flames in all upper limit mixtures containing more than 38% CH₄. The speed of the uniform movement of flame was approximately

constant in all the limit mixtures, and was the same as the limiting speed in limit mixtures of air with the paraffin hydrocarbons, namely, 20 cm. per sec. in the tube used. The limits of inflammability of the paraffin hydrocarbons in air were: methane 5.8% and 13.3%, ethane 3.3% and 10.6%, propane 2.4% and 7.3%, butane 1.9% and 6.5%, and pentane 1.6% and 5.4%. Convection currents have a marked effect upon the limits, and must be taken into account in any attempt to calculate the limits of inflammability of a gas from its thermal constants.

—W. P.

Flame; Propagation of — in complex gaseous mixtures. Part II. The uniform movement of flame in mixtures of air with the paraffin hydrocarbons. W. Payman, Chem. Soc. Trans., 1919, 115, 1446—1453. (See preceding abstract.)

SINCE the speed of the uniform movement of flame is approximately the same in all limit mixtures in a given tube, then all mixtures of gas mixtures having this limiting speed will show the same speed of propagation of flame. This is shown to apply to other speeds besides those in limit mixtures. A "speed generalisation," namely, that all combinations of mixtures in which the speed of the uniform movement is the same will propagate flame at the same speed, is shown to hold accurately for different mixtures of air with methane and pentane respectively. The generalisation affords a means of calculating the speed of the uniform movement in any complex inflammable gas mixture, provided the maximum speed of flame is the same for each combustible gas. Where they are not the same the calculation is limited by the maximum speed of flame which is lowest. In this case the maximum-speed mixture (with air mixtures) can be calculated by the method given in an earlier paper (this J., 1919, 126 A), and its speed by means of the following expression:—

$$S = \frac{aS_s + bS_b + cS_c + \dots}{a + b + c + \dots}$$

where S is the speed required, a, b, c, \dots are the amounts present of each maximum-speed mixture with air as calculated, S_a, S_b, S_c, \dots are the speeds of flame in those mixtures respectively. The maximum speed of flame for mixtures of any complex combustible gas with air can also be found by a simple graphical method. The curves obtained by such calculations are shown to agree closely with those obtained from experimental determinations of the speeds in mixtures of pentane and methane and of pentane and hydrogen. The speeds of the uniform movement of flame in a 2.5 cm. tube were determined for each of the paraffin hydrocarbons in admixture with air. With the exception of methane, for which the maximum speed of flame was 67 cm. per sec., the maximum speeds of flame in air mixtures of these gases were approximately the same, namely, about 82 cm. per sec. In each instance the mixture having the maximum speed of flame contains more combustible gas than is required for complete combustion.—W. P.

Flame; Propagation of — in complex gaseous mixtures. Part III. The uniform movement of flame in mixtures of air with mixtures of methane, hydrogen, and carbon monoxide, and with industrial inflammable gases. W. Payman, Chem. Soc. Trans., 1919, 115, 1451—1462. (See preceding abstracts.)

THE application of the speed generalisation to the calculation of the speeds of the uniform movement of flame in industrial gas mixtures is complicated by the fact that with carbon monoxide, which is usually present in such mixtures, the speed of flame is dependent on the amount of water vapour present. Hydrogen, and any gas into the composition

of which hydrogen enters, acts towards mixtures of carbon monoxide and air in a manner comparable with that of water vapour. Such gases are generally present in industrial gas mixtures in sufficient quantity to render the presence of water vapour negligible. For the calculation of the speeds of flame in these mixtures the "effective speeds" of carbon monoxide-air mixtures can be calculated from the speeds of the flames in mixtures of carbon monoxide, hydrogen, and air, and although the effective maximum speed of carbon monoxide-air mixtures (125 cm. per sec.) was about double the speed found in one series of determinations (60 cm. per sec. at 12° C. and 750 mm., mixture saturated with water vapour), the values so obtained gave good results when used for the calculation of the speeds of complex mixtures. The methods of calculation are applied to simple mixtures of methane, hydrogen, and carbon monoxide taken two or three at a time in admixture with air. A straight coal gas and a producer gas were also examined, the maximum speeds of flame in their mixtures with air being 151 cm. per sec. (17.9% coal gas) and 72.2 cm. per sec. (39.0% producer gas) respectively. The speed of flame in mixtures of air with gas, such as producer gas, containing a large proportion of nitrogen can be calculated on the assumption that the cooling or retarding effect on the flame of excess of air or of nitrogen will be the same, since their specific heats are the same. This assumption is not quite correct, since the presence of reactive gas slightly opposes the retarding effect of air. The presence of a large proportion of nitrogen in the combustible gas has the effect of diminishing the "displacement" of the maximum-speed mixture from the mixture containing combining proportions of combustible gas and oxygen.—W. P.

Ether-alcohol-air and acetone-alcohol-air mixtures; Ignition of — in contact with heated surfaces. A. G. White and T. W. Price, Chem. Soc. Trans., 1919, 115, 1462—1505.

THE "sub-ignition temperatures" of various mixtures of air with ether, alcohol, and acetone have been determined, and also the limits of inflammability of these mixtures. The effect of adding small amounts of glyceryl trinitrate (nitroglycerin) and the peroxides of ether to these mixtures has also been examined, these being possibly present in the vapours from cordite stoves. The mixtures were allowed to flow rapidly into an evacuated tube heated to a known temperature in an electric furnace, the lowest temperature at which inflammation occurred being called the "sub-ignition temperature." It was found necessary to use a fairly long tube with the sealed end projecting well out of the furnace to prevent what the authors term "shock ignition," by means of which ether-air and carbon bisulphide-air mixtures could be ignited at ordinary temperatures. Shock ignition took place when the pressure was suddenly and rapidly equalised in the apparatus. The ignition of dilute ether-air mixtures usually resulted in the luminous, incomplete combustion of the ether (Perkin, Chem. Soc. Trans., 1882) with the formation of what has been termed a "cool flame." Tubes of different diameters were used with different leading-in tubes to bring about different periods of equalisation of pressure. The sub-ignition temperatures may be taken as: ether 187° C., alcohol and acetone about 500° C. The sub-ignition temperatures were lowered with decrease of pressure, and were lower if ethyl hydrogen peroxide were present. They were not affected by the presence of diethyl peroxide or glyceryl trinitrate. The limits of inflammability were determined in glass tubes 2.5 and 5 cm. in diam. and in iron tubes 5 and 15 cm. in diam. The extreme limits found were: ether 1.73% and 23.30%,

alcohol 4.16% and 18.95% (upper limit at 60° C.), and acetone 2.88% and 12.40%. The limits of inflammability of mixtures could be calculated by the Le Chatelier formula (this J., 1919, 126 A) for downward propagation of flame, but deviations were noted for upward and horizontal propagation. The flame travelled steadily only in the case of downward propagation, being often jerked out in the other cases, giving less trustworthy results. Increase of temperature and pressure both widened the limits. Mixtures were made up by a weighing method, but were not analysed before sparking.

—W. P.

Gas producers; Calorific value of the gas formed in the recovery of low temperature tar in —. F. Fischer. Ges. Abhandl. zur Kennt. der Kohle, 1918, 3, 105—108. Chem. Zentr., 1919, 90, IV., 632—633.

WHEN coal yielding 30% of volatile constituents was heated in a tubular retort at 500° C. before being discharged into the main shaft of the gas producer there were obtained, per kilo. of coal, 100 grms. of tar, 50 litres of distillation gases having a heating value of over 8000 cals. per cub. m., and 3.5 cub. m. of producer gas of 1200 cals. per cub. m. In working such a producer, however, in order to obtain as high a yield of tar as possible, a considerable quantity of the producer gas must be drawn through the retort, and the yield will then amount to about 500 litres of distillation gases of 1800 cals. per cub. m., together with 3050 litres of producer gas of 1200 cals. per cub. m. It is far more difficult to wash out all the tar oils from such a diluted distillation gas, and a cooling down of the producer gas proper on addition of the washed distillation gas cannot be avoided. The recovery of the light constituents of the low temperature tar is therefore very difficult in the ordinary Mond producer plant.

—J. F. B.

Purifier installations; Some mechanical aspects of [gas] —. F. R. Parsons. Gas World, 1920, 72, 25—26.

THE present-day tendency in gas purifier construction is towards the provision of a larger superficial area of purifying surface per thousand cubic feet of gas treated. The older type of construction was characterised by the requisite strength and rigidity being accompanied by a sufficiency of elasticity. The modern type of purifier-box is characterised by complete absence of flexibility. The prevailing practice of revivification *in situ* is accompanied by the production of high temperatures within the purifying material. Serious damage may result therefrom owing to expansion of the purifying material. This in general expands in lateral directions only, and causes bulging of the sides or division plate of the purifier. To avoid this it is suggested that one or more V-shaped furrows should be formed in the layer of oxide, to reach across the full width or length of the box. The oxide is furrowed on all four sides of the box, and such furrows afford space for expansion of the oxide.

—J. S. G. T.

[Spent] oxide; Revivification of — in situ. O. B. Evans. American Gas Assoc. Gas J., 1919, 148, 726.

In common gas-works practice 0.75—2% of air is mixed with coal gas, containing 100 grains of hydrogen sulphide per 100 cub. ft., at the inlet of the purifiers for the purpose of continuous revivification of the oxide. In the intermittent method the fouled box is put out of service and a large volume of air blown through the oxide mass. This method has been abandoned by some works on account of the tendency to cause fire, but its use is becoming more general, and it is in common

practice on the Pacific coast. The method is most satisfactory, with ample purification capacity and frequent revivification. To decrease the danger of firing, shallow beds are advised. The admixture of inert gas is helpful and more useful than steam. Cooling the gas is also advised, with the removal of as much as possible of the tar fog. Local heating may be prevented by drawing air through the oxide under slight suction instead of blowing air through. Using this method of working the process has been in use in California for 28 years without a fire or a case of overheating.—W. P.

Petroleum spirit, ether, etc.; Elimination of the danger of ignition of electrically-charged —. D. Holde. Ber. deut. Pharm. Ges., 1919, 29, 569—574.

IN further experiments with Dolezalek's apparatus (this J., 1914, 1147) it was found that when the Leyden jar, containing the electroscope and connected to the vessel into which the petroleum spirit etc. had been forced under pressure, was electrically charged by means of a rubbed ebonite rod the charge was retained for a considerable time even after the vessel containing the petroleum spirit had been earthed. Hence it would seem that in the experiments previously described (this J., 1915, 649) the electroscope attachment immersed in the petroleum spirit had not conveyed any appreciable charge during the introduction of the liquid into the vessel. It was also found that when the insulating property of the petroleum spirit was particularly good the negative charge taken up by the connection between the electroscope and the petroleum spirit vessel could remain there when the vessel was earthed, the petroleum spirit thus playing the part of the glass in the Leyden jar. The conclusions formerly drawn with regard to the dissipation of electrical charges were therefore substantially confirmed. Other experiments indicated that the electrical charges taken up by the petroleum spirit vessel were essentially due to the friction of the liquid upon the walls of the tubes which conveyed it. No electrical charges were taken up when the petroleum spirit was introduced under a pressure of 5—6 atm., with the atmospheric moisture at 75 to 95%, whilst at 57 down to 30% of moisture charges were invariably received. Variations in the atmospheric temperature (13° to 31° C.) had no appreciable influence upon the strength of the charge received or the insulating capacity of the petroleum spirit. Not only the petroleum spirit tank, but also all metal parts in contact with it ought to be earthed as a safeguard against the danger of sparks. In the case of technically pure ether and benzene the conditions in the electrically-charged condition differ somewhat from those observed in the case of petroleum spirit, for these substances can give up high charges to the walls of the vessels and tubes merely in consequence of their own electrical conductivity ($\epsilon = 10^{-9}$ to 10^{-12}).—C. A. M.

Mineral oils; Freezing point of —. E. Lantos. Chem.-Zeit., 1919, 43, 853.

MINERAL oils containing paraffin wax in solution do not answer to the ordinary cryoscopic rules. In the oil industry the real freezing point of oils is not determined, but the so-called "setting point," i.e., the temperature at which an oil placed in a tube, which is chilled in a freezing mixture of snow and sodium hydroxide solution and then turned mouth downwards, does not alter its meniscus for 30 secs. As a rule the smaller the viscosity of an oil the lower is its setting point. On refining two Rumanian oils, however, it was found that although the viscosity was reduced the setting point was raised, e.g., from -20° C. to -15.5° C. Oils of Galician origin (e.g., those from Limanowa) which

contain paraffin wax show high setting points. For example, a refined oil, rich in paraffin wax, with viscosity 2.29, showed a setting point of $+10^{\circ}\text{C}$. On extraction of the wax the viscosity was reduced to 1.85, whilst the setting point was then -20°C . These results point to the conclusion that solutions in which the solvent and dissolved substance are homologous do not obey the cryoscopic laws.—C. A. M.

Fullers' earth [for treatment of mineral oils]; Preparation of —. W. C. Phalen. Chem. and Met. Eng., 1919, 21, 469.

The bleaching action of fullers' earth on mineral oils is independent of its chemical composition, and depends on its power of selective absorption. The following factors are important: The longer the column of earth the better the bleaching effect; the coarser the earth the more rapid the flow and the poorer the bleaching effect; the higher the temperature the faster the flow and the poorer the bleaching effect. Filtration should, therefore, take place at the lowest temperature compatible with the fluidity of the oil. The first burning of the earth should take place at about 600° – 700°F . (315° – 370°C .); this merely removes water and organic matter. There should be no fusion. After use the earth may be revived by further burning at 1050° – 1100°F . (565° – 590°C .); with Florida earth the optimum temperature is 1100° – 1200°F . (590° – 650°C .). Very serious loss in efficiency results from over-burning.—A. E. D.

Oil and gas storage tanks. Gardner. See I.

Carbon monoxide. Lamb and Larson. See XIXb.

Gas analysis. Jones and Neumeister. See XXIII.

Interferometer. Edwards. See XXIII.

Viscometer. Baume and Vigneron. See XXIII.

PATENTS.

Fuel; Manufacture of a solid —. E. K. Koblischek, Vienna. Ger. Pat. 313,469, 30.6.18.

RESIDUES from petroleum refineries consisting of silicates containing paraffin wax are melted and filtered; mineral wax or beeswax is added, and the product is mixed with powdered wood charcoal, together with an oxygen carrier, such as manganese dioxide, barium peroxide, or potassium chlorate. The mass is continuously stirred, poured into moulds at about 50°C ., and allowed to cool. The fuel is easily ignited and burns slowly.

—J. F. B.

Coal; Apparatus for feeding and burning —. E. C. R. Marks, London. From Combustion Economy Corporation, Chicago, Ill., U.S.A. Eng. Pat. 136,121, 21.7.19. (Appl. 18,128/19.)

POWDERED coal is fed by a horizontal screw conveyor into a vertical conduit, through which a stream of air passes upwards. At the point of delivery into the air conduit the coal is fed between two ribbed plates, one of which is stationary and surrounds the delivery orifice, and the other is attached to the rotating conveyor spindle at an adjustable distance from the first plate. The coal is thereby disintegrated and its delivery rendered more uniform. The velocity of the mixed fuel and air remains high until it reaches the burner, the cross-section of the conduit being less than the opening of the burner. A flared connection is interposed between the conduit and the burner. The powdered coal conveyor and the blower for producing the air blast are both driven by the same motor, the former through a variable speed gear, so that the proportion of coal to air may be varied. The volume of air may be varied independently by

a valve in the delivery conduit from the blower, and an indicator may be combined with the valve, graduated in accordance with pre-determined requirements of air blast for different rates of fuel feed. The conduit adjacent to the burner is provided with two annular sets of stationary inclined deflecting blades to ensure complete mixing. The burner opening may be varied by a pair of hinged vanes, one on either side of the opening. The vanes are mounted on hollow trunnions, and water is circulated through them to prevent injury by overheating.—W. F. F.

Fuel; Combustion of —. C. Dellwik, Chorley Ltd., London. Eng. Pat. 136,212, 9.11.18. (Appl. 18,392/18.)

Wood, Herts, and Techno-Chemical Laboratories, POWDERED fuel is introduced by means of a jet of a small quantity of cold air or steam, gas, or vapour into a further volume of air which has been preheated by the waste heat of combustion to a point above the ignition temperature of the fuel. The quantity of air is regulated so that the fuel is burned mainly to carbon monoxide to produce a reducing atmosphere. The use of preheated air ensures that a much larger quantity of heat is available from a given weight of fuel, for maintaining a reducing atmosphere at a given temperature. The hot gases are afterwards passed through regenerators alternately with the air for combustion, so that the latter is heated only by the sensible heat of the combustion products without further combustion of the carbon monoxide.—W. F. F.

Pitch; Combustion of —. Rowntree and Co., Ltd., and F. G. Fryer, York. Eng. Pat. 135,886, 28.9.18. (Appl. 15,809/18.)

PITCH is powdered, mixed with air in regulated amount, and burnt. Since pitch usually contains only about 0.5% of ash, the flame may be used for heating, by direct contact, materials in which it is desirable to avoid contamination by ash.—W. F. F.

Vertical gas retort settings. F. Cummins, Manchester. Eng. Pat. 136,398, 28.2.19. (Appl. 5002/19.)

In order to dispense with the vertical waste gas flues at the back of retorts of the Glover-West type, holes are provided in the floors of all the combustion chambers and circulating chambers at the rear ends of the chambers. The waste gases from each combustion chamber travel upward through the rear ends of the combustion chambers and into the circulating chambers, gas outlets from here to the chimney being controlled by dampers. The number of dampers required is much less than in the usual type of setting.—W. P.

Gas producing [and steam generating] plant. W. Climie, Ayr, and W. Lees, Glasgow. Eng. Pat. 135,959, 9.12.18. (Appl. 20,392/18.)

In a complete gas producing and steam generating plant the gas producer is contained within a steam generator designed and of sufficient strength to generate steam at a pressure capable of performing all the work desired. The generator supplies steam to a steam engine, which is used for operating the fuel elevator, the air blow, the fuel rake, and the purifier pump. The steam pipe from the generator is provided with a by-pass and a surplus valve, the steam generated in excess of that taken up by the engine being led away and utilised as desired.

—W. P.

Gas; Production of [mixed] —. S. N. Wellington, London. Eng. Pat. 136,186, 30.7.17. (Appl. 10,972/17.)

A MIXED gas for industrial purposes consists of an admixture of a rich gas obtained by the destructive

distillation of coal at a comparatively low temperature (400° – 620° C.), with producer gas having an approximate calorific value of 125–150 B.Th.U. per cub. ft. The gases are mixed in the proportions necessary to give a power gas of a calorific value of 300 B.Th.U. per cub. ft.—W. P.

Gasous fuels; Apparatus for use in mixing — S. N. Wellington, London. Eng. Pat. 136,200, 30.7.17. (Appl. 13,737/18.)

In order to maintain the calorific value of a mixed gas (see preceding abstract) approximately constant, use is made of a calorimeter, which automatically varies the proportion of one of the gases admitted to the mixing chamber. This calorimeter consists of two tanks containing liquid, within which floats are immersed suspended from opposite ends of a beam. Beneath one tank a supply of the mixed gas is drawn off and burned. The expansion of the liquid raises the surface of the liquid and the float in this tank. The beam is provided with a contact arm operating between two contacts corresponding, for example, to the values 310 B.Th.U. and 290 B.Th.U. These electrically control a water cock, which admits or releases water from the water-loaded governor controlling the admission of the low temperature gas into the mixing chamber.

—W. P.

Producer gas plant. A. H. Lynn, London. Eng. Pat. 136,189, 2.3.18. (Appl. 3715/18.)

A CYLINDRICAL vessel is divided by a partition into two superposed compartments, each provided with gas inlet and outlet, and serves as a gas cooler and an air-heater and saturator. Liquid for cooling the gas is circulated through the upper compartment and passes by gravity through the lower compartment for preheating and saturating the air with water vapour. (See also Eng. Pats. 11,452 of 1912 and 744 of 1913; this J., 1912, 1167; 1913, 1147.)—W. P.

Gas producers. H. W. Bamber and E. Goldsmid-Abrahams, London. Eng. Pat. 136,333, 13.1.19. (Appl. 901/19.)

THE velocity of the exhaust gases from an internal combustion engine is used to induce a current of air through the fire of a gas producer when starting up. The combustion engine is operated temporarily with petrol, paraffin, or coal gas as fuel.—W. P.

Purifying gas and obtaining valuable by-products therefrom; Methods of and apparatus for — F. A. Umsted, Chicago, U.S.A. Eng. Pat. 135,931, 3.12.18. (Appl. 20,002/18.)

DURING purification the gas passes through a series of contiguous chambers arranged to form one complete apparatus, and is subjected to two distinct series of processes. The first series consists in fractionally cooling the gases in order to remove the heavy constituents in successive stages, during which the gases do not come into contact with the cooling liquid. The desired temperatures are maintained automatically. In the second series purification is effected by direct contact with suitable reagents and by filtration. The method of purification is designed primarily to deal with the gases obtained by the dry distillation of waste material, such as municipal waste, sewage, street sweepings, peat, sawdust, etc.—W. P.

Hydrocarbons; Continuous and uniform treatment of — with sulphurous acid. Allgem. Ges. für Chem. Industrie, Berlin. Eng. Pat. 114,845, 9.4.18. (Appl. 6067/18.) Int. Conv., 21.3.17.

UNIFORMITY in the supply and discharge of the liquid sulphur dioxide is achieved by the provision of intermediate vessels, which are placed between the main and auxiliary evaporators and between the

latter and the discharge pumps. Pressure equalisers and supply pipes connect the main evaporators to the intermediate vessels, and gauge glasses indicate the progress of the operations, which must be so controlled that no more sulphur dioxide flows into the evaporators from the mixing chamber than is removed therefrom.—A. E. D.

Petrol; Production of — F. Tinker, Birmingham. Eng. Pat. 135,015, 19.12.18. (Appl. 21,289/18.)

CRUDE oil is distilled and the vapour produced is passed into a superheater or cracking chamber, on leaving which it is brought into contact with the crude oil entering the still. Heat interchange is thereby effected, the vapour passing into a dephlegmator, and the "topped" crude oil being directed into the still.—A. E. D.

Low-boiling oil; Manufacture of — from higher boiling petroleum or related oil. E. B. Cobb, Jersey City, Assignor to Standard Oil Co., Bayonne, N.J. U.S. Pats. (A) 1,322,878 and (B) 1,322,762, 25.11.19. Appl., (A) 25.4.14, and (B) 4.10.19.

(A) THE oil is distilled with anhydrous aluminium chloride, which is dissolved in part of the oil under treatment. The process is so conducted that only the minor part of the carbon content of the oil remains as coke. (B) The distillation as above is continued until the residue remains only just sufficiently fluid to be withdrawn as a liquid. This residue is washed with water and there is obtained oil together with coke and an aqueous solution of aluminium chloride.—A. E. D.

Oil from oil-bearing shale; Apparatus for distilling oil from — H. R. Straight, Adel, Iowa. U.S. Pat. 1,323,204, 25.11.19. Appl., 31.7.18.

A SERIES of cars which carry the shale travels through a brick chamber so arranged that the lower half is a combustion space, whilst the upper half is a vapour space. Sealing devices separate the successive cars, which themselves divide the chamber horizontally into the vapour and combustion sections.—A. E. D.

Gasoline from hydrocarbon gases; Process of obtaining — H. C. Cooper, Clarksburg, W. Va., Assignor to Hope Natural Gas Co., Pittsburgh, Pa. U.S. Pat. 1,323,837, 2.12.19. Appl., 18.1.17.

THE gas is first subjected to a spray and subsequently to liquid gasoline-absorbing menstruum. The gasoline is finally separated from the solvent. —A. E. D.

Gasoline; Process of manufacturing — A. D. Smith, Vinita, Okla., Assignor to J. T. Milliken, St. Louis, Mo. U.S. Pat. 1,324,075, 9.12.19. Appl., 16.10.15.

IN the process for manufacturing gasoline from oils of the paraffin series described in U.S. Pat. 1,239,423 (this J., 1917, 1125) the pressure in the still, i.e., 125–150 lb. per sq. in., is maintained on the vapours during condensation and on the condensed liquid in the receiving tank. The condensed liquid is drawn off and immediately mixed with low gravity naphtha, without reducing the pressure, to prevent loss of the more volatile hydrocarbons.

—L. A. C.

Petroleum and other oils; Process for obtaining distilled products from — Apparatus for obtaining distilled products, particularly gasoline, from petroleum and other oils. A. A. Stapp, Denver, Colo. U.S. Pats. 1,324,212 and 1,324,213, 9.12.19. Appl., 4.12.17.

IN the continuous production of low-boiling hydrocarbons, such as kerosene, benzine, and gasoline, from petroleum oils, the oil is first heated to a tem-

perature sufficient only to remove the gasoline fraction; the residual oil passes into a vaporiser and thence into a cracking tube. The vapours from the cracking tube are separated by fractional condensation into gasoline and high-boiling hydrocarbons, and the latter fraction is returned to the vaporiser. The uncondensed gas produced both during the initial heating of the oil and in the cracking treatment is superheated and mixed with the vapours undergoing the cracking treatment.

—L. A. C.

Liquid fuel. A. A. Backhaus, Baltimore, Md., Assignor to U.S. Industrial Alcohol Co. U.S. Pat. 1,324,765, 9.12.19. Appl., 28.11.17.

A PETROLEUM distillate and alcohol are blended by means of carbon bisulphide.—W. J. W.

Hydrocarbons; Conversion of —. R. Fleming, Swampscott, Mass., Assignor to The Richard Fleming Co. U.S. Pat. 1,321,766, 9.12.19. Appl., 17.11.16.

A COLUMN of heavy liquid hydrocarbon is heated to its boiling point under a pressure of 75 lb. and above, the vapour of a lower boiling hydrocarbon being at the same time passed through it during a prolonged period. The resulting vapours are thus heated above the cracking point under a pressure sufficient to retard formation of unsaturated hydrocarbons and gases.—W. J. W.

Hydrocarbons; Process and apparatus for treating —. R. R. Rosenbaum, Chicago, Ill. U.S. Pat. 1,324,983, 16.12.19. Appl., 3.10.17.

CRUDE hydrocarbons are heated in a still, and the vapours are separated by fractional condensation into two portions. The lighter fraction is collected, and the heavier fraction is superheated "under sufficient pressure to store kinetic heat energy therein," and is returned to the bottom of the still, where it vaporises. The "liberated kinetic heat energy" breaks up the vapours and any oil in the still, liberating further quantities of the lighter fraction.—L. A. C.

Mineral oil of high boiling point; Process of converting — into products having lower boiling points. R. Koetschau, Waldheim, Germany, Assignor to The Chemical Foundation, Inc. U.S. Pat. 1,325,299, 16.12.19. Appl., 19.3.14.

In the treatment of mineral oils of high boiling point with a catalyst to obtain products of lower boiling point the oils are subjected during the time of contact with the catalyst to the action of a material capable of increasing the speed of the catalytic reaction. For example, the oils are treated with the hydrocarbon addition products of an aluminium halide.—L. A. C.

Gaseous fuel burners. A. C. Ionides, jun., London. Eng. Pat. 136,296, 19.12.18. (Appl. 21,314/18.)

Fuel; Liquid —. E. C. R. Marks, London. From U.S. Industrial Alcohol Co., New York. Eng. Pat. 136,452, 5.5.19. (Appl. 11,177/19.)

SEE U.S. Pat. 1,306,244 of 1919; this J., 1919, 565 A.

Gas furnaces. Eng. Pat. 136,356. See I.

Ammonia. U.S. Pat. 1,321,979. See VII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Charcoals; Some chemical characters of ancient —. T. C. Cantrill. *Archæol. Cambrensis*, 1919, 365—392.

THE charcoals (i.e., charred and blackened wood)

found in the course of archaeological excavations are much more readily attacked by nitric acid and potassium chlorate and by hypochlorite solution than are charcoals produced under modern conditions (true charcoals); the ancient charcoals differ amongst themselves as regards their solubility, bleaching, and disintegration when treated with the reagents mentioned, but this may be due to the varying species of wood from which the charcoal was derived. The author adduces evidence, chemical and physical, to show that there is no reason to doubt that the ancient charcoals were produced by the action of heat or fire, and that the blackening of the wood was not caused by the action of peaty soils.—W. P. S.

Decolorising carbon; Preparation of — by prolonged heating. C. E. Coates. *Lat. Bull.*, 161, 8.

IT is stated that ordinary wood charcoal from sawdust (the decolorising power of which is very slight) may be converted into a carbon equal in efficiency to "Norit" by prolonged heating at 1200° C. in a closed crucible (cf. Ger. Pat., 250,741; this J., 1912, 1022).—J. P. O.

PATENTS.

Incandescent gas mantles; Material for strengthening and increasing the luminosity of —. H. Pope, Tibshelf, Derby. Eng. Pat. 135,797, 25.6.19. (Appl. 15,939/19.)

YELLOW resin, 1 part, washing soda, 4 parts, and chalk, 10 parts, are ground to a powder, and the mixture used alone or formed into a liquid with methylated spirit, for treating incandescence gas mantles.—B. N.

Filaments for electric lamps. Westinghouse Lamp Co., Bloomfield, N.J., Assignees of R. D. Hall, East Orange, N.J., U.S.A. Eng. Pat. 121,596, 27.11.18. (Appl. 19,595/18.) Int. Conv., 19.12.17.

THE refractory metal of the filament, e.g., tungsten, is mixed, during the process of manufacture, with 2—7% by volume (2.5—10% by weight) of silica, and either 1—5% by volume (2—10% by weight) of alumina or 0.1—0.4% by volume (0.05—0.2% by weight) of thorium.—B. N.

Distilling shale. Eng. Pat. 136,076. See X.

III.—TAR AND TAR PRODUCTS.

Benzene; Effect of pressure and of dissolved air and water on the melting point of —. T. W. Richards, E. K. Carver, and W. C. Schumb. J. Amer. Chem. Soc., 1919, 41, 2019—2028.

BENZENE saturated with air under atmospheric pressure melts 0.003° below the true triple point, and the freezing point is not much changed by pressure. An increase in pressure on benzene free from air changes the freezing point 0.029° per atmosphere. Saturation with water lowers the freezing point of benzene 0.095°. The true freezing point of benzene saturated with air is 5.493° C., and the true triple point 5.496° C.—J. F. S.

Toluene; Criteria of the degree of purity of commercial —. J. S. Lumsden. *Chem. Soc. Trans.*, 1919, 115, 1366—1372.

COMMERCIAL toluene contains varying amounts of a liquid which cannot be nitrated. This liquid was not isolated, but for the tests described the assumption was made that it may be represented by a paraffin mixture which boils close to the boiling point of toluene. By fractionation of petrol a quantity of such a liquid was obtained boiling at 108°—112° C. Pure toluene at 15° C. was found to

have sp. gr. 0.8712, the paraffin mixture sp. gr. 0.743. There is no change of volume on mixing toluene and paraffin, and the determination of the density of commercial toluene gives a fair indication of the percentage of impurity present, 1% of paraffin producing a lowering of sp. gr. by 0.0013. The estimation of toluene by the temperature of miscibility with acetic acid as described by Orton and Jones (this J., 1919, 811 A) is too complicated for commercial use. A modified method is described, which is quickly carried out, and although only comparative is trustworthy. Nitration and estimation of the excess nitric acid by the nitrometer is believed to be the most accurate test, and the weighing of the solid after nitration gives the actual practical yield of nitro-compound. Nitration was effected by means of potassium nitrate and sulphuric acid, the reaction stopping quantitatively when all the toluene had been converted into the dinitro-compound. By none of the methods, under ordinary conditions of working, can an accuracy closer than 0.5% be expected.—W. P.

Phthalic anhydride. H. D. Gibbs. J. Ind. Eng. Chem., 1911, 41, 1031—1032.

In experiments on the oxidation of the methyl group of toluene, mixtures of oxygen and toluene and of atmospheric air and toluene were subjected to contact with various catalysts at temperatures from the boiling-point of toluene to about 550° C. The oxides of the metals of the fifth and sixth groups of the periodic system were the most effective catalysts, vanadium being the best and molybdenum the next best. Similar reactions were applied to the manufacture of phthalic anhydride from naphthalene (Gibbs and Conover, Eng. Pats. 119,517 and 119,518; this J., 1918, 684 A), and the conditions to obtain the optimum yield have been determined. The best laboratory results showed 82% of the theoretical conversion. The phthalic anhydride produced is remarkably pure and is quite free from chlorine or sulphur compounds which were common impurities in the phthalic anhydride formerly on the market. (See also this J., 1920, 57 A.)—C. A. M.

Emulsification by adsorption at an oil [nitrobenzene] water interface. S. E. Sheppard. J. Phys. Chem., 1919, 23, 634—639.

Emulsions of nitrobenzene in sulphuric acid and hydrochloric acid may be readily prepared by making the acid of the same density as the nitrobenzene. Similar moderately stable emulsions may be prepared by using as aqueous medium sulphuric acid saturated with lead sulphate or calcium sulphate, or hydrochloric acid saturated with lead chloride or sodium chloride, in both cases the acid solution being of the same density as the nitrobenzene.—J. F. S.

Carbonising variations. Middleton. See IIa.

Ignition of electrically charged petroleum spirit etc. Holde. See IIa.

Nitrotoluenes. (1) Bell and Sawyer. (2) Bell and Cummings. See XXII.

Nitro-compounds. Van Duin. See XXIII.

PATENTS.

Fractionation of crude benzol and the like; Process and apparatus for the continuous distillation and —. E. C. R. Marks, London. From Soc. Franco-Belge de Fours à Coke, Brussels. Eng. Pat. 136,450, 1.5.19. (Appl. 10,864/19.)

A SINGLE distilling column receives crude benzol at the top and steam at the bottom; mixtures of water and benzol vapours are taken off at various levels

from top to bottom and immediately condensed. After separating the water the benzol fractions are of commercial quality, and uniformity of products with varying crude benzol can be maintained by adjustment of valves in the pipes leading from the still to the condensers.—B. M. V.

Ortho- and para-toluene sulphonic chlorides; Process for separating —. F. E. Matthews and E. H. Strange, London. Eng. Pat. 135,987, 20.12.18. (Appl. 21,402/18.)

TOLUENE-*o*-SULPHONIC CHLORIDE is separated from mixtures of the ortho and para isomerides, containing 60–70% of the former (such as are obtained by cooling the sulphonation mixture to –20° C. and separating the para compound which crystallises out) by seeding the liquid mixture at a temperature a few degrees below 0° C., e.g., –6° C., with a crystal of the ortho-sulphonic chloride, and removing the crystals which separate out by centrifuging or other suitable means.—G. F. M.

Quinone and quinol; Method and apparatus for the production of —. F. T. Kitchen, West New Brighton, N.Y., Assignor to K. and T. Chemical Corporation, New York. U.S. Pat. 1,322,580, 25.11.19. Appl., 3.3.16. Renewed 3.4.19.

BENZENE is introduced into the lower part of an electrolytic tank containing an electrolyte of greater specific gravity, so that it rises through the liquid and is at the same time oxidised at the anode to quinone, which dissolves in the excess of benzene employed. The solution thus obtained is continually subjected to the electrolytic oxidation until the concentration of quinone becomes sufficiently high. The apparatus also comprises a reducing tank into the lower part of which the quinone solution is introduced, and means are provided for increasing the intimacy of contact between this and the reducing agent.—G. F. M.

Toluene; Manufacture of —. B. T. Brooks, Pittsburgh, Pa., Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,324,143, 9.12.19. Appl., 10.6.15.

TOLUENE is prepared by reacting on a high-boiling aromatic hydrocarbon with a catalytic material, distilling off the toluene, and condensing the vapours formed to obtain benzene, which is added to another charge of high-boiling aromatic hydrocarbon, the above operation being then repeated.—G. F. M.

Aromatic bodies; Oxidation of —. C. E. Andrews, Assignor to The Selden Co., Pittsburgh, Pa. U.S. Pat. 1,324,715, 9.12.19. Appl., 25.5.18.

AROMATIC hydrocarbons are oxidised by heating with sulphuric acid and a catalyst containing a compound of vanadium or molybdenum.—G. F. M.

Anthraquinone; Purification of crude —. C. E. Andrews, Assignor to The Selden Co., Pittsburgh, Pa. U.S. Pat. 1,324,716, 9.12.19. Appl., 1.7.18.

ANTHRACENE is separated from anthraquinone by vapourising into a current of gas at about 300° C. a part at least of the mixture and separately crystallising at different locations the volatilised substances.—G. F. M.

Anthracene press-cake; Treatment of —. C. E. Andrews, Assignor to The Selden Co., Pittsburgh, Pa. U.S. Pat. 1,324,717, 9.12.19. Appl., 1.7.18.

AN improved recovery of methylanthracene, phenanthrene, and anthracene is obtained by heating the press-cake to about 200° C. in a current of gas, and gradually cooling the vapours to produce crystallisation at different locations.—G. F. M.

Combustion of pitch. Eng. Pat. 135,886. See IIa.

IV.—COLOURING MATTERS AND DYES.

Dyes; Explosibility and inflammability of —. B. Humiston, W. S. Calcott, and E. C. Lathrop. *J. Ind. Eng. Chem.*, 1919, 11, 1075—1076.

ANY risk in the drying of dyestuffs is due to the increased temperature, since little dust is produced in the process. A rise in temperature may cause oxidation leading to spontaneous ignition, as in the case of Sulphur Black, or there may be decomposition, with the evolution of gases, as in the case of Victoria Green. In the decomposition of the latter dyestuff an exothermic reaction sets in, and the gases might become explosive in a closed space. The apparatus used for studying these properties was a large test-tube, enclosed in an air-bath. A small amount of the dyestuff was heated in the test-tube, through which a slow current of air passed, while a thermo-couple was immersed in the dyestuff, and a note was taken of the temperature at which the latter ignited. A similar apparatus was used for determining the danger of decomposition of the dyestuff, but no air was passed through the tube, and the gases given off were conducted to a gas burette. The point of initial decomposition was noted and whether the decomposition was endothermic or exothermic. The temperature at which the drying plant should be worked should, as a rule, be 25°—30° C. below that of the exothermic decomposition or ignition of the particular dyestuff. In grinding machines of the ball mill type temperatures up to 75° C. may be experienced, and over 100° C. in the impact crushing type of mill. Using apparatus of the type employed by the U.S. Bureau of Mines for determining the explosibility of mixtures of coal dust and air, the explosion points of mixtures of dye dusts with varying quantities of air ranged from 400° to 1100° C., duplicate determinations agreeing within about 50° C. The results agreed fairly with experience in the works as to the relative safety of the different dyes examined. More accurate results were obtained by the use of an apparatus consisting essentially of a large test-tube, fixed in an air bath, and having a tube for the introduction of air to the bottom of the test-tube. The latter was gradually heated while a current of air sufficiently rapid to keep the tube constantly filled with dust was admitted, and the temperature of decomposition was noted. The temperatures obtained with the same series of dyestuffs as tested in the Bureau of Mines apparatus ranged from 250° to 550° C., and duplicate determinations agreed within 10°—15° C. In the standardisation of dyestuffs an additional factor is introduced in the possibility of a reaction between the standardising reagent and the dyestuff. For example, the accidental addition of soda ash to Victoria Green would probably cause a reaction with the oxalic acid in the dyestuff, which might raise the temperature to the decomposition point of Victoria Green.—C. A. M.

Dyes; Problems in the identification of —. E. F. Hitch and I. E. Knapp. *J. Ind. Eng. Chem.*, 1919, 11, 1076—1078.

THE composition of azo-dyes may often be determined by reduction and isolation and identification of the products of the reduction, but amino-derivatives of second components are not easily separated or identified. For the separation and identification of components in a mixture, microscopical examination, fractional dyeing, solution tests with alcohol and water, and extraction with immiscible solvents may be employed. In some cases mixtures may be separated by adsorption on kaolin, talc, etc., followed by washing with water or alcohol. For the separation and identification of mixtures on the fibre methods of fractional reduction, extraction with various solvents, and spectro-

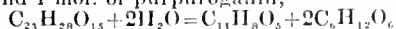
scopic examination may be used. The quantitative results obtained by means of the spectrophotometer would be sufficient to identify a dyestuff, but for this purpose it would be necessary to obtain the data of the absorption bands of a large number of pure dyestuffs by means of a calibrated instrument.—C. A. M.

Dyestuffs; Estimation of the strength of —. W. H. Watkins. *J. Ind. Eng. Chem.*, 1919, 11, 1079—1080.

THE principal causes of disagreement between seller and buyer in the case of dyestuffs are differences of standard and variations in samples due to incomplete mixing of the dyestuff with the inert material. The preparation of the solution used in dyeing tests may also affect the apparent strength. As a rule a little dilute acetic acid should be used with basic dyes. The influence of hard water upon a dyestuff has led to disputes between buyer and seller, especially when the dyestuff is partly in the form of its calcium salt, and a little soda has been used to correct this and to neutralise the effect of hard water. In such cases if the solution is not heated at a sufficiently high concentration complete conversion of the calcium salt into the sodium salt may not be effected, so that the former will not contribute to the dyeing process. Tests of the accuracy of judgment of an experienced dyer have shown that he will usually not be able to detect variations of less than 3%.—C. A. M.

Red pea gall; Colouring matter of the —. M. Niedenstein. *Chem. Soc. Trans.*, 1919, 115, 1328—1332.

THE red colouring matter of the red pea gall was isolated by extracting the de-fatted and powdered gall with boiling alcohol and precipitating the tannin from the aqueous solution of the extract by the addition of caseinogen. The purified and recrystallised substance, to which the name dryophantin is given, formed deep-red glistening needles, melting at 219°—220° C., and having the empirical formula $C_{23}H_{28}O_{15}$. Its alcoholic solution gives a red precipitate with ferric chloride, whilst ammonia and alkali hydroxides produce a colour change to deep blue, which once more becomes red on acidification. On hydrolysis with sulphuric acid dryophantin yields 2 mols. of dextrose and 1 mol. of purpurogallin,



The substance is therefore not an anthocyanin, and as the other so-called anthocyanins of plant galls are probably of a similar nature, it is proposed to classify these red pigments in a new group of organic colouring matters, to which the name gallorubrones is assigned.—G. F. M.

Electrochemical production of organic compounds. Thatcher. See XI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Vegetable fibres; Economy of cellulose and caustic soda in the isolation, purification, and bawking of —. M. Freiburger. *Deut. Baumwollind.*, 1919, 1, 38, 55. *Chem.-Zeit.*, 1919, 43, Rep., 292.

IN the treatment of cotton with caustic soda by various methods the loss of weight owing to the solubility of the cellulose in the caustic lye or to the formation of oxycellulose, if the cotton is not properly de-aerated, may be extremely large, especially in the case of strong caustic liquors. A regenerated lye, re-causticised, and decolorised by oxidising agents, still contains sufficient reducing substances to protect the cellulose against a large loss of weight, but not such as to stain it during

bowking, and such a lye gives better results than either fresh caustic liquor or old spent liquors. The regeneration of spent liquors also effects an economy in caustic soda, as these liquors can be used several times in succession.—J. F. B.

Soda pulp [washing]; Report of committee on —. M. L. Griffin. Paper, 1919, 25, 132—138.

It is important in washing the pulp to conserve the original concentration of the black liquor with as little dilution as possible. The ideal way would be to squeeze out the original liquor thoroughly and then wash out the remainder by counter current in a closed system. This is not realised in any known system, but the author suggests the possibility of using a cage through which the wet material is forced by a screw propeller, squeezing out the fluid contents and delivering the mass relatively dry. If such machines were arranged in multiple series, the material being mixed with dilute liquor or water between successive squeezing units, the process could be made continuous. In the ordinary system the choice lies between a large number of shallow washing tanks in which washing can be quickly performed but with considerable dilution of the liquor, and a smaller number of deep tanks in which the time required to wash the pulp is unduly prolonged. The best method is to work between these two extremes, using a closed system of small connected tanks operated in a cycle, each tank being cut out, discharged, recharged, and connected up again in turn. When using open pans it is convenient to have them of such dimensions that each pan will hold the contents of one digester. Rectangular pans are most generally used; for a charge of about 5200 lb. of soda pulp, a wash-pan of 18×16×5 ft. is suitable. The pans are provided with perforated plates leaving a space of 6 ins. above the bottom from which a 4- or 6-in. pipe leads to the distributing main, with gate valves communicating with the strong liquor tank, the weak liquor tank, or the drain. Each pan has a 10-in. wash-out main at each end, through which the drained pulp is washed to the screens. In the separate washing system each pan drains independently of the others; in some mills there are two strong liquor tanks and one weak. The liquor in the first strong liquor tank is used for rinsing out the next digester, whilst that collected in the second is sent to the recovery plant. The original liquor has a density of 12° B. (sp. gr. 1.086) at 15° C., and the washings are collected in the strong liquor tank for recovery until the density falls to 5° B. (sp. gr. 1.034), after which they are collected in the weak liquor tank until they test 1° B. (sp. gr. 1.007). The average density of strong liquor sent to the evaporators is 9° B. (sp. gr. 1.063). In working the cycle system the average density of liquor to the evaporators is 10.25° B. (sp. gr. 1.072), and this indicates a considerable economy of coal, but the time required for washing is considerably longer. If the pulp is allowed to remain in contact with the black liquor its colour suffers permanently. A great advantage is gained by washing the stock with hot water.—J. F. B.

Sulphite-cellulose waste liquors. W. Philippsthal. Umschau, 1919, 23, 718—719. Chem.-Zeit., 1919, 43, Rep., 292.

SULPHITE pulp waste liquor is said to contain substances which act as a protection against rodents, and it may be used as a dressing for textiles. In the manufacture of artificial stones from marble cement small additions of sulphite lye to the water increase the absorption by 30%, and the finished stone possesses greater strength. A paste of china clay which has been rendered more fluid by addition of sodium carbonate solution becomes stiffer again on addition of traces of sulphite liquor. The liquor

may also be used as a catalyst in the manufacture of rubber. Clayey soil is used for minor building purposes, but does not bind well with lime mortars. If milk of lime be mixed with sulphite liquor and the loam or clay stirred in, then slabs made of the resulting material can be built up like ordinary bricks with lime or cement mortars. A little sulphite liquor should be mixed with the mortar to obtain better binding. Existing structures may simply be coated with a mixture of milk of lime and sulphite liquor.—J. F. B.

Starch in paper; Estimation of —. II. Determination of dextrins in the presence of beater starch. O. Kamm and F. H. Tendick. Paper, 1919, 25, 460—461.

THE starchy matter present in paper may be partly in the form of normal starch added to the pulp in the beating engine, and partly in the form of modified starch or dextrin applied to the surface of the paper in the process of tub sizing. The modified starch may be extracted from the paper by digesting 5 grms. of the sample with 200 c.c. of water at 60° C. for 45 minutes. The extract is filtered off under suction, and the soluble carbohydrate is hydrolysed by acid and the sugar estimated by Fehling's solution. The normal starch remaining in the paper may then be extracted by means of dilute acetic acid according to the method previously described (this J., 1919, 757 A). The separation of the two types of starch is sufficiently accurate for practical purposes. A small quantity of normal starch, amounting to 0.2—0.3% on the paper, is extracted along with the modified starch by the water at 60° C.—J. F. B.

PATENTS.

Cotton bolls; Process for maturing —. F. D. Maisch. Philadelphia, Pa. M. F. Maisch, executrix. U.S. Pat. 1,317,983, 7.10.19. Appl., 19.5.15.

IMMATURE or frost-bitten cotton bolls are subjected to a heated atmosphere above 100° F. (38° C.), whereby the shell of the cotton boll is contracted and caused to open and liberate the cotton, which is then expanded by evaporating the moisture under the continued application of the heat.—J. F. B.

Hood; Textile fibre from —. K. Mutter, Triptis. Ger. Pat. 305,141, 11.11.17.

THE fibres are isolated only from the summer wood portions of the annual rings, which contain the strongest fibres. The annual rings are peeled off from the woody stem and are freed from the soft, fibreless layers of the spring growth. The coherent layer of summer growth is treated chemically for the removal of the whole or a part of the lignin and resins, and the resulting cellulose fibres are isolated by a mechanical treatment.—J. F. B.

Basket or binding material [from hop stems]; Manufacture of a —. Chem. Bleicherei E. Jetter, Ortrand. Ger. Pat. 306,307, 14.11.17.

THE woody residues of hop stems from which the fibrous bast has been removed are boiled for about 6 hours under high pressure with caustic soda solution of 3°—4° B. (sp. gr. 1.020—1.027). The hop stems become sufficiently soft and pliable for basket-making after a single treatment, and if treated twice or more the material becomes completely pliable and can be used for binding.—J. F. B.

Lupin stems; Manufacture of textile fibres from —. Hoffmanns Stärkefabriken A.-G., Salzdahl. Ger. Pats. (A) 306,362, 28.10.17, and (B) 306,496, 24.5.17.

(A) LUPIN stems are boiled with water and then submitted to a fermentation process. The time required for retting in the ordinary way is 14—21

days, but if the stems be boiled for 15–30 minutes and then retted, the fibre is ready for stripping after 4–8 days' retting. (a) The raw material is boiled with dilute aqueous salt solutions, then washed, dried, and worked up in the usual way. Salts of the alkali metals with inorganic or organic acids may be used or cheap waste salt solutions from the Stassfurt district.—J. F. B.

Straw, grasses, etc.; Process for obtaining textile fibres from —. C. Wolf, Schweinsburg, and E. Trommer, Crimmitschau. Ger. Pat. 307,688, 9.4.18.

ONLY the upper parts of the stems containing the finer fibres are employed for the manufacture of textile fibres by digestion with alkalis. These upper portions may be again subdivided in order to obtain materials for fine and coarse spinning. The lower portions of the stems are used for litter or other agricultural purposes.—J. F. B.

Textile yarns; Manufacture of durable —. M. Müller, Limbach, and S. Herzberg, Charlottenburg. Ger. Pat. 313,520, 15.1.18.

STRING, rope, etc., or the constituents used in preparing them, particularly materials composed of paper yarn, are coated with metals by the spraying process. The metal-coated textiles are distinguished by a high tensile strength and resistance to external influences.—J. F. B.

Viscose threads; Machine for spinning, washing, and drying —. M. Denis, Mons, Belgium. Eng. Pat. 125,394, 8.4.19. (Appl. 8878/19.) Int. Conv., 8.8.13.

THE viscose is fed through one of a pair of interchangeable filter-boxes into a bent glass tube which delivers it to the squirting nozzle directed upwards. The coagulating liquid is contained in a vessel shaped like a test-tube which can be adjusted vertically in any desired position to afford a suitable immersion of the nozzle in the bath. The coagulating liquid is continuously circulated through inlet and overflow pipes with flexible connections. The thread passes upwards out of the bath and is caught by hooks attached to an endless travelling metallic web, preferably of aluminium, on which it travels downwards in an inclined path. During its passage in contact with the web the thread is sprayed by various suitable liquids, e.g., a fixing and a washing liquid, which are collected in troughs arranged behind the wire-cloth support. At the bottom the thread is detached from the web by a jet of liquid directed through the latter in the reverse direction, and is passed through a drying chamber while supported by a similar web travelling upwards through a current of hot air.—J. F. B.

Cellulose acetate; Flexible product having a base of —. J. Dupont, Assignor to Soc. Anon. des Etablissements J. Dupont, Argenteuil, France. U.S. Pat. 1,317,276, 30.9.19. Appl., 18.1.18.

A PLASTIC composition is made with cellulose acetate to which a mixture of *o*- and *p*-hydroxybenzyl alcohols has been added.—J. F. B.

Cellulose formations; Process for producing —. P. Minck, Hermsdorf, Germany. U.S. Pat. 1,317,306, 30.9.19. Appl., 2.9.14.

A CUPRAMMONIUM solution of cellulose is projected into a precipitating bath containing copper carbonate and an alkali hydroxide.—J. F. B.

Paper or paper board. E. C. R. Marks, London. From Cedaroid Co., Inc., Rochester, N.Y., U.S.A. Eng. Pat. 133,732, 11.9.18. (Appl. 14,784/18.)

PAPER or paper board intended for containers or receptacles, or for keeping away insects or germs,

is made by the incorporation with the paper pulp of an odorous substance or essential oil, mixed with a suitable substance to promote emulsification. A mixture suitable for tobacco paper is composed of: water, 25; cedar oil, 25; sugar, 15; liquorice, 10; vinegar, 10; alcohol, 10; and glycerine, 5. This mixture is slowly added, in the proportion of 10–20 to the pulp in the beater.—J. F. B.

Paper-size and process of making same. J. A. De Cew, Montreal, Canada. U.S. Pat. 1,317,616, 30.9.19. Appl., 31.5.17.

ROSIN soap containing over 25 of free rosin is diluted and then discharged into a cold solution of sodium silicate until the total dilution corresponds approximately to 2 of total solids.

Paper product and process of making same. J. A. De Cew, Montreal, Canada. U.S. Pat. 1,317,618, 30.9.18. Appl., 15.1.18.

WATERPROOF paper is made by incorporating with the paper stock, as a sizing agent, a precipitated mixture of wax, rosin, and alumina, then drying the product, and heating to the fusion temperature of the sizing mixture.—J. F. B.

Paper-size; Process of treating —. J. A. De Cew, Montreal, Canada. U.S. Pat. 1,317,619, 30.9.19. Appl., 15.4.18.

A SUFFICIENT quantity of alkali is added to the paper stock in the beaters or mixers so that the concentration of the basic ions will prevent the formation of alkaline-earth resins by the salts existing in solution; a resin size is then added and precipitated by aluminium sulphate.—J. F. B.

Paper for spinning, paper yarns, and fabrics; Process for softening —. C. Hasenbring, Hamburg. Ger. Pat. 300,695, 6.1.17.

THE goods are treated with a relatively strong solution of calcium chloride and then rinsed in such a manner that the calcium chloride is not completely removed. Alternatively the goods may be impregnated with a smaller quantity of a dilute solution of calcium chloride and then stoved without washing. The treated material has a soft and slightly soapy feel, and the tensile strength is scarcely affected.—J. F. B.

Paper yarn fabrics; Process for compacting —. F. Rawitscher, Berlin. Ger. Pat. 301,361, 3.5.17.

THE finished fabric is subjected to a prolonged boiling treatment in order to convert its constituent yarns into a permanently swollen condition. If the boiling be continued for a sufficient length of time, the swollen condition of the individual threads becomes fixed without destroying their twist. Even if the strength of the single threads is diminished by this treatment, that of the complete fabric is rather increased thereby, owing to the fact that the yarns of the fabric are considerably compacted. If it be desired to compact the fabric without making it more absorbent, the treated fabric may be impregnated with some water-repellent substance.—J. F. B.

Paper textiles and articles made therefrom; Waterproof impregnation of —. M. Linke, Remscheid. Ger. Pat. 305,024, 26.6.17.

PAPER fabrics or the stitched articles made up from them are steeped in water until they show no further shrinkage, and are then treated, slightly damp or after drying, with brushes, being subsequently painted in the same condition on one or both sides with a water-repellent impregnating material which also makes them pliable. The impregnating material may be composed of chalk, lithopone, a binder consisting of an oil lacquer containing fatty matter and a thinner such as oil

of turpentine or a substitute. The brushing treatment must always be performed in the same direction, in order to make the surface of the paper fabric uniform and flat and to smooth down the fibres which have been raised by the steeping, also to allow of a uniform distribution of the impregnating material.—J. F. B.

Paper fabrics; Process for impregnating —. Gebr. Heyl und Co. A.-G., Charlottenburg. Ger. Pat. 307,771, 14.8.17.

The goods are impregnated with mixtures containing wood tar or wood tar oils and cellulose esters, particularly in combination with amyl acetate. The impregnated fabric may be used as a material for water buckets, tent covers, ground cloths, wagon sheets, etc.—J. F. B.

[*Paper-making machines;*] *Steam inlet and water outlet connections for drying cylinders* [of —]. J. O. Lundberg, Borge, Norway, and H. S. Wahlberg, Sandarne, Sweden. Eng. Pat. 136,485, 25.6.18. (Appl. 15,999/19.)

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleaching of cotton fabrics containing coloured threads. R. Aue. Sealed note No. 2117, Aug. 28, 1911. Bull. Soc. Ind. Mulhouse, 1919, 85, 44—45. Report by T. Stricker. *Ibid.*, 45—46.

THE author attributes the bleeding and loss of colour of indigo-dyed threads to the action of the dressings in the material, which exert a reducing action during the alkali boil, and proposes the addition of an oxidising agent to the liquor. Satisfactory results are obtained by the use of sodium peroxide. This method was used in a works at Tagiewa, Baku, in 1909, the kier liquor containing 1½ kilos. of sodium peroxide in 2000 litres of water; boiling was effected at a pressure of 0·3 atm. Further addition of caustic soda is unnecessary; the colour remains unchanged and the whites are well bleached. Stricker points out that the addition of an oxidising agent is not entirely novel, as several methods have been proposed (see Fr. Pat. 392,858 of 1908 and Ger. Pats. 218,254 of 1908 and 250,397 of 1910; this J., 1909, 89; 1910, 419; 1912, 1122). It is pointed out that the addition of sodium peroxide to the lye with ordinary apparatus leads to a serious loss of oxygen.—L. L. L.

Aniline Black; New process for production of — on wool. M. Prud'homme. Sealed note No. 1436, Dec. 30, 1903. Bull. Soc. Ind. Mulhouse, 1919, 85, 39—41. Report by M. Battagay. *Ibid.*, 43—44.

THE author describes an improvement on a process described previously (this J., 1913, 908). The fibre is mordanted and oxidised by means of an acid bath containing 10—20 grms. of chromic acid per litre. The material is steeped for 5—10 minutes, wrung lightly, washed, and then hung up to dry. The dyeing is carried out by working the oxidised material for one hour, or sufficiently long to obtain even impregnation, in a cold bath of aniline and an acid, e.g., hydrochloric acid or an organic acid, preferably formic acid; the material is squeezed and then allowed to lie in heaps. The oxidation is complete in about 1—1½ hours. The material is then simply well washed and dried, further treatment being unnecessary. The use of organic acids prevents too rapid oxidation of the aniline in the bath, and the oxidation is partly obviated by use of a concentrated bath, 100 grms. of aniline per litre. The method is applicable to other bases, e.g., *o*- and *p*-toluidine, benzidine, etc., giving blacks or

browns. It is also applicable to other colouring matters susceptible to oxidation; with these a slight steaming after exposure may be necessary.

—L. L. L.

Aniline Black; Dyeing of — on wool and on mixed wool and cotton, or wool and silk fabrics. M. Prud'homme. Sealed note No. 1454, Mar. 10, 1904. Bull. Soc. Ind. Mulhouse, 1919, 85, 42—43. Report by M. Battagay. *Ibid.*, 43—44.

AN improvement on the process described in the preceding abstract consists in the addition of a ferric salt to the mordanting bath, with or without an acid. The bath contains 1·5 grms. CrO₃, 20 grms. Fe₂Cl₆ of 45° B. (sp. gr. 1·453), 100 grms. of water. The material is steeped for 10 mins. in the cold bath, squeezed, exposed to the air for 10 mins. and washed. It is then dyed by immersing in a cold bath containing 100 grms. of aniline, 80 grms. of hydrochloric acid of 21° B. (sp. gr. 1·17), 100 grms. of formic acid (60%), and 1800 grms. of water, after which it is exposed to the air for 1—1½ hours. Goods containing cotton are preferably mordanted in a bath at 45°—50° C. A considerable improvement is effected by the addition of 0·05—0·1 gm. of potassium permanganate to the iron bichromate bath. For wool alone a passage through a weak formic or oxalic acid bath at 50° C., and with half-wool or "gloria" an acid bath of bichromate, suffices to complete the fixation of the black.

—L. L. L.

Indigo; Direct printing of — on unprepared fabric. L. Caberti, C. Barzaghi, and P. Roggieri. Sealed note No. 1408, July 28, 1903. Bull. Soc. Ind. Mulhouse, 1919, 85, 46—47. Report by T. Baumann. *Ibid.*, 47—48.

THE authors print the material with a paste from which hydrosulphite is formed in the fibre, and thus reduces the indigo. The paste contains gum arabic solution (1/1), 200 grms.; indigo 20% M.L.B., 150 grms.; anhydrous sodium sulphite, 180 grms.; ammonium oxalate, 180 grms.; zinc powder, 60 grms.; soda ash, 50 grms.; borax, 40 grms.; glycerin, 140 grms. After printing the material is dried, steamed for 80—90 secs. at 101° C., and passed through a bichromate bath. Full shades are obtained. The paste appears to give better results after it has been kept for some time. Baumann states that the results are good, and that the colouring matter is entirely reduced to a yellowish green, the colour remaining without alteration for eight days. By double decomposition normal ammonium sulphite is formed in the paste, and this is reduced by the zinc to normal ammonium hydrosulphite.—L. L. L.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Contact sulphuric acid manufacture; Thermal problem in —. F. C. Zeisberg. Trans. Amer. Electrochem. Soc., 1919, 195—202. [Advance proof.]

THE contact process for manufacture of sulphuric acid is outlined with special reference to the control of temperature so as to obtain maximum conversion of the sulphur dioxide. The temperature may be kept below the maximum limit by supplying the reacting gases to the catalyst so slowly that the heat is dissipated by radiation; by diluting the reaction gases with an inert gas; by returning a portion of the converted gases to the entering gas stream and so lowering the concentration; by carrying out the conversion in stages with intermediate cooling of the gases; or by cooling the catalyst and converter by means of pipes and cooling jacket. A résumé is given of many processes embodying the

above principles, all of which, except the re-circulation of a portion of the converted gases, have been used in the past, and a list of U.S. patents is appended.—W. J. W.

Ammonia; Determination of —. Relative accuracy of colorimetric and titrimetric methods. E. R. Allen and B. S. Davisson. *J. Biol. Chem.*, 1919, 40, 183—197.

The volumetric methods are on the whole considered to be the more reliable for general use, although when great care is taken with the experimental technique it is possible that the Nessler process gives results of slightly greater accuracy. Methyl red is undoubtedly the best indicator for the titration of ammonia. (See further, *J. Chem. Soc.*, Feb., 1920.)—J. C. D.

Absorbent for ammonia respirators. G. St. J. Perrott, M. Yablick, and A. C. Fieldner. *J. Ind. Eng. Chem.*, 1919, 11, 1013—1016.

PUMICE-STONE impregnated with copper sulphate is shown to have a large capacity for absorbing ammonia. A canister containing 45 lb. in. of this material will protect a man for at least 5 hrs. in air containing 2% of ammonia, or for 2.5 hrs. in air containing 5% of ammonia. Tests at rapid breathing rates showed that the canister affords ample protection even during severe exertion. Cobalt chloride, ferrous sulphate, silicic acid, and boric acid are also good absorbents of ammonia.

—W. P. S.

Ammonium nitrate; Properties of —. Part I. Freezing point and transition temperatures. R. G. Early and T. M. Lowry. *Chem. Soc. Trans.*, 1919, 115, 1387—1401.

The freezing point of ammonium nitrate is very sensitive to the influence of slight traces of moisture. By careful purification and drying it has been raised to 169.6° C. The two higher transition points were determined from the arrests of temperature on heating and cooling the salt suspended in a liquid of similar density. The mean values found were 125.2° C. and 84.2° C. respectively. The lower transition point was determined dilatometrically and found to be 32.1° C. The arrest points due to the change of state at this temperature do not coincide with the transition point, but are determined by the rate of loss or gain of heat, as the velocity of change of state is much less than at the higher transition points.

—G. F. M.

Nitrogen; Fixation of — in the form of hydrogen cyanide by means of an electric arc. E. Briner and A. Baerfuss. *Helv. Chim. Acta*, 1919, 2, 663—666.

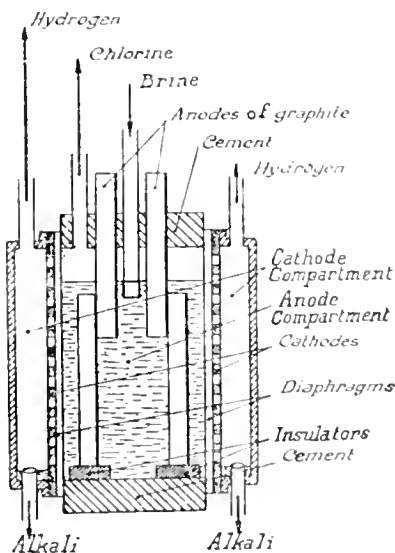
MIXTURES of hydrocarbons and nitrogen, when submitted to the action of an electric arc, produce hydrocyanic acid in yields much superior to the yields of ammonia from nitrogen and hydrogen. The mixture of gases is circulated at a speed of 8—10 litres per hour in a chamber where an arc of 0.020 amp. is burning between platinum electrodes 7 mm. apart. Using a mixture of 12 parts of methane, 23 of nitrogen, and 65 of hydrogen at a pressure of 635 mm., 0.8% by volume of hydrogen cyanide is obtained. This gives a yield of 0.46 grm. HCN per kilowatt-hour in addition to a yield of 0.14 grm. of ammonia per kilowatt-hour. In this case the P.D. applied was 1020 volts. With the same mixture, but with a voltage of 420, 0.12% by volume HCN was obtained, which is 1.75 grm. HCN per kilowatt-hour and 0.44 grm. NH₃ per kilowatt-hour. The best results were obtained with a mixture of 1 part of methane and 5 parts of nitrogen at 505 volts, when a yield of 0.75% by volume of HCN

was obtained, which is 7.39 grms. HCN per kilowatt-hour and 0.48 grm. NH₃ per kilowatt-hour.

—J. F. S.

Chlorine and alkali; Manufacture of — in the United States by modern electrolytic processes. E. Chaleyser. *Rev. Prod. Chim.*, 1919, 22, 613—620.

The various types of electrolytic cells employed for the manufacture of chlorine and alkali are divided into the following classes:—Cells with a permeable diaphragm between the anode and cathode; cells with an intermediate mercury electrode; cells in which stratified layers are formed in the electrolyte; cells employing molten sodium chloride as electrolyte with a lead cathode. The cells with diaphragms are the only ones used to a large extent commercially in the United States, and these may be divided into three groups:—(a) Cells with diaphragms immersed in the electrolyte, with separate arrangements for feeding and circulating the anodic and cathodic electrolytes; (b) cells in which the diaphragm is in contact on the anode side only with the electrolyte, the latter passing from the anode to the cathode compartment; (c) cells with diaphragms immersed in the electrolyte, with circulation of the latter from one compartment to another. In cells containing immersed diaphragms hypochlorites and chlorates are formed, giving rise to oxygen at the anode, with resulting deterioration of anodes and contamination of chlorine by carbon dioxide, together with a low electrolytic yield. The cells of group (b), which include the Townsend, Wheeler, Allen-Moore, and Nelson types, do not suffer from this defect, and are described in detail. The Billiter-Siemens type belongs to group (c),



but is little used in the United States. The Allen-Moore cell is shown in transverse section in the diagram, the dimensions of each cell being: length, 3.4 m.; breadth, 0.55 m.; height, 1.04 m. The electrolyte is freed from calcium, magnesium, sulphate, and suspended organic matter; it contains 310—315 grms. of salt per litre, and is slightly acid (0.01% HCl). The products of electrolysis, chlorine, caustic soda, and hydrogen, are very pure, the purity of the chlorine being 98.6%, and the caustic soda liquor contains 8—10% NaOH and 12% NaCl. For the production of 5 tons of chlorine and 5.6 tons of caustic soda per day of 24 hours, based on a

guaranteed efficiency of 92%, the following figures are given:—

No. of cells.	Ampères.	Volts.	Consumption in K.W.H.	
			Per ton chlorine.	Per ton caustic soda.
150	1200	3.8	3129	2772
130	1400	4.05	3335	2954
120	1500	4.2	3458	3063

The principal advantages claimed for this type of cell are: the cells are constructed of a cheap cement, which is non-conductive and therefore prevents losses of current; dismantling for cleansing and repairing is easily effected, thus avoiding losses of time; a high concentration is obtained in the caustic liquor, reducing expense in evaporation; the life of the anodes is greater than in the cases when hypochlorous acid is formed; practically no chlorate is formed.—B. N.

Alkali chlorides; Comparative electrolysis of —. E. Briner, A. Tykociner, and B. Allmöff. *Helv. Chim. Acta*, 1919, 2, 666—672.

SOLUTIONS of the chlorides of lithium, sodium, and potassium of concentration 3.1N when electrolysed by a current of 50,000 coulombs showed a total current yield of 75%, 82.5%, and 87% respectively. (See also *J. Chem. Soc.*, Feb., 1920).—J. F. S.

Halogens; Indirect electrolytic estimation of —. J. H. Reedy. *J. Amer. Chem. Soc.*, 1919, 41, 1898—1902.

HALOGENS may be estimated electrolytically by the following method. The solution of a halogen salt is electrolysed between a silver gauze anode and a bright platinum cathode with a current of potential 0.59 volt. This ensures the deposition of the whole of the halogen on the anode as silver halide, but towards the end of the process some silver goes into solution. To make sure all the halogen is deposited the current is passed for about 15 mins. at 0.59 volt; at the beginning the current is large, but it falls to a negligible value after this time; the voltage is then raised to 0.60 volt and the current passed for a further 5 mins. The anode is washed, dried, and weighed, and then made the cathode in the electrolysis of a dilute solution of sodium hydroxide until there is a free evolution of hydrogen. This reduces the silver halide to black silver, which is not very adherent and must be handled carefully. The electrode is washed, dried, and heated in an electric furnace until it becomes white and the whole is sintered together. It is then weighed, and the loss in weight gives the weight of the halogen. The method gives results comparable in accuracy with those obtained by the usual gravimetric method.—J. F. S.

Perchlorate; Electrolytic formation of — from chlorate. J. G. Williams. *Faraday Soc.*, Dec., 1919. [Advance copy.]

THE formation of perchlorate by electrolysis is considerably improved if a higher temperature (up to 60° C.) is maintained in the electrolyte, and the frequent addition of acid still further improves the efficiency. During the electrolysis chloride is produced, and there appears to be an equilibrium value for each temperature, varying from 2.3 grms. per litre at 31° C. to 4.7 grms. per litre of sodium chloride at 59° C.—B. N.

Potassium permanganate; Manufacture of — from ferro-manganese by electrolysis. M. de K. Thompson. *Chem. and Met. Eng.*, 1919, 21, 680—681.

THE commercial manufacture of potassium permanganate from ferro-manganese was investigated. A preliminary small-scale experiment was carried

out with ferro-manganese of the following composition:—Mn, 75.1%; Fe, 16.7%; C, 6.1%; Si, 0.9%; P, 0.2%; Cu, Ni, Ca, Mg, As, traces. Using potassium hydroxide solution (24 grms. per 100 c.c.) with an anode current density of 180 amp. per sq. ft. at 45°—50° C., electrolysis proceeded for a few hours with a current efficiency of 15%, but the anode became coated with oxides. This trouble was obviated by substituting an electrolyte of 20% potassium carbonate solution. A large-scale experiment which was carried out showed the necessity of keeping the temperature of the electrolyte down to 40° C. to prevent coating of the anode. Sheet-iron cathodes and asbestos cloth diaphragms were used. Current density was 65 amp. per sq. ft. at 4.2 volts, with total current of 350 amp., and current efficiency was about 17%. The electrolytic concentration was 200 grms. per litre. The process was continued for five days, but arrested each night. The liquid and anode mud were then removed and heated to 90° C., and the solution filtered and crystallised, the crystals being finally centrifuged. About 40 lb. of 98% potassium permanganate was made. The estimated cost was \$0.38 per lb. The process is stated to be commercially feasible.—W. J. W.

Potash; Wood ashes and production of —. E. Bateman. *Chem. and Met. Eng.*, 1919, 21, 615—619.

THE ash content of American species of wood varies from 0.05 to 3.02% in hardwoods, and from 0.02 to 0.82% in softwoods, with an average of 0.61% and 0.30% respectively, but the content of the same species may vary within wide limits. Most of the ash from softwoods is lost in burning, being carried up the chimney. The potash content of pure wood ashes is considerably higher than that of commercial ashes which contain sand, sawdust, charcoal, and other impurities. A large number of analyses of commercial ashes gave an average of 3.6% K₂O. The potash is recovered from the ashes by leaching. The author describes the plant and its operation and gives details of the manufacturing costs. During normal times the manufacture of potash from wood ashes would not be remunerative except under specially favourable local conditions.

—S. S. A.

Silicate of soda; Properties of commercial —. J. G. Vail. *J. Ind. Eng. Chem.*, 1919, 11, 1029—1031.

ALL commercial preparations of sodium silicate contain more silica than corresponds to the formula Na₂SiO₃, one grade containing four times that amount. The ratio between sodium oxide and silica varies between 1:4 and 2:3. Products more alkaline than the latter are not made owing to their tendency to crystallise, whilst the limit in the other direction is fixed by the low solubility of preparations containing more silica. The green or yellow colour of the commercial products is due to small amounts of ferrous or ferric iron, the latter being most frequent in the alkaline types. The solution with 1:4 ratio may be concentrated to about 37° B. (sp. gr. 1.305; about 34% of total solids) and then has the consistence of a jelly; if cooled to 5° C. it may be moulded into balls. A silicate of 2:3 ratio may be concentrated to about 69° B. (sp. gr. 1.916; about 62.5% total solids), and is then a tough mass which may be drawn into threads; it absorbs moisture from the air. The possible concentration increases with the rise in the alkali content. The ordinary 40° silicates freeze at about -3° C., becoming white and opaque, whilst solutions of above 60° B. (sp. gr. 1.71) do not lose their transparency on freezing, but become harder and finally brittle. The precipitates formed in sodium silicate solutions by most salts of heavy metals are said to contain free silicic

acid. Precipitation is also effected by various dehydrating agents, such as alcohol, glycerin, salt brine, and strong ammonia solution. The precipitates may be re-dissolved, but the resulting solutions differ from the original solution, notably, in their viscosity. Solutions rich in silica increase slowly in viscosity until the condition of jelly is reached, when there is a very pronounced increase. This is the case whether the rise in viscosity is due to decrease in alkalinity, decrease in temperature, or increase in concentration. Advantage is taken of this property in preparing rapid-setting adhesives from sodium silicate. The change from a liquid to a solid condition may take place with the loss of 1% of moisture, which may be rapidly absorbed by a layer of paper-board. Solutions suitable for this purpose when dried in the air do not contain less than about 20% of moisture. The resulting solid solution is more soluble than anhydrous silicate of the same relative composition. If suddenly exposed to a temperature above 100° C. such a solution expands into a mass of permanent bubbles, and has an apparent sp. gr. as low as 0.01. In this condition it is an excellent thermal insulator. Gels of widely different physical characters are made by treating silicate solutions with varying concentrations of acid. Strongly acid gels are used to prevent the splashing of acid from storage batteries, whilst very hard neutral gels may be used in the preparation of material for the adsorption of gases. The tensile strength of sodium silicate mixtures for acid-proof cements is easily brought up to 1700 lb. per sq. in. for air-dried briquettes, whilst a baked mixture of sodium silicate and clay, as used in the manufacture of abrasive wheels, has a tensile strength above 2000 lb. per sq. in.—C. A. M.

Ammonium chloride; Transition of dry —. A. Smith, H. Eastlack, and G. Scatchard. J. Amer. Chem. Soc., 1919, 41, 1961—1969.

AMMONIUM chloride dried over phosphorus pentoxide in a vacuum for three years at ordinary temperatures or for 45 days at 155°—165° C. undergoes the same transition to a second crystalline form at 184.5° C. (See also J. Chem. Soc., Feb. 1920.)—J. F. S.

Precipitates; New physico-chemical method of analysis of —. Application to the study of the calcium phosphates. P. Jolibois. Comptes rend., 1919, 169, 1161—1163.

Using the rapid method for mixing solutions previously described (this J., 1920, 81a), the author has studied the products obtained by mixing solutions of calcium hydroxide and phosphoric acid in varying proportions. In each case when equilibrium was reached the precipitate was collected and analysed and the supernatant liquid was analysed. Dicalcium phosphate can only be formed in a solution containing at least 0.80 gm. P_2O_5 per litre in the form of monocalcium phosphate, and similarly tricalcium phosphate is only stable in a solution containing less than 0.1 gm. P_2O_5 per litre. A new phosphate, $Ca_2P_2O_7 \cdot Ca_3(PO_4)_2 \cdot 10H_2O$, was obtained in a crystalline form.—W. G.

Stannous chloride; Decomposition of — by water and potassium hydroxide solutions. C. M. Carson. J. Amer. Chem. Soc., 1919, 41, 1969—1977.

THE compound, $2SnCl_2 \cdot 7Sn(OH)_2$, is the most basic of all the basic stannous chlorides. The slightly variable crystalline material formed by the action of boiling potassium hydroxide on stannous chloride has the composition, $3SnCl_2 \cdot 5SnO \cdot 3H_2O$. (See also J. Chem. Soc., Feb., 1920.)—J. F. S.

Chromic sulphate; A new complex form of —. A. Recoura. Comptes rend., 1919, 169, 1163—1166. A NEW complex chromic sulphate, in which the

whole of the sulphate is masked, has been prepared as follows. A $N/2$ solution of chromic sulphate was allowed to stand until equilibrium was reached as far as the formation of the green sulphate is concerned, and then the solution was evaporated to saturation point in a vacuum. Excess of alcohol was added and the lilac-grey precipitate obtained was immediately collected, washed with ether, and allowed to dry. So prepared it contained $18H_2O$, and the whole of its SO_4 was precipitable by barium chloride. On exposure to air it rapidly and spontaneously changed, and at the end of two days, whilst retaining its colour, only contained $16H_2O$ and no longer gave a precipitate with barium chloride. When left in a desiccator its water content dropped to $12H_2O$.—W. G.

Lead dioxide; Colorimetric determination of — in litharge. W. V. Morgan. J. Ind. Eng. Chem., 1919, 11, 1055.

FIVE grms. of the litharge is boiled for 1 min. with 2 grms. of aniline hydrochloride dissolved in 10 c.c. of water and 5 c.c. of concentrated hydrochloric acid; the mixture is then cooled, filtered, and the colour of the filtrate compared with standards prepared with known amounts of lead peroxide under the same conditions. The lead peroxide oxidises the aniline with the formation of Aniline Purple, and the intensity of the coloration is proportional to the amount of peroxide.—W. P. S.

Lead peroxide; Reguline —. W. Palmaer. Med. K. Vetensk. Nobelinst., 1919, 5, No. 31, 1—15.

COMPACT lead dioxide is prepared by the electrolysis of lead nitrate solution. The product is greyish-black and has a crystalline appearance. It contains no impurities, has hardness 5—6, sp. gr. 9.360. It is similar to the mineral plattnerite. The specific resistance at 18° C. is 0.000815 ohm, and the specific conductivity 1180 ohm⁻¹.—J. F. S.

Hydrogen peroxide; Catalytic decomposition of —. G. Phragmén. Med. K. Vetensk. Nobelinst., 1919, 5, No. 22, 1—13.

IN phosphate mixtures the catalysis of hydrogen peroxide reaches a maximum at $pH=11.8$. The velocity decreases with both increasing and decreasing hydrogen ion concentration. This is due to the formation of a stable sodium salt of hydrogen peroxide in strong alkaline solutions. Fresh yeast decomposes hydrogen peroxide without sending a soluble enzyme into the surrounding solution. The reaction is of the first order, and the reaction constant is proportional to the quantity of yeast. The catalytic action per cell or per gm. can be increased by treating the yeast with sugar solution before use.—J. F. S.

Mercury compounds; Microchemical identification of soluble and insoluble —. G. Denigès. Bull. Soc. Pharm. Bordeaux, 1919, No. 2. Ann. Chim. Analyt., 1919, 1, 383—385.

CHARACTERISTIC crystals are formed when a small quantity of a mercury compound or salt is treated on a microscope slide with a drop of potassium iodide-bromide solution. Mercurous compounds and mercuric cyanide are treated previously with bromine-water. In the case of mercuric iodide, the crystals obtained on evaporating its alcohol or acetone solution serve for the identification. —W. P. S.

Protactinium; Chemical properties of —. Part I. Separation of protactinium from pitchblende. O. Hahn and L. Meitner. Ber., 1919, 52, 1812—1828.

PROTACTINIUM was separated from the other radioactive substances in pitchblende by three different

methods based on the assumption, thereby proved correct, that this new radioactive element belongs to the tantalum group. Since by all methods the yields of protactinium as measured by the activity of the final residues per grm. of pitchblende only varied by about 10%, it was concluded that within this limit the element had been quantitatively separated long with the added tantalum. The activity of the protactinium residue, however, was much lower than would be expected relative to the activity of the uranium associated with it in the pitchblende if the proportion of uranium atoms disintegrating into the actinium series was as much as 8%, as given by Rutherford. A value of 3% seems more in accord with the facts, and is, moreover, in better agreement with the value found by Antonoff (Phil. Mag., 1913, 26, 1058) in the case of uranium Y, the immediate parent of protactinium. The question whether it may be possible to separate protactinium as a new chemical individual depends largely on the at present uncertain life of the element. A lower limit for this could probably be found by an investigation of uranium salts of known age, say 25 or 50 years old, for their protactinium content.—G. F. M.

Sulphur; Method of dissolving elementary — for analysis. A. P. Bierregaard. J. Ind. Eng. Chem., 1919, 11, 1055.

ABOUT 0.1 grm. of the sulphur is dissolved in dry bromine, 10 c.c. of concentrated nitric acid is added, and the mixture is warmed. After removing excess of bromine and nitrous fumes by continuing the heating, the solution is diluted with 100 c.c. of water, a few c.c. of hydrochloric acid is added, and the solution boiled to expel the remainder of the nitric acid. The sulphuric acid is then precipitated with barium chloride.—W. P. S.

Hydrogen cylinders. Boek. See X.

Alkali sulphides. Bennett and Bennett. See XV.

Carbon monoxide. Lamb and Larson. See XIXb.

Potash. Keitt and Shiver. See XXIII.

PATENTS.

Nitric acid and tetroxide of nitrogen [from nitric acid containing oxides of nitrogen]; Process of producing pure concentrated —. O. Jensen, Assignor to Norsk Hydro-Elektrisk Kvaestof-aktieselskab, Christiania, Norway. U.S. Pat. 1,324,255, 9.12.19. Appl., 4.2.19.

THE acid to be purified is passed in counter current to nitric acid vapour, and the nitrogen tetroxide expelled is condensed. The remaining acid is subjected to distillation and dephlegmation to produce dilute, liquid acid and vapour of highly-concentrated acid, a portion of the latter being condensed and the remainder employed for treating a further quantity of the impure acid.—W. E. F. P.

Ammonia; Apparatus for the synthetic production of —. General Chemical Co., Assignees of F. W. de Jahn, New York. Eng. Pat. 124,762, 28.2.18. (Appl. 7872/19.) Int. Conv., 3.11.17.

AN apparatus for the conversion of mixed hydrogen and nitrogen gases into ammonia consists of a pressure system comprising a cylindrical metal shell screw-threaded at each end, with apertured top and bottom covers each held in place by an annular screw plug engaging with the thread on the shell and provided with plates for the apertures, bolted to the covers, combined with a heat interchanger comprising a shell enclosing a series, at least three in number, of concentric tubes, gas passages for the gases flowing in opposite directions being formed between the walls of the tubes.—S. S. A.

Ammonia; Purification of —. C. L. Parsons, Washington, D.C., and L. C. Jones, Syracuse, N.Y., U.S.A. Eng. Pat. 136,342, 20.1.19. (Appl. 1442/19.)

AMMONIA is freed from phosphine by treating it at 50°–60° C. with air in the presence of a catalyst comprising carbon or a carbonaceous substance coated or impregnated with a metal or metallic compound, preferably of the silver group, reduced or partially reduced by ignition in an atmosphere of hydrogen. The catalyst, packed in removable containers, is placed in a series of chambers which are operated alternately in parallel and are provided with steam jackets whereby the desired temperature is obtained to expel ammonia after the oxidation of the phosphine. The ammonium phosphate produced during the process is held by the catalyst, and when the catalyst becomes clogged the ammonia is expelled and recovered by heating the chamber jacket and blowing through the catalyst steam and air heated in pipes passing through the jacket. The carbon is revived by thorough washing and drying.—S. S. A.

Ammonia; Method of extracting — [from crude ammoniacal liquor]. E. Piron, New York. U.S. Pat. 1,324,979, 16.12.19. Appl., 20.7.18.

CRUDE ammoniacal liquor is mixed with alkali solution at atmospheric temperature, and the mixture is passed continuously into the bottom of a decanting vessel. The clear liquor passes continuously into a still for extraction of the ammonia, and the solid matter which settles in the bottom of the decanting vessel is withdrawn from time to time.—L. A. C.

Alumina poor in iron; Process for producing —. Det Norske Aktieselskab for Elektrokemisk Ind. Norsk Industri-Hypotekbank, Christiania, Norway. Eng. Pat. 125,578, 24.12.18. (Appl. 21,659/18.) Int. Conv., 8.4.18.

LABRADORITE or a similar mineral is treated with nitric acid on the counter current principle to obtain a neutral solution to which, after filtration from insoluble matter, a basic precipitant, e.g., alumina, is added, thereby precipitating iron and silica which are removed. The solution is evaporated and the residue is calcined at about 300° C. by means of hot gases from a nitric acid plant in the absorption tower of which the nitrous gases produced by the resulting decomposition of the aluminium nitrate are recovered. The undecomposed calcium and sodium nitrates are separated from the alumina by lixiviation and recovered by evaporation for use as fertilisers.—S. S. A.

Alumina [from alkaline solutions containing silica]; Process of recovering —. G. G. Miner, Berkeley, Assignor to The Miner Chemical Corporation, San Francisco, Cal. U.S. Pat. 1,324,318, 9.12.19. Appl., 19.12.17.

ALUMINA and silica are precipitated separately by carbonating the alkaline solution at successively higher and lower temperatures.—W. E. F. P.

Alkali sulphides [and iron]; Method for the production of —. E. Bergve, Notodden, Norway. Eng. Pat. 129,629, 13.6.19. (Appl. 14,950/19.) Int. Conv., 8.7.18.

A SUITABLE mineral is fused in a shaft furnace at a temperature of about 1250° C. with a mixture of pyrites and silicon in the form of ferro-silicon. The resulting products separate into layers, iron at the bottom and alkali sulphides at the top, which can be drawn off through suitably placed tap-holes.—S. S. A.

Cyanides; Manufacture of —. F. J. Metzger, Assignor to Air Reduction Co. U.S. Pat. 1,322,195, 18.11.19. Appl., 7.3.19.

ALKALI cyanide is produced by heating to a suitable temperature a mixture of carbonaceous material, alkali metal compound, and finely-divided iron, in presence of nitrogen, the amount of iron being so small that no appreciable quantity of ferrocyanide is formed when the furnace product is leached with an aqueous solvent.—J. H. L.

Alkali metal cyanides; Production of —. C. B. Jacobs, Bloomfield, N.J., Assignor to Air Reduction Co. U.S. Pat. 1,321,119, 9.12.19. Appl., 19.12.16.

AN alkaline-earth carbide is heated with an excess of carbon and an alkali metal compound, and the heated mass is subjected to the action of nitrogen.—S. S. A.

Sodium fluoride; Method of producing —. F. C. Bowman, Assignor to General Chemical Co., New York. U.S. Pat. 1,324,030, 9.12.19. Appl., 9.3.18.

A suspension of sodium fluosilicate is treated with soda at such a rate that the mixture remains acid or neutral.—S. S. A.

Lead arsenate; Method of making —. M. W. Butler, Assignor to The Harshaw, Fuller, and Goodwin Co., Cleveland, Ohio. U.S. Pat. 1,324,300, 9.12.19. Appl., 8.12.17.

THE lead compound obtained as a by-product in the preparation of sodium nitrite, by heating sodium nitrate with metallic lead, is treated with arsenic acid.—W. E. F. P.

Soluble salts from flue-gases; Apparatus for collecting and isolating —. L. D. Gilbert, P. S. Taylor, J. G. Dean, and L. E. Elder, Victorville, Cal. U.S. Pat. 1,324,737, 9.12.19. Appl., 25.6.17.

IN an apparatus for recovering soluble salts in suspension in superheated steam the latter is passed into a condensing chamber, and the condensed liquid is passed through a filter into an evaporating chamber maintained under reduced pressure, the two chambers being separated by a wall through which heat is readily transmitted. Means are provided for withdrawing a portion of the liquid from the evaporating chamber, for cooling the portion so withdrawn to effect crystallisation of the soluble salts, and for returning the mother liquor to the evaporating chamber.—W. E. F. P.

Manganese peroxide; Process of obtaining —. E. H. Westling, Antioch, Cal. U.S. Pat. 1,325,129, 16.12.19. Appl., 14.3.18.

A STEEL in the production of manganese peroxide consists in treating a mixture of manganese oxide ore and a liquid with hot sulphurous acid, thus producing a solution of manganese sulphate.—B. M. V.

Phosphine; Process of oxidising — and apparatus therefor. J. D. Davis, Washington, D.C. U.S. Pat. 1,325,145, 16.12.19. Appl., 19.8.18.

A GASEOUS mixture containing phosphine and oxygen is brought into contact with a catalyst capable of oxidising phosphine selectively and contained in a steam-jacketed chamber. The steam jacket is traversed by tubes which communicate at one end with the space containing the catalyst, and at the other end with a supply of compressed air.—W. E. F. P.

Aluminium chloride; Process of manufacturing —. J. R. Mardick, New York. U.S. Pat. 1,325,203, 16.12.19. Appl., 12.3.19.

AN aluminium compound or ore is heated with

carbon bisulphide and chlorine at such a temperature that aluminium chloride is evolved.—B. V. S.

Sulphur dioxide; Preparation of — from magnesium sulphate or double salts of magnesium. Chemische Fabr. Rhénania, Stolberg. Ger. Pat. 300,716, 26.5.16.

HYDROGEN sulphide is passed over magnesium sulphate heated to redness, the magnesium sulphate being kept in excess throughout the process by moving it in an opposite direction to the gas. If the hydrogen sulphide is pure the sulphur dioxide produced will be free from impurities and can be easily condensed, but it may be contaminated with nitrogen if the hydrogen sulphide is derived from the Chance process. If double salts of magnesium and alkalis are used these are converted into sulphides, which may be freed from magnesium oxide by treatment with water and converted into carbonates by means of carbon dioxide.—W. J. W.

Sulphur dioxide; Method of removing — from gases by thionate solution. W. Feld, Gasabteilung, G.m.b.H., Linz. Ger. Pat. 314,627, 27.8.18.

THE gases are washed with a solution of ammonia, and the resulting solution of sulphite or bisulphite is treated with tetrathionate to produce thiosulphate. Aqueous solutions of alkaline ammonium salts may be used for washing the gases. Instead of tetrathionate a solution of tetrathionate with other thionates may be employed. The treatment of the sulphite with thionate may be carried out continuously or intermittently.—W. J. W.

Oxides of sulphur; Manufacture of —. Badische Anilin- u. Sodafabrik, Ludwigshafen. Ger. Pat. 300,763, 20.4.15.

FINELY-POWDERED wood charcoal, lignite, or coke is mixed with magnesium sulphate in the proportion of one atom of carbon per molecule of magnesium sulphate. To bring about an intimate admixture of the two ingredients a solution of magnesium sulphate may be used. The mixture is heated to about 600°–700° C., decomposition commencing at a dull red heat. The process yields magnesium oxide and sulphur dioxide without simultaneous formation of magnesium sulphide.—W. J. W.

Aluminium sulphate; Production of an iron-free solution of — from clay. Papierfabrik Köslin A.-G., Köslin. Ger. Pat. 301,614, 28.9.16.

CLAY is treated with an alkali bisulphate in presence of an oxidising agent such as barium peroxide, the mixture being heated to 300°–400° C. for a long period. Iron compounds are thus converted into basic iron sulphate, which is insoluble in water or alum solutions.—W. J. W.

Zinc sodium sulphate; Preparation of —. Farbenfabriken vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 301,782, 20.2.17.

A DOUBLE sulphate of zinc and sodium which may be used instead of zinc sulphate is prepared by treatment of solutions, containing both sulphates, with a bisulphate; or substances containing zinc may be dissolved in a solution of sodium bisulphate, and the zinc sodium sulphate separated from the solution by addition of bisulphate.—W. J. W.

Zinc compounds; Preparation of pure — from impure materials. Farbenfabriken vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 303,802, 20.2.17.

THE method described in Ger. Pat. 301,782 (see preceding abstract) may be applied to the recovery of zinc from zinc-containing compounds such as pyrites waste, zinc oxide, etc.—W. J. W.

Alkali carbonates; Process for manufacture of —. E. Hene, Stassfurt, and A. van Haaren, Leopoldshall. Ger. Pat. 302,535, 17.11.16.

MONOALKALI cyanamide is decomposed by water at a temperature above 100° C., or better, above 120° C. The cyanamide is first freed from calcium compounds by addition of a carbonate. The decomposition may take place under pressure. The products obtained are an alkali carbonate and ammonia together with carbon dioxide. The solution is treated with a caustic alkali and the ammonia is driven off. It may alternatively be strongly heated and the expelled gases allowed to react with bases.

—W. J. W.

Iodine; Process of purifying —. R. M. Carter, Baltimore, Md., Assignor to U.S. Industrial Alcohol Co. U.S. Pat. 1,324,761, 9.12.19. Appl., 24.12.18.

IODINE containing iodine compounds as impurities is subjected to the action of a heated gaseous medium, by which means the iodine is separated from the impurities and vaporised.—W. J. W.

Sulphur; Process for manufacture of —. Harburger Chem. Werke Schön und Co., and W. Daitz, Harburg. Ger. Pat. 303,233, 29.9.16.

SULPHUR dioxide and hydrogen sulphide enter the top and bottom respectively of a reaction chamber and interact in presence of steam. Escape of undecomposed hydrogen sulphide is prevented by employing a tower-shaped chamber having the upper portion divided into compartments placed one above another and connected by pipes. Salt solution may be sprayed into the chamber. The sulphur is readily obtained in the form of grains. The process is conveniently employed in connection with the manufacture of sulphuric acid, and the hydrogen sulphide may be obtained from the calcium sulphide derived from the Leblanc soda process.—W. J. W.

Ammonia; Process for effecting fixation of atmospheric nitrogen and production of —. E. W. Haslup, New York. Eng. Pat. 135,889, 24.10.18. (Appl. 17,364/18.)

SEE U.S. Pats. 1,310,478–9 of 1919; this J., 1919, 629 A. (Reference has been directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 4037 and 16,760 of 1893, 18,792 of 1894, and 10,305 of 1907; this J., 1894, 517, 886; 1895, 967; 1908, 1052.)

VIII.—GLASS; CERAMICS.

Thermo-couple installation in annealing kilns for optical glass. E. D. Williamson and H. S. Roberts. Papers on Optical Glass, No. 15. Geophysical Lab., Washington. Bull. No. 152, Amer. Inst. Min. and Met. Eng., Aug., 1919, 1445–1453.

IN the annealing of optical glass, temperatures up to 650° C. have to be measured correct to within 5° C. The necessity for careful regulation of temperature during the beginning of cooling demands the use of a temperature-measuring device, of which the sensitivity is well within 5° C. A pyrometer installation for use with an installation of annealing kilns comprising eleven kilns in one room, twenty in a second, and four in a third is described. The couples employed were constituted of chromel-alumel, and were used in conjunction either with a potentiometer or a modification thereof of the pyrovolt type. The system of wiring, whereby all temperatures could be read at one central station, is detailed. The system is such that the leads from the constant temperature junctions and the leads from the variable temperature

junctions are led to terminals enclosed in a box, so that all these terminals are at the same temperature. Copper leads are employed to connect these terminals with the potentiometer. It appears that best results in the annealing of optical glass are secured if the glass is maintained for six hours at the temperature at which the strain disappears and is thereafter cooled at a rate that is rapidly increased as the temperature falls. The temperature should be taken at more than one point in a kiln. No difficulty was experienced in training men to take the readings properly, and errors in reading were eliminated entirely.

—J. S. G. T.

Red glass; Production of selenium —. F. A. Kirkpatrick and G. G. Roberts. J. Amer. Ceram. Soc., 1919, 2, 895–904.

IN producing selenium red glass the details of the working appear to be almost as important as the composition of the glass, but when the correct conditions are obtained selenium is more reliable than copper and cheaper than gold. Two kinds of glass were made: a soft-working zinc-alkali glass (sand 53.70, potash 16.25, soda ash 16.25, zinc oxide 11.37, and cadmium sulphide, selenium, and borax each 0.81%), and plate glass (sand 62.60, soda ash 18.60, hydrated lime 17.30, cadmium sulphide 0.85, and selenium 0.65%). The materials were mixed in 30 lb. batches and introduced in three portions into a closed "monkey pot," which had previously been heated to 1400° C. After three hours the glass was allowed to fine for five hours, and after a further three hours the pot, with its contents, was allowed to cool slowly until ready for use. Shortly before withdrawing any glass the molten material should be skimmed, the "scum" being used as cullet in the next melt. During the melting and fining periods the pot must be kept closed to prevent the escape of selenium vapour. Portions of the zinc glass were withdrawn at 680° C. on a blowpipe and gathered, rolled, pressed in a mould, allowed to cool, flashed in the glory hole, allowed to cool, and then placed in thelehr, the whole of these operations occupying 130 secs.; the portions withdrawn from thelehr after 36 hours were all deep red in colour. Portions of the plate glass were gathered, cooled to 400° C., flashed, pressed in the mould, and then put in thelehr, the whole of these operations occupying 110 secs, but these times may not apply to larger batches. Cooling to 400°–700° C. immediately after gathering appears to be essential to the production of a red colour; in the case of blown ware the cooling may occur during the blowing, but the glass must not be reheated until it has been cooled sufficiently. After this reheating has no effect. The ware blown in moulds sometimes did not develop its colour until after it had been placed in thelehr.—A. B. S.

Clays; Relations between the chemical composition, microscopic structure, and ceramic qualities of —. L. Bertrand and A. Languine. Comptes rend., 1919, 169, 1171–1174.

THE petrographic study of a large number of samples of clays shows that the view, often held and used in the interpretation of the results of chemical analysis, that the alkalis present are in the form of mica is incorrect and must be abandoned. Micaceous plates were found only very exceptionally. In respect to the classification of clays as "fat" or "lean," the authors find that there is no rigorous relationship between the character of a clay in this respect and its relative proportions of silica and alumina. Certain clays rich in silica are abnormally "fat," owing to the fact that their free silica is in a colloidal state or else in the form of extremely fine quartz grains. Similarly, many exceptions were found to the rule that the higher the

alumina content the more refractory is the clay. The chemical composition of a clay does not suffice as a criterion of its ceramic qualities.—W. G.

Refractories Manufacturers' Association; Work of the technical division of —. [Manufacture of firebrick.] R. M. Howe. J. Ind. Eng. Chem., 1919, 11, 1145—1146.

THE composition and properties of clay deposits vary greatly, and the miner should be able to accept or reject at sight each piece as it is obtained. A systematic investigation has shown that some of the best clays are often rejected through ignorance. The proportion of water used in preparing the clay paste affects the porosity and modulus of rupture of the burned product. The greater the proportion of water (within working limits) the less the porosity and the greater the strength of the burned product. An excess of water reduces the strength, but does not affect the density so much as an insufficiency. Prolongation of the pugging also increases the strength. Slow drying is more effective than rapid drying in reducing shrinkage, in preventing the "creeping" of arches and "opening" of joints, and in producing a firm bond and resistance to abrasion. Studies of the porosity-burning temperature graphs of clays give useful information as to the heat-treatment necessary to obtain a good product and indicate to the user how different clays are likely to behave in service, i.e., whether they will remain open or become dense. The investigations show that every step in the manufacture of firebricks is important, and that this industry is not so crude as is often supposed.

—A. B. S.

Refractories for industrial furnaces; Selection of —. W. F. Rochow. J. Ind. Eng. Chem., 1919, 11, 1146—1149.

A SUPERFICIAL consideration of the chemical and physical properties of a refractory material is not sufficient and is often misleading, as good results are sometimes obtained from products, the chemical composition and physical properties of which appear to make them unsuitable; thus silica bricks have been used for many years for lining kilns used for burning magnesite and in the roofs of tanks and pots for melting glass, although they are subjected to alkaline vapours from the glass batch. *Silica bricks.*—The most important properties of silica bricks are their high thermal conductivity, mechanical strength, resistance to abrasion at furnace temperatures, expansion when heated, tendency to spall, and refractoriness. Their good conductivity is specially useful in coke-ovens and muffle-furnaces. Spalling may be reduced and often eliminated by heating and cooling slowly through the critical range at which most of the expansion occurs (below 500° C.). Unlike fireclay and magnesia refractories, there is only a small difference between the softening and melting points of silica, so that deformation does not occur until the latter is almost reached. Hence in arches silica bricks with a covering of kieselguhr or other insulator are efficient where fireclay bricks would soften gradually and collapse. The temperature at which bricks are deformed when heated under a pressure of 25 lb. per square inch is a good indication of their usefulness. The sp. gr. is a criterion of the extent to which the permanent expansion should be carried at the first firing, 238 being suggested as the upper limit for a well-burned American silica brick. An examination of the various zones in a used firebrick often shows the manner in which deterioration occurs. *Magnesite bricks.*—American magnesite—unlike the Austrian material—is deficient in iron, but the addition of 4.5–8% of ferric oxide widens the range of vitrification and develops good bonding properties at furnace temperatures and other properties similar to

those of Austrian magnesite. At high temperatures magnesite bricks are mechanically weak and have a feeble resistance to abrasion. When magnesite is heated it expands considerably, the maximum expansion occurring at 1350° C.; on this account bricks tend to spall when cooled too rapidly. In several cases—especially in back walls, bulkheads, and gas-ports of basic open-hearth furnaces, and in the side walls of electric steel furnaces—soft steel boxes filled with compressed dead-burned magnesia are preferable to magnesite bricks. The exposed face of the steel container melts and impregnates the magnesia forming a jointless surface with very small tendency to spall. Specially shaped refractory blocks and bricks should be avoided wherever possible, as they are more liable to defects in workmanship than the standard shapes.

—A. B. S.

Refractories in the iron and steel industries. C. E. Nesbitt and M. L. Bell. J. Ind. Eng. Chem., 1919, 11, 1149—1151.

THE most important working qualities of silica bricks can be determined by spalling and hot crushing tests, and those of fireclay bricks by these two tests and a slagging test. Silica bricks show a fall in crushing strength when heated to 1350° C. Fire-cracks also reduce the durability of bricks exposed to great thermal changes. Roofs of open-hearth furnaces should last for 200 heats, and they then have a spalling loss of about 20%. In a recent shipment of clay chequer bricks the average spalling loss was 1.4% for normal bricks and 60% for hard-burned ones. The finer the material of which silica bricks are made, the greater the spalling loss and the shorter the "life" in the furnace. Variation in the quality of refractory bricks is also shown by measuring the depths of penetration of a steel ball under a pressure of 1600 lb. at 1350° C. and by an impact test in which a steel ball weighing 21 lb. is dropped on the heated brick from heights increasing successively by 2 in.—A. B. S.

Refractories; Superior —. R. C. Purdy. J. Ind. Eng. Chem., 1919, 11, 1151—1153.

THE limiting factor in the development of electric furnaces is the lack of adequate refractories. Refractories of superior quality are also required for high-tension insulators and for the manufacture of new alloys and glasses. Although special materials, such as pure silicon carbide, fused alumina, sintered magnesia, fused spinels, crystallised sillimanite, calcined zirconia, etc., will be used, most of the new requirements will be met by intelligent adaptation of materials in general use, probably by fusing them so that they attain complete chemical stability and constancy in volume. The ordinary classification of refractories as neutral, basic, or acid has little value except for steel-making, and a few slag tests will show that the corrosive effect of a slag is the same on either basic or acid refractories. There is no need to have a neutral course between basic and acid bricks in a furnace.

—A. B. S.

Sheet steel and iron; Cleaning — for enamelling purposes. R. R. Danielson. J. Amer. Ceram. Soc., 1919, 2, 883—894.

IN addition to the customary methods of cleaning sheet steel and iron by scaling and then pickling in acid solutions, the author advocates other methods, including the use of chemical grease-removers, such as a boiling solution of sodium hydroxide or carbonate, especially with the aid of an electric current. Naphtha and gasoline are not suitable for the removal of grease from sheet metal prior to enamelling, as they leave a thin film of grease on the metal. For heavy sheets, sand-blasting is effective but not for light-gauge sheets and special shapes. Electrolytic pickling in dilute sul-

phuric acid is quicker and cheaper than simple acid pickling, and the effect is more uniform. In all cases any acid used should be removed in a neutralising bath consisting of a 0.4% solution of soda ash or preferably a mixture of 12 lb. of caustic soda, 6 lb. of soda ash, and 400 galls. of water. Immediately after withdrawal from this bath the metal should be rinsed in water and dried by artificial heat at 150°–230° F. (55°–110° C.), a current of clean air being preferable to the use of steam coils, as the latter do not always dry the ware thoroughly.

—A. B. S.

Refractory problems of gas industry. Fulweiler and Taussig. See IIa.

Sulphite-cellulose waste liquors. Philippsthal. See V.

Thermal expansion. Gray. See XXIII.

PATENTS.

Glass furnace; Apartment tank —. *Regenerative glass-furnace.* W. N. Mathews, Wellsburg, W. Va. U.S. Pats. (A) 1,324,917 and (B) 1,324,918, 16.12.19. Appl. 3.12.18.

(A) A GLASS furnace tank is composed of three adjacent, independent compartments, each of which is used successively as a melting, a planing, and a flowing pot. A combustion chamber and muffle arches are placed over the tank, the arches separating the compartments from the combustion chamber. (B) A regenerative glass furnace comprises a pair of adjacent but independent refining chambers, a cooling chamber in front of the refining chambers and connected to them, a combustion chamber above all the chambers, and muffle arches separating the refining and cooling chambers from the combustion chamber.—A. B. S.

Glass-tank furnace. L. T. Sherwood, Connellsville, Pa., Assignor to Pennsylvania Wire Glass Co., Philadelphia, Pa. U.S. Pat. 1,325,172, 16.12.19. Appl., 31.7.16.

In a glass tank furnace, with a side wall built of refractory material, water-boxes are arranged on the outside of the wall, above and below the glass level, one face of each water-box being in direct contact with the wall.—A. B. S.

Ovens or kilns for use in the manufacture of tiles, pottery and other ware, and for other purposes. J. H. Marlow, Stoke-on-Trent, Eng. Pat. 136,127, 11.8.19. (Appl. 19,688/19.)

In a tunnel kiln a current of cold air is drawn through passages leading to a pair of metal air chambers arranged longitudinally at each side of the cooling zone and heated by radiation from the cooling goods and truck. The supply of air is controlled by dampers. The heated air passes to the flame conduits of the kilns or to another portion of the works.—A. B. S.

Abrasive and process of making the same. W. Strutt, Pittsburgh, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,324,215, 9.12.19. Appl., 4.3.15.

BLOCKS of abrasive material are impregnated with an oil, and oil adhering to the surface is then removed by burning.—L. A. C.

Refractory composition. E. T. Ferngren, Washington, D.C. U.S. Pat. 1,324,546, 9.12.19. Appl., 26.2.15.

A REFRACTORY composition consists of zirconium oxide, calcined magnesite, and alumina in equal parts, with a clay bond.—A. B. S.

Glass; Machines for forming articles of —. W. J. Miller, Swissvale, Pa., U.S.A. Eng. Pats. 130,986 and 130,987, 8.8.18. (Appls. 19,290 and 19,291/19.) Int. Conv., 11.8.17.

IX.—BUILDING MATERIALS.

Concrete; Effect of vibration, jiggling, and pressure on fresh —. D. A. Abrams. *Structural Materials Research Lab., Lewis Inst., Chicago, Ill.* 3. Reprint from Proc. Amer. Concrete Inst., 1919, 15. 23 pages.

TESTS showed that tamping, vibration, and jiggling are of value in forcing the concrete into its place in intricate moulds or where reinforcement is used, but when applied after the completion of the moulding such treatment does harm. Tamped concretes were usually weaker than those which were hand-puddled, a large tamper being less effective than a smaller one. The advantages derived from tamping, vibrating, or jiggling during moulding are that the concrete is placed more certainly in position and is finished dryer; any excess of water is brought to the surface and should then be removed, and coarser aggregate may be used and therefore a less proportion of water employed. When the concrete has begun to set tamping, vibration, or jiggling will notably reduce its strength.—A. B. S.

Marbles of the U.S.A.; Physical and chemical tests on the commercial —. D. W. Kessler. U.S. Bureau of Standards, Tech. Paper, No. 123, 15.7.19. 54 pages.

FIFTY average samples of American marble were tested for crushing and tensile strength, resistance to freezing, absorption, specific gravity, porosity, staining, permeability, expansion, and electrical resistivity. The average crushing strength of cubes of white marble of uniform texture was 9174 lb. per sq. in., that of prisms of the same material and cross-sectional area was 7828 lb. per sq. in. Prisms and cylinders of the same height had approximately the same crushing strength. Other marbles varied from 7850 to 50,205 lb. per sq. in., the dolomites being stronger than the calcite marbles. When the test-pieces were wet the crushing strength was 7856–36,156 lb. per sq. in. Transverse tests on bars 3 in. × 1½ in. × 6–12 in. were made in both directions of the bedding. The transverse strength is always very low when applied parallel to the bedding. The transverse strength (modulus of rupture) perpendicular to the bed was 900–4388 lb. per sq. in. Tensile tests showed the occurrence of definite lines of weakness not otherwise ascertainable, but harmful to the stone, as they allow more ready access of water to the interior of the marble. The tensile strength was 328–2254 lb. per sq. in. Repeated freezing and thawing of wet cubes resulted in a loss of weight and strength in most cases, though a few specimens gained slightly. In one case the loss in strength was over 28%. On repeatedly heating to 150° C. the specimens lost 1.9% by weight. The absorption (which is much less than the true porosity) should be expressed by volume; if expressed as a percentage of the weight of the specimen, a light marble might appear to be more absorptive than a heavier one, when actually the reverse is the case. In the specimens examined the absorption never exceeded 1.2%, the average being 0.2%. The apparent sp. gr. of calcite marbles is 2.70–2.73, and that of dolomitic marble 2.84–2.86. The porosity varied from 0.40 to 2.09, and the relation of absorption to total pore space is important in predicting the resistance of the marble to frost. The permeability of marble to air is much greater than to water; there is no relation between this permeability and the loss of strength on freezing. The

staining power was determined by drilling a $\frac{3}{8}$ in. hole to the centre of a 2 in. cube of the marble, filling the hole with eosin solution, and after six hours sawing the cube in half and noting the extent of the penetration. The results varied greatly, but could not be correlated with any other property. Volume-resistivity tests for showing the electrical insulating power indicated that the resistivity is greatly affected by moisture, but the values obtained with samples dried for several days in the laboratory air appear to be satisfactory. They lie between 1.0 and 1.2×10^6 . The effect of soaking test-pieces for three months in a solution of carbonic acid varied with different specimens, the dolomitic marbles being least affected. The permanent expansion between 0° and 100° C. was 0.2 – 0.3 mm. for each 3 ft. of length. On repeatedly heating to about 500° C. the permanent expansion, as well as the rate of expansion, was gradually reduced and became negligible after five or six heatings. Marble which has expanded on heating does not return to its original volume.—A. B. S.

Silicate of soda. Vail. See VII.

Sulphite-cellulose waste liquors. Philippsthal. See V.

PATENTS.

Concrete; Manufacture of —. D. Dale, Newcastle-under-Lyme, Staffs. Eng. Pat. 136,075, 16.4.19. (Appl. 9678/19.)

THREE parts of sand and one of cement are mixed in a dry state, then moistened with water and feathers added and the whole thoroughly mixed. After being allowed to stand until partly set the mixture is beaten up so as not to allow it to set too firmly. The plastic concrete thus produced is frequently mixed whilst it is being used. The feathers are preferably added gradually during the mixing.—A. B. S.

Cement; Process of making —. H. D. Baylor, Sellersburg, Ind., Assignor to Louisville Cement Co., Louisville, Ky. U.S. Pats. (A) 1,323,952 and (B) 1,323,953, 2.12.19. Appl., 17.3.19.

CEMENT is rendered slower setting (A) by mixing the comminuted cement with quicklime in a proportion between 6 to 1 and 10 to 1 by weight, and stirring into the mixture the amount of water approximately necessary completely to hydrate all free calcium oxide, including that resulting from the break up of aluminates; or (B) by adding quicklime to the cement, comminuting the mass, adding oily or waxy material at the same time as the water necessary to hydrate all the calcium oxide, and grinding. In the case of (B) a plastic cement is obtained.—E. W. L.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron deposition; Electrolytic —. W. A. Macfadyen. Faraday Soc., Dec., 1919. [Advance proof.]

EXPERIMENTS were conducted in order to find the most favourable conditions for effecting a substantial electrolytic deposition of iron upon mild steel, using a solution of ferrous ammonium sulphate as electrolyte. Current was supplied from accumulators to ensure regularity, and the anodes consisted of plates of Swedish iron. The ratio of the active area of anode to cathode was always large. The mild steel cathodes were cleaned thoroughly, both chemically and by abrasion, and a film of copper was applied before the electrolytic deposition of iron. It was found later that it was possible to deposit iron directly upon steel

if the steel had been subjected previously to an acid electrolytic cleaning bath, making it the anode instead of the cathode, as is the usual practice. The effect of variation in concentration and acidity of the electrolyte at atmospheric temperature showed that solutions containing 315 grms. of ferrous ammonium sulphate per litre yielded deposits equally as good with current densities as high as 16 amp. per sq. ft. as those obtained from dilute solutions (50–100 grms. per litre). Some intermediate concentrations (100–300 grms. per litre) consistently gave pitted deposits. Trouble was experienced in neutral solutions from deposits, presumably some form of ferrous hydroxide, which was almost eliminated when the electrolyte was acidified. The cathode efficiency reached a maximum (98%) when the solution was of about 0.001 *N* acidity, and decreased progressively when the acidity was increased. After protracted use of the same solution the quality of the deposits deteriorated owing to the accumulation of organic impurities, but it was found possible to expel these by boiling the solution or to absorb them by means of wood charcoal. Experiments made at different temperatures showed that 65° C. was most satisfactory, as deposition could be effected from a concentrated solution at fifty times the rate possible in a cold dilute solution. If the current density exceeds 200 amps. per sq. ft. the deposit tends to have a specular appearance and the adhesion suffers. If the steel base with deposited iron be annealed above the A3 point for pure iron, the two become strongly welded together. Subsequent case-hardening is usually advisable to harden the electrolytic iron.

—C. A. K.

Ferromanganese; Saving of — by use of fluorspar in open-hearth furnace practice. E. Goldmann, Stahl u. Eisen, 1919, 39, 1385–1387.

INSTEAD of adding only a small quantity of fluorspar as in ordinary practice for fear of damaging the hearth, the author has added 1000 kilos. per charge without undue injury to the hearth. The greatest advantage of its use lies in the saving of ferromanganese. The author made cast steel shell with 0.6–0.9% Mn without the addition of ferromanganese or spiegeleisen. The phosphorus content was 0.1–0.15%, but other qualities were made with a maximum of 0.04% P. It was not possible to produce iron with carbon as low as 0.15% without the use of manganese. It is important to use a pig iron with the highest possible manganese content, this being attained if necessary by large additions of open-hearth furnace slag to the mixture. This keeps the sulphur low, and by the use of fluorspar a thick layer of thin fluid slag is formed, which protects the original manganese in the charge, and makes possible the removal of phosphorus and part of the sulphur.—T. H. Bu.

[Steel] process; Acid electric furnace —. L. B. Lindemuth. American Foundrymen's Assoc. Blast Furnace and Steel Plant, 1919, 7, 595–596.

THE acid process of steel making differs from the basic process in that it is a single slag process, and the slag varies considerably both in composition and quantity. The controlling factor of the acid process seems to be the quantity of iron oxide present at the time the charge is melted, irrespective of the method of its introduction. Before any additions are made to the charge the molten acid slag is principally a complex silicate of iron and manganese containing 50–60% SiO_2 . About 90% of the manganese in the charge passes into the slag as oxide, and the oxidation of the bath and of subsequent additions of manganese appears to be controlled by the FeO content of the slag. Iron oxide in the slag is not reduced satisfactorily by the addition of carbon, because an acid silicate dissociates less easily than a basic silicate, and also iron is more

stable in a silicate in which iron is the predominating base. Further, silica is reduced by carbon at the temperature attained near the arc, and the silicon in the metal may be increased by this reaction during a "blow." Iron oxide in the slag is reduced partially by additions of lime and manganese ore, thus enabling the necessary reducing conditions to be produced. A typical finishing slag contains SiO_2 58.70%, CaO 21.25%, MnO 12.01%, FeO 3.10%, MgO 1.15%, Al_2O_3 3.35%, P 0.003%, and S 0.005%. In order to overcome the variables introduced by rust or other oxidised products, the most satisfactory method would be to withdraw the slag immediately after the charge is melted. The view is expressed that the basic electric process gives conditions which tend to a fewer number of inclusions in the metal than the acid process.

—C. A. K.

Steel: Relationship between transverse rail fissures, flakes and defects in fusion welds in —.
S. W. Miller. Chem. and Met. Eng., 1919, 21, 729–733.

ALL the evidence indicates that transverse fissures, flakes and intergranular ruptures in welds are due to the same cause, *viz.*, films of oxide, which are possibly ultramicroscopic. These films are usually present in the ingot, and may be produced by overheating of the metal in the course of manufacture. When they are due to iron oxides in small quantity only they may be removed by heat treatment, as stated by Giolitti (this J., 1919, 289 A) and Humfrey (Carnegie Schol. Memoirs, Iron and Steel Inst., 1914, 4, 80), but this would probably not be possible when they are caused by larger amounts of iron oxide. Such defects in welds as are visible under the microscope may be eliminated by thorough fusion in the welding process, so as to bring all the oxide and dirt to the surface. This is more difficult in electric welding owing to oxidising conditions being more pronounced, and to the fact that the metal is being constantly added from the electrode. It is possible that ultramicroscopic films in welds may be eliminated by heating the weld in a reducing atmosphere. It is suggested that the examination of sound rails and of those known to contain fissures by polishing and etching small sections and bending them under the microscope would give further information, especially in the case of steel showing low elongation. Experiments indicated that welds made with good material will give from 22 to 30% elongation in the weld when tested in a piece $\frac{1}{2}$ in. thick, whereas welds containing films of oxide, even though invisible, will only show an elongation of 2 to 10%. The same low elongation is also to be observed in the case of flaky steel and defective rails.—C. A. M.

Steel; Solution theory of —, and the influence of changes in carbide concentration on the electrical resistivity. E. D. Campbell. Faraday Soc., Dec., 1919. [Advance proof.]

It is becoming recognised generally that the electrical resistivity of metallic solutions is dependent on the concentration and degree of dissociation of the solutes just as truly as is the conductivity of aqueous solutions, and it is only logical to express the concentrations in similar terms, *e.g.*, millatoms (mgrm.-atoms) per c.c. The influence of decarburisation of steel on the electrical resistivity has been examined. Similar bars cut from steels of different type were decarburised in an atmosphere of moist hydrogen, and quenched, and the resistivity of the bars before and after decarburisation was compared with that computed from Benedick's formula. Benedick's law assumes that equi-atomic concentrations in iron exert equal influence on the resistivity, and the specific resistance under consideration must be that of steel in the

hardened condition. The differences found between the measured and computed resistivities are so great that the formula cannot be considered as having any particular significance. The conclusions of Le Chatelier (this J., 1898, 669) concerning the influence of C, Si, Mn, and Ni, are confirmed, but his view that Cr, W, and Mo have but little influence on the resistivity of steel is not substantiated. If a steel, containing such elements, which possess a stronger affinity for carbon than has iron, is annealed, a large part of these elements will be found in the form of precipitated carbides when the metal is annealed, so that, not being in solution, these elements can have little effect. If the steel, however, is decarburised, a marked increase in resistivity is noted, due to the solution of the liberated element in the iron. Manganese and nickel show distinctly less influence on the resistivity than would be due to an equal atomic concentration of carbon. Figures obtained show that there must be some marked differences in the constitution of the carbides in various steels.

—C. A. K.

Chrome-nickel steels; Substitutes for —. E. Kothny. Stahl u Eisen, 1919, 39, 1341–1348.

WAR conditions in Germany necessitated economy in the use of nickel, and an investigation was carried out to find substitute alloy steels for nickel and chrome-nickel steels in highly stressed parts. The requisite properties after suitable heat treatment are high elastic limit, good elongation and reduction of area even with high tenacity, high resistance to impact, and fibrous fracture of longitudinal and transverse test pieces in combination with high tenacity. The alloying elements available were manganese and chromium, together with silicon. Tests were carried out on electric and Martin furnace heats first on small specimens and then in some cases on specimens of practical size. The results showed that only plain chromium or plain manganese steels will give the desired properties. The compositions recommended are C 0.20–0.45%, Mn 0.4–0.6%, Si 0.2–0.3%, Cr 1.0–1.3%, and C 0.2–0.4%, Si 0.2–0.3%, Mn 1.5–1.8% respectively. Silicon-steels, chromium-silicon steels, and manganese-silicon steels are not satisfactory for the purpose desired. As a case-hardening steel the following is recommended: C 0.1–0.2%, Mn 0.4–0.6%, Si 0.2–0.3%, Cr 0.8–1.1%.—T. H. Bu.

Self-hardening steels; Critical points of —. P. Dejean. Comptes rend., 1919, 169, 1043–1045.

A DISTINCT discontinuity between the formation of pearlite or troostite, and martensite has been previously noted (this J., 1917, 965). The critical point corresponding to the formation of the former has been denoted "A," and to the formation of the latter "B." From a study of the cooling curves of a nickel-chrome-copper steel, cooled from 700° to 100° C. in about 150 mins., but from different initial temperatures, the formation of martensite appears to occur in two stages, denoted by critical points B₁ and B₂. The point "A" is indicated only when the initial temperature of cooling is 790° C. When cooled from 800° C., "A" is considerably suppressed and a second critical point (B₁) is observed about 420° C. A further point (B₂) occurs at about 230° C. if the steel is cooled from 850° C., and when cooled from still higher temperatures B₁ disappears and the point B₂ exists alone. The steel is relatively soft if only the critical point "A" is in evidence during cooling, but becomes harder when B₁ predominates, with still greater hardening when point B₂ is evident. It is concluded that the maximum hardness is attained in at least two stages and that for the same steel there are several marsensitic forms.

—C. A. K.

[Steel] hydrogen cylinders; Results of cold working of —. E. Bock. *Stahl u. Eisen*, 1919, 39, 1467—1470.

MECHANICAL tests were made on pieces of a hydrogen cylinder which exploded disastrously in use although it had successfully withstood the acceptance test under a pressure of 225 kilos. per sq. cm. The fragments showed generally longitudinal fracture, i.e., in the direction of drawing. The mechanical tests showed high elastic limit and small elongation and reduction of area, and the effects of cold working were traceable throughout the cross-section of the pieces. The tenacity was satisfactory, but brittleness was shown in bending and impact tests, especially on transverse specimens. The brittleness could be removed by annealing for 10 mins. at 600° C. Microscopical examination showed non-metallic inclusions elongated in the direction of drawing, also that the material had been drawn into layers.—T. H. Bu.

Sulphur and chromium in steel; Determination of —. L. A. Goldenberg. *Chem. and Met. Eng.*, 1919, 21, 628.

SULPHUR is determined by the evolution method and chromium by a slight modification of the silver nitrate-ammonium persulphate method. The sample is placed in an Erlenmeyer flask which is closed by a two-holed rubber stopper carrying a thistle funnel through which sulphuric acid is added, and a tube leading into a vessel containing ammoniacal cadmium chloride solution. The sample is heated with the sulphuric acid, and when evolution is complete the cadmium chloride is titrated, with the addition of starch solution and hydrochloric acid, to a deep blue end point with standard potassium iodide-potassium iodate solution. For the determination of chromium, nitric acid-silver nitrate solution is added to the contents of the Erlenmeyer flask, which are boiled gently until nitrous fumes are expelled, the mixture is diluted, heated to boiling, ammonium persulphate solution is added, and the boiling is continued until manganese dioxide is completely precipitated. Hydrochloric acid is then added to prevent sudden boiling over and to destroy the manganese dioxide, and the contents of the flask are boiled for about 15 mins. until the silver chloride is coagulated. After cooling, potassium ferriocyanide is added, and the mixture is titrated with standard ferrous ammonium sulphate solution which has been standardised against a standard steel. The method presents considerable advantage in point of time over the older permanganate process, there is less risk of inaccuracy through splashing, and the end point of the titration is more definite. Moreover, by using sulphuric acid as the solvent in the sulphur determination the difficulty experienced when using hydrochloric acid of acid fumes passing into the cadmium solution and causing loss of ammonia by volatilisation as ammonium chloride is obviated.—S. S. A.

Steel; Determination of gas in —. P. Oberhoffer and A. Beutell. *Stahl u. Eisen*, 1919, 39, 1584—1590.

THE gases dissolved in iron and steel are determined by means of a new apparatus consisting of a quartz tube in a magnesia crucible heated externally by an electric furnace and exhausted to a vacuum by means of a mercury pump. Steel together with twice its weight of a 50/50 tin-antimony alloy is placed in the tube, which is then exhausted and heated to a temperature of 1100° C. In 30—40 mins. the whole of the dissolved gas is evolved. This is then collected and analysed. A number of samples of acid and basic steels both before and after deoxidation and at various intervals of casting were examined. The composition

of the evolved gases varied within the following limits: CO, 0—15.4, CO₂ 43.7—88.7%, H₂, 3.8—41.0, N₂, 1.8—27.9. The volume of gas evolved ranged from 38.7 c.c. per 100 grms. of steel in the case of a basic steel to 195 c.c. per 100 grms. in the case of an acid steel. Deoxidising reduced the volume of gas in acid steel and increased it in basic steel. Rolling an acid steel diminished the amount of gas evolved from 118 c.c. to 63 c.c. per 100 grms.

—J. W. D.

Soaking pits; Electrically heated — in the steel industry. T. F. Baily. *Amer. Iron and Steel Inst.*, May, 1919. *Chem. and Met. Eng.*, 1919, 21, 624—627.

FOR heating operations subsequent to melting and refining, electrically heated furnaces effect considerable saving of labour as compared with fuel-fired furnaces, and on account of the greater precision of the treatment the percentage of material rejected through defective heat treatment is much lower. Thus, in electrically heated soaking pits, although the fuel cost is higher than in fuel-fired pits, the material produced is not so liable to defects caused by lack of uniformity in the temperature of the heated ingot, excessive oxidation of the ingot, and the like. The author describes a continuous type of electric reheating furnace adapted for rolling mills of relatively small capacities or for reheating steel of high quality, and a combined fuel and electric furnace suitable for dealing with a large tonnage. For exact work in annealing, hardening, and other heat treatment the higher fuel cost of electric heating plant is more than compensated by the superiority of the material produced.—S. S. A.

Electric rotating furnace; The Booth — [for melting metals]. C. H. Booth. *Amer. Inst. Chem. Eng.*, June, 1919. *Chem. and Met. Eng.*, 1919, 21, 636—638.

THE furnace, which is built in several sizes, comprises a shell carried on motor-driven rollers working without gearing in tracks on the shell, and is arranged to revolve at the rate of two revolutions per minute. The current is carried to the electrodes by short pieces of flexible cable which connect to the track and the current is supplied to the tracks by shoes which press against them and form a sliding contact. The electrodes are regulated in the smaller furnaces by hand screws and in larger plant automatically. The lining is made with as few joints as possible and the door is in one piece with a hole in the centre to admit the electrode. In this type of furnace graphite electrodes are used on account of their greater conductivity. The furnace and electrical connections are simple in design, the charge is uniformly mixed, and the cost of operation and upkeep charges are low.—S. S. A.

Melting non-ferrous metals and their alloys in the electric furnace. E. F. Collins. *Chem. and Met. Eng.*, 1919, 21, 673—679.

THE requirements for melting non-ferrous metals and their alloys are continuity of operation, freedom from oxidation and volatilisation, that the slags should not mix but be easily removable, and that the furnace and its atmosphere should be entirely free from sulphur. The transfer of heat is best accomplished by radiation to the top of the bath and conduction to the bottom. The best type of furnace is one in which a readily controllable amount of heat is generated between a fixed and a movable electrode, the distance between them being maintained so that they give rise to many chains of series and multiple contact or resistance areas, enveloping adjacent electrode faces. This type is described in detail. Comparative costs of melting

in electric and fuel-fired furnaces are tabulated and sources of loss in each type are outlined. From a summary of the results obtained the author concludes that for brass melting the electric furnace is superior.—W. J. W.

Aluminium corrosion as affected by basic pigments.
H. A. Gardner. Paint Manufacturers' Assoc., U.S.A., Circ. No. 79. Dec., 1919. 2 pp.

UNDER severe conditions of exposure, aluminium shows very rapid corrosion, resulting in a rough surface to which soot will adhere tenaciously. Pigments of a basic nature, i.e., red lead, white lead, zinc oxide, etc., when ground in water etch aluminium rapidly, whilst silicious earth and other neutral crystalline pigments have no such action. Thus, if aluminium surfaces for such purposes as automobile bodies, etc., be primed with a paint composed of a mixture of basic pigments and silicious earth pigments, under favourable conditions, etching of the surface with production of a firm foundation for succeeding coats will take place. Progressive corrosion could be inhibited by application of many outer coats. For other forms of aluminium which may be exposed to the weather, the use of a high-grade spar varnish is recommended to obviate obscuration of the bright colour of the metal.—A. de W.

Metal deposits: Measurement of the minimum thickness of — by their electromotive force. S. Procopiu. Comptes rend., 1919, 169, 1030—1031.

WHEN zinc is deposited upon an electropositive metal the E.M.F. diminishes rapidly at first and more slowly later. If the current is stopped, the deposited layer dissolves and gives rise to a counter E.M.F. which gradually increases to a point at which a sharp increase takes place. Thus, with a deposit of zinc 3.5μ in thickness, on platinum, the E.M.F. after stopping the current increases from 0.011 to 0.14 volt in 7 mins., to 0.85 in 8 mins., and to 1.02 volts in 9 mins. If deposits of more than minimum thickness are allowed to dissolve and a curve is drawn with the thickness of the metal as abscissa and the time taken to reach the break in the E.M.F. as ordinates, a point is obtained which represents the minimum thickness deposited electrolytically, assuming the E.M.F. to be set up as soon as the metal is deposited. If solution only of the metal occurred the curves representing the thickness of the deposit : time, would be of equal inclination to the abscissa, but this is not the case :—

	Metal on which zinc is deposited.					
	Al.	Pt.	Fe.	Ag.	Cu.	Ni.
Tangent of angle ..	0.13	0.24	0.3	0.4	0.7	1.1
Minimum thickness of zinc deposit (μ) ..	0.3	0.8	1.6	2.8	4.2	4.8

The varying values for the minimum thickness of a zinc deposit on different metals are explained by the extent of diffusion of the zinc layer into the underlying metal, or by an alloying action between the two metals.—C. A. K.

Antimony ores; Treating —. G. P. Hulst. Amer. Inst. Min. and Met. Eng., Sept., 1919. Chem. and Met. Eng., 1919, 21, 727.

THE demand for antimony for shrapnel during the war made it profitable to work ores containing as little as 20% Sb, and many small mines were opened. Low-grade sulphide ores (20—45% Sb) were produced in Nevada, California, Idaho, Utah, and Mexico, whilst the principal oxide ores came from Mexico and Oregon. High-grade sulphide ores (stibnite) with 55—60% Sb were imported into the United States from Bolivia, China, and

Alaska. The ores were mixed with waste material such as battery plates, lead oxide, paint, etc., to furnish the necessary lead, and were treated in residue and blast furnaces. The charges consisted of various sulphide ores, containing 20—60% Sb and 6—45% SiO_2 , or of oxide ores with 20—40% Sb and 10—45% SiO_2 . All ores containing silver (e.g., 3.40%) were treated in the residue furnace, the sulphur, iron, and copper-forming matte containing part of the silver, whilst the bulk of the latter was found in the lead bullion. The antimony slag was sufficiently low in silver to justify its being smelted in the blast furnace to antimonial lead. Owing to the high proportion of zinc and arsenic in lead refinery by-products a slag was used containing:— SiO_2 , 26%; FeO , 40%; and CaO and ZnO , 20—24%. The average amounts of antimony and lead in the slag were 0.66 and 2.36% respectively. The blast furnace loss was 2.4% Sb and 1.5% Pb. The furnace charge varied from 2500 to 3000 lb., and the coke ratio was 13%. Blast pressure was maintained at 10—12 oz. The two furnaces smelted 60—90 tons of material per day, and produced 30—35 tons of antimonial lead of the following average composition:—Sb, 13.00; Cu, 0.15; As, 0.75; and Pb 86.1%.—C. A. M.

Lead-sodium-mercury and lead-sodium-tin alloys.
T. Goebel. Z. Ver. deuts. Ing., 1919, 424—430. Chem.-Zeit., 1919, 43, Rep., 312.

WHEN an alloy of lead, sodium, and mercury is cast in a cold mould, "pipes" are formed in the casting. These "pipes" are more pronounced the greater the mercury content of the alloy, and the more nearly the percentage of sodium present approximates to an average value of 2—3%. There is very little pipe formation if the alloy is poured into a warm mould. The maximum value of the hardness of a lead-sodium-mercury alloy containing 1.8% Na is the same as the maximum hardness of a lead-sodium or a lead-sodium-tin alloy containing 0.8% Na. In the case of lead-sodium-mercury and lead-sodium-tin alloys, the same hardness is obtained with a larger percentage of sodium present than in the case of the binary lead-sodium alloys. The tenacity of these latter alloys is considerably increased by addition of tin. The tenacity of the lead-sodium-mercury and lead-sodium-tin alloys diminishes with increase in the percentage of sodium present. Lead-sodium-mercury alloys containing 1%—2% Na, on account of their hardness and the small tendency to crack which they exhibit, are specially suitable for use as bearing metals.—J. S. G. T.

Cobalt; Passivity of —. H. G. Byers and C. W. Thing. J. Amer. Chem. Soc., 1919, 41, 1902—1908.

WHEN cobalt is used as anode in the electrolysis of 0.02N sulphuric acid or sodium sulphate at 0° C. it becomes passive if a high current density is employed, but if a low current density is used it remains active and passes into solution. When potassium bichromate is present cobalt readily assumes the passive condition under all circumstances. It differs from iron and nickel mainly in the fact that it will not become passive when used as anode with low current density, but if it has once assumed the passive condition it will remain so even though the current density is reduced. (See also J. Chem. Soc., Feb., 1920.)—J. F. S.

Metals; Atomic structure of — in solid solution.
A. L. Feild. Chem. and Met. Eng., 1919, 21, 566—570.

SOLID solution alloys, comprising hardened steels and electrical resistance alloys, differ in physical properties from their components. They probably are analogous to supercooled liquids with the same

atomic structure as the molten metals, and they do, in fact, approach the latter in similarity of properties such as electrical resistance and temperature coefficient of resistance. The formation of an amorphous phase is assumed and is confirmed by experiments on the specific resistance of nickel-chromium and gold-silver alloys. The hardness of alloys is also due to the presence of amorphous metal. Electrical methods of examination of metals are recommended.—W. J. W.

Coagulation [of gold sols]. A. Westgren. *Arkiv Chem. Min. Geo.*, 1917-18, 7, No. 6, 1-30.

The velocity of coagulation by means of electrolytes is independent of the size of the colloidal particles. The velocity of coagulation by means of electrolytes is greater the larger the migration velocity of the anion. The dependence of the velocity of coagulation on the temperature is due both to the change in the velocity of the Brownian movement and to the change in the specific coagulation power of the electrolyte with temperature. (See also J. Chem. Soc., Feb., 1920.)—J. F. S.

Refractories in iron and steel industries. Nesbitt and Bell. See VIII.

Hydrogen overvoltage. MacInnes and Contieri. See XI.

Galvanised iron. Gardner. See XIII.

Blast-furnace waste gases as fertiliser. Riedel. See XVI.

Thermal expansion. Gray. See XXIII.

Thermal analysis. Merica. See XXIII.

PATENTS.

Cast-iron; Refining —. H. Barnes, Assignor to R. H. Genter, Chattanooga, Tenn. U.S. Pat. 1,322,516, 25.11.19. (Appl. 6.2.19.)

Pig-iron is melted in a chamber having a curved and tapering bottom portion and run direct into the rear end of a refining chamber, at the front end of which is a gas producer. Rear blast pipes, with internal fuel jet pipes, project downwards and rearwards into the bottom of the melting chamber, and front blast pipes project in a similar manner into the front end portion of the refining chamber above the hearth.—E. W. L.

Alloy steel; Surface-stable —. P. A. E. Armstrong, Londonville, N.Y. U.S. Pat. 1,322,511, 25.11.19. (Appl. 24.5.19.)

An alloy steel of high surface stability contains 3-50% Cr and 0.5-3.5% C. The carbon content must not be more than 10% of the chromium and silicon together if the latter be less than 13%, or more than 16.6% if the chromium plus silicon be more than 13%. Silicon (5-8%) should be present to the extent of over twice the carbon content, and silicon and chromium together between 5 and 58%. —C. A. K.

Magnesium; Process of electrodepositing —. G. O. Seward, New York. Eng. Pat. 120,908, 11.11.18. (Appl. 18,467/18.) Int. Conv., 19.11.17.

See U.S. Pats. 1,310,119-50 of 1919; this J., 1919, 643 A. The molten electrolytic bath contains magnesium, sodium, and barium fluorides, and has sp. gr. above 3 and preferably approximating 4.

Crucible furnaces. W. A. Shilton and R. A. Munden, Coventry. Eng. Pat. 135,620, 17.10.18. (Appl. 19,810/18.)

A CRUCIBLE furnace, heated by gas or hydrocarbons, is fitted with a cover having a central opening for the escape of the hot gases from the furnace. The

cover contains an annular air chamber through which air for combustion at the burner is circulated and preheated, and the refractory slab forming the bottom of the cover may be shaped so as to direct the hot gases into the crucible.—C. A. K.

Electric arc shaft furnaces. J. Bibby, London. Eng. Pat. 135,905, 28.11.18. (Appl. 19,671/18.)

IS furnaces of the type having a crucible surmounted by a shaft, from which gases are withdrawn and re-introduced into the crucible portion for cooling purposes, coal dust or other finely-divided carbonaceous material is caused to enter with the gases in order to reduce the oxidation of the electrodes. The endothermic reaction between the carbon and the carbon dioxide in the gases also increases the cooling effect.—C. A. K.

Furnace; Metal-melting —. L. C. Harvey, Malden. Eng. Pat. 136,213, 12.11.18. (Appl. 18,529/18.)

A FURNACE, capable of being rocked, is fitted with a charging tube through the arch, which allows the charge to be fed underneath the slag covering molten metal on the furnace bottom. The central portion of the bed of the furnace is level, and one end inclines gradually to a pouring spout for the complete removal of metal and slag. The opposite end of the bed has a sharp inclination adapted for the agitation of the metal during rocking, and a tubular spout, dipping below the surface of the metal, allows the withdrawal of small quantities of molten metal free from slag.—C. A. K.

Furnaces [for heat treatment]. H. Fuller, Manchester, and R. A. Bedford, Sheffield. Eng. Pat. 136,270, 12.12.18. (Appl. 20,739/18.)

For the tempering of steels by the salt bath process, two crucibles mounted in a single furnace casing are employed. One of the crucibles is heated in the usual manner by one or more gas burners, and the second is heated wholly, or partly, by the hot products of combustion passing from the first crucible chamber.—C. A. K.

Alloys of copper, zinc, and lead; Manufacture of —. J. P. Arend, Dommeldange, Luxembourg. Eng. Pat. 135,702, 31.1.19. (Appl. 2195/19.)

THE alloys contain copper and zinc in about the same proportion as for brass (15-45 Zn, 85-55 Cu) together with a quantity of lead (e.g., 10-60%) in excess of that necessary for the formation of a saturated solution of lead in the other metals. The excess of lead renders the alloys plastic and suitable for the construction of bearings. The alloys possess the physical qualities of phosphor bronze.—C. A. K.

Cast metal objects; Process of producing —. E. C. R. Marks, London. From Kohler Co., Kohler, Wis., U.S.A. Eng. Pat. 135,893, 1.12.19. (Appl. 17,731/18.)

METAL moulds are coated with a material consisting of 88% of quartz, 10% of white clay, and 2% of borax, which is applied by means of a spray and acts as a heat insulator, thereby protecting the casting from chilling.—J. W. D.

Copper-aluminium alloy. W. J. Haywood, London. Eng. Pat. 135,963, 10.12.18. (Appl. 20,550/18.)

A COPPER aluminium bronze containing 6.2-11.0% Al, 0.5-5.0% Fe, 0.1-6.0% Ni, 0.06-1.0% Ti, and 0.03-3.0% Mn.—J. W. D.

Lead and silver from sulphide ores and metallurgical products; Recovery of —. Amalgamated Zinc (De Bavay's), Ltd., Melbourne, Australia, and S. Gancelin, Brooklyn, N.Y., U.S.A. Eng. Pat. 135,968, 12.12.18. (Appl. 20,761/18.)

THE ore is heated in a closed furnace with zinc

chloride, and the lead and silver chlorides in the product are either extracted together with a cold concentrated brine solution or a brine solution containing cupric or ferric chloride, or extracted separately by leaching out the lead chloride with hot concentrated brine containing 3—4% of zinc chloride and removing the silver chloride with a cyanide solution or a brine solution containing 2—4% of ferric chloride. Zinc-free lead is recovered by crystallisation of the chloride and remelting of the crystals in a bath of zinc chloride in the presence of molten lead and zinc, when zinc chloride is formed and pure lead tapped off.

—J. W. D.

Sintering concentrates, distilling shale, and the like; Apparatus for —. J. M. Berglund, Ludvika, Sweden. Eng. Pat. 136,076, 17.4.19. (Appl. 9903/19.)

A hollow cylinder divided into compartments by radial partitions, is mounted on a horizontal hollow shaft. Shallow trays with perforated bottoms are fitted in the separate compartments at the periphery of the drum, and are adapted to receive material to be heated as the drum rotates. Suction is applied to the central shaft and slots on the upper portion allow air to be drawn through each compartment in turn as it passes over the opening. An ignition appliance, e.g., a fire-grate, in contact with the drum, is fixed near to the feed hopper. Guide-plates allow of the convenient feeding into the sintering trays, and of the discharge of the material after heating.—C. A. K.

Ores; Concentration of —. S. Tucker, E. Edser, and Minerals Separation, Ltd., London. Eng. Pat. 136,255, 10.12.18. (Appl. 20,541/18.)

Is a froth-flotation process the mineral separating agent consists of a mixture of a soap solution with an alkali sulphide or a mixture of the ingredients necessary to produce soluble soaps and alkali sulphides. Such a mixture may be formed, for example, by treating an alkali hydroxide or carbonate with sulphur and adding a fatty acid.

—C. A. K.

Flotation apparatus and process. W. L. Ziegler, Sunset, Idaho. U.S. Pat. 1,324,139, 9.12.19. Appl., 4.4.17.

A FLOTATION process for the treatment of ore pulp composed of a mixture of ore in fine division, water, and a frothing agent, consists in injecting air into an upwardly moving column of the pulp to produce a body of froth at the top, and beating the froth in a confined space at the top of the column as it leaves the column so as to break up the larger air bubbles and produce a creamy froth whilst permitting the excess of air to escape. The froth and the gangue are then separated.—G. F. M.

Heat-treating and quenching device. T. F. Baily and F. T. Cope, Assignors to The Electric Furnace Co., Alliance, Ohio. U.S. Pat. 1,322,750, 25.11.19. Appl., 12.8.18.

A QUENCHING tank is located beneath, and extends beyond one side of, a vertical furnace. Articles to be treated are carried on a track above the tank, and raised into the furnace, afterwards being discharged into the tank, in which they may be held suspended from the track.—C. A. K.

Gold; Method of recovering —. R. Thayer, Philadelphia, Pa. U.S. Pat. 1,323,124, 25.11.19. Appl., 9.7.18.

COLLOIDAL gold-bearing sands or ground rock ores containing colloidal gold are roasted in presence of an alkali, to render the gold more readily recoverable.—C. A. K.

Cyaniding process; Electrolytic —. W. A. Hussey, Oakland, Cal., Assignor to J. H. Alling and F. M. Wright, San Francisco, Cal. U.S. Pat. 1,324,352, 9.12.19. Appl., 15.4.13. Renewed 17.4.19.

METAL-BEARING material is mixed with a solution of sodium cyanide together with a halogen sodium compound, and the solution is electrolysed, using a mercury cathode. A current of sufficient intensity to form a halogen-cyanogen compound and sodium is employed, and the metallic sodium is removed from the action of the current.—C. A. K.

Solder; Aluminium —. C. L. Bonsteel, Moose Jaw, Sask., Canada. U.S. Pat. 1,323,520, 2.12.19. Appl., 11.1.19.

An aluminium solder comprising a mixture of 29 lb. of tin, 11 lb. of zinc., 2 lb. of phosphor-tin, 2 lb. of aluminium, and 2 grains of bismuth.—C. A. K.

Metals; Precipitation of — from solutions. A. Gordon, Johannesburg, Transvaal. U.S. Pat. 1,323,588, 2.12.19. Appl., 18.2.18.

METALS are precipitated from solutions by means of amalgamated aluminium.—E. W. L.

Alloy. A. Cohn, New York. U.S. Pats. (A) 1,323,833 and (B) 1,323,834, 2.12.19. Appl., (A) 2.5.19 and (B) 6.6.19.

(A) THE alloy contains platinum together with from 1—15% of gold. (B) The alloy contains an excess of refined platinum, together with iridium and gold.
—A. E. D.

Oxidation of molten metal; Method of preventing —. F. F. McIntosh, Glen Osborn, Pa. U.S. Pat. 1,324,458, 9.12.19. Appl., 29.5.19.

WHEN the metal is being poured a protecting blanket of non-oxidising vapour heavier than air is generated by the heat of the metal from a substance which is liquid at atmospheric temperatures and pressures.—J. W. D.

Coating metals with mercury; Method of —. J. P. A. McCoy, Wilkinsburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,324,835, 16.12.19. Appl., 12.4.15.

THE surface of a piece of tin is coated with mercury and the mercury transferred from the tin to the surface to be coated by placing the coated surface of the tin, wet with hydrochloric acid, upon the surface to be coated, rubbing the two surfaces together, and removing the tin and acid.—J. W. D.

Hardness of metals on the Brinell system; Portable spring-actuated devices for testing the —. F. C. Fairholme, W. H. Hatfield, and G. Stanfield, Sheffield. Eng. Pat. 136,129, 22.8.19. (Appl. 20,617/19.)

Steel; Apparatus for the production of — directly from pig iron. A. Nielsen, Copenhagen. U.S. Pat. 1,318,906, 14.10.19. Appl., 6.8.10.

SEE Ger. Pat. 313,756 of 1917; this J., 1920, 29A.

Managanese and its alloys; Manufacture of — in the electric furnace. C. L. Lenior, Arreau, France. Eng. Pat. 126,303, 30.4.19. (Appl. 10,755/19.) Int. Conv., 8.1.14.

SEE Fr. Pat. 474,927 of 1914; this J., 1915, 1099.

Alkali sulphides [and iron]. Eng. Pat. 129,629. See VII.

XI.—ELECTRO-CHEMISTRY.

Temperature regulator; [Electric] furnace —. W. P. White and L. H. Adams. *Phys. Rev.*, 1919, 14, 44—48.

By making the heating coil of an electric furnace one arm of a Wheatstone bridge and combining this with a galvanometer regulator, thus keeping the resistance of the coil constant, the temperature of electric furnaces may be kept constant. The device is effective regardless of variations in the current supply and requires no attention, particularly in the case of furnaces which are not directly influenced by the temperature of the room or where the surrounding air is kept constant. The arrangement operates as follows: Changes in the temperature of the furnace and consequently in the resistance of the heating coil operate a beam which either hits or misses a contact maker which controls a suitable relay. This relay operates a larger magnet which controls the main current. The power available in this regulator is very large, nothing has to be inserted in the furnace cavity, and there is practically no lag. By means of this regulator furnaces at temperatures between 500° C. and 1400° C. may be kept constant to 0.1° for hours.—J. F. S.

Electrode; The Söderberg self-baking continuous —. J. W. Richards. *Trans. Amer. Electrochem. Soc.*, 1920, 1—15. [Advance copy.]

AN unbaked electrode mixture of coke, anthracite, pitch, and tar is fed continuously to the furnace, and the mass is baked by heat conducted from the lower end of the electrode, or by the heat contributed by hot gases or by radiation, or by heat derived from the passage of current through the baked portion. The electrode mixture is stamped into an iron casing, and new sections of the casing are bolted, riveted, or welded to the upper part of the casing as required. The thin casing may be provided with internal perforated projecting ribs, the casing thus serving to support the unbaked mixture, to reinforce mechanically the unbaked portion, and to facilitate the passage of current from the electrode holder to the electrode. The holder encircles the iron casing near the roof of the furnace, and the baking usually begins a short distance above the electrode holder. The baked carbon is hard, dense, and of the quality of previously-baked carbon; the use of the electrodes improves the regularity of running of electric furnaces, and they are cheaper than ordinary carbon electrodes in furnaces in which they can be used.—B. N.

Electrochemical production of organic compounds; Commercial possibilities in —. C. J. Thatcher. *Amer. Electrochem. Soc.*, Sept., 1919. *Chem. and Met. Eng.*, 1919, 12, 663—666.

THE advantages of and objections to the adoption of electro-chemical methods of producing organic compounds are enumerated, and the catalytic processes involved and the use and nature of the diaphragms used are briefly discussed. Suggestions for processes of commercial practicability include manufacture of alcohol, aldehydes, azo and hydrazo compounds, dyestuffs of the triphenylmethane type, sulpho acids, etc. The industry has reached its greatest development in Germany.—W. J. W.

Hydrogen overvoltage. II. Applications of its variation with pressure to reduction, metal solution and deposition. D. A. MacInnes and A. W. Contieri. *J. Amer. Chem. Soc.*, 1919, 41, 2013—2019.

A CHANGE in pressure on chemical reactions involv-

ing the evolution of hydrogen brings about changes in the rate of reaction and in the reaction efficiency which are in the direction expected from the change of hydrogen overvoltage with pressure. These include a decrease in the rate of solution of metals in electrolytes, an increase in the efficiency of reduction by metals, and an increased efficiency of metal deposition.—J. F. S.

Colloids; New method of electric synthesis of —. T. Svedberg. *Med. K. Vetensk. Nobelinst.*, 1919, 5, No. 10, 1—18.

HIGHLY disperse gold and silver sols may be obtained by the following method: A quartz tube, 1 mm. diam., in which a small hole is bored carries two electrodes which enter at the top and bottom and are arranged so that the arc will be formed opposite the hole. A current of nitrogen enters the quartz tube both at the top and at the bottom. This tube is placed in a glass jacket, which contains about 30 c.c. of the dispersion medium (alcohol), and which in its turn is surrounded by a cooling jacket containing ice and salt or solid carbon dioxide and alcohol. An electromagnet is placed with its poles on either side of the small hole in the quartz tube. The current (1 amp., 220 volts) is switched on and an arc of the usual type appears for about a second, then, owing to the melting of the lower electrode (anode), the quartz tube becomes somewhat stopped and the electrode is protected from the dispersion medium; the arc becomes a pointed flame, and this is drawn through the hole in the tube by the action of the magnet. Metallic clouds appear and are absorbed by the dispersion medium.—J. F. S.

Fixation of nitrogen. Briner and Baerfuss. *See VII.*

Chlorine and alkali. Chaleyser. *See VII.*

Electrolysis of alkali chlorides. Briner and others. *See VII.*

Electrolytic estimation of halogens. Reedy. *See VII.*

Perechlorate. Williams. *See VII.*

Potassium permanganate. Thompson. *See VII.*

Lead peroxide. Palmaer. *See VII.*

Passivity of cobalt. Byers and Thing. *See X.*

Electric rotating furnace. Booth. *See X.*

Potentiometers. White. *See XXIII.*

Electrometric titration. (1) Treadwell. (2) Treadwell and Weiss. *See XXIII.*

Electrolytic estimation of anions. Lasala. *See XXIII.*

PATENTS.

Electric furnaces. E. and W. Waring, Sheffield. *Eng. Pat.* 135,674, 31.12.18. (*Appl.* 21,863/18.)

THE furnace is provided with a circular undulating hearth sloping downwards from the walls into a recess, and rising again to a projection extending upwards in the centre of the hearth. The electrodes are arranged in three sets of groups, one set of groups in the outermost higher part of the hearth, an intermediate set of groups in the lower part of an undulation nearer the centre, and a central set of groups radiating from the centre and extending between groups of the intermediate set. The electrodes are grouped in such a manner that the largest number and smallest size are in the outermost groups, with a smaller number of larger electrodes in the groups nearest the centre, and in the

radiating groups the largest electrodes are also nearest the centre of the hearth. A number of electrodes may be interconnected upon a water-cooled box or frame to form a group, and any number of the boxes or frames may be interconnected by water pipes to form a single water-cooled system. A three-phase supply system is connected in delta, in such a manner that each outer main is connected to a series of groups of electrodes consisting of intermediate groups alternating with outermost groups, each series being closed upon itself to form a single electrode. The inner main is directly connected to one of the central set of groups, which are interconnected amongst themselves to form the third electrode, the sides of the delta being formed through the molten material.

—B. N.

Electric furnace. F. Jacoviello, Milan, Italy. U.S. Pat. 1,324,156, 9.12.19. Appl., 3.1.19.

THE material to be heated is contained in a number of cup-shaped vessels arranged in line, and separated by and embedded in powdered material of low electrical conductivity. The vessels themselves have a relatively high electrical conductivity. The conducting electrodes are embedded in the powdered material at the two ends of the series of vessels, so that the current traverses the material of low conductivity alternately with the vessels of high conductivity. The material surrounding the vessels is thus highly heated by the current, but the vessels themselves are not directly heated by the current owing to their low resistance.—W. F. F.

Electro-osmotic apparatus for removing liquids from substances; Method and apparatus for charging material to —. Apparatus for electro-osmotically removing liquids from materials. Elektro-Osmose A.-G. (Graf. Schwerin Ges.), Berlin. Eng. Pats. (A) 135,815 and (B) 135,819, 25.6.18. (Appls. 10,453 and 10,458/18.) Int. Conv., 20.10.17.

(A) THE material is charged into an electro-osmotic apparatus of the filter-press type by feeding it along the electrode at which the liquid is separated. The charging openings are arranged next the electrodes at which the liquid is separated, and the charging ducts may extend through the electrodes and terminate in nozzles directed radially. (B) The anodes, in apparatus of the chamber filter-press type, consist of solid plates hollowed out on each side to form double chambers, and are arranged alternately with two perforated plates as cathodes held by intermediate and outer frames to form permeable chambers.—B. N.

Resistance material; Electrical — and process of making. O. Hutchins and A. W. Olver, Assignors to The Carborundum Co., Niagara Falls, N.Y. U.S. Pat. 1,322,573, 25.11.19. Appl., 25.3.19.

A SHAPED body of clay, containing iron oxide and silicon carbide substantially free from carbon, is heated to a temperature sufficient to fuse the clay and reduce at least a portion of the iron oxide.

—B. N.

Compounds; Method of decomposing — [by electrolysis]. V. M. Weaver, Harrisburg, Pa., Assignor to Weaver Co., Milwaukee, Wis. U.S. Pat. 1,323,936, 2.12.19. Appl., 24.1.16.

THE compound is decomposed by electrolysis, one of the products liberated being trapped at one portion of the top of the mass, whilst a second liberated product is made to pass off at another portion, by directing all the electrolytic action transversely from the vertical zone of one of the products to the vertical zone of the second portion.—B. N.

Electrolytic apparatus. A. J. MacDougall, Assignor to National Electro Products, Ltd., Toronto, Canada. U.S. Pat. 1,324,511, 9.12.19. Appl., 16.4.19.

A BI-POLAR electrode is provided with a separate chamber behind each face, and a series of transverse ribs are formed on each face, with a series of gas openings immediately below the ribs, and communicating with the respective chambers. A series of electrolyte openings are arranged in each face, immediately above each rib, communicating with the respective chambers, and a sheath of porous non-conducting material is fitted over the electrode so as to embrace the opposite faces.—B. N.

Electrolytic apparatus. L. W. Chubb, Edgewood Park, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,324,797, 16.12.19. Appl., 27.4.17.

IN electrolytic apparatus comprising a number of cells adapted to give off combustible gas during their operation, a manifold conduit communicates with the interior of each of the cells, and means are provided for burning the gases which collect in the conduit by means of air supplied thereto.—B. N.

Electric arc furnaces. W. E. Moore, Pittsburgh, Pa., U.S.A. Eng. Pat. 136,034, 13.2.19. (Appl. 3555/19.)

SEE U.S. Pat. 1,309,045 of 1919; this J., 1919, 644 A.

Electric furnace control apparatus. H. A. Winne, Schenectady, N.Y., U.S.A. Eng. Pat. 136,500, 18.7.19. (Appl. 17,988/19.) Int. Conv., 2.1.19.

SEE U.S. Pat. 1,310,109 of 1919; this J., 1919, 644 A.

Precipitating matter from gases. U.S. Pat. 1,325,124. See I.

Electrical treatment of gas. U.S. Pat. 1,325,136. See I.

Quinone and quinol. U.S. Pat. 1,322,580. See III.

Electric furnaces. Eng. Pat. 135,905. See X.

Camphor. U.S. Pat. 1,324,140. See XX.

XII.—FATS; OILS; WAXES.

Olive oils; Refining of —. P. Artmann. Oel- u. Fettind, 1919, 1, 196—199. Chem. Zentr., 1919, 90, IV., 627.

DALMATIAN olive oils with a greenish tint require special treatment in refining. Decolorising is accompanied by small physical and chemical changes. Three kinds of colouring matters are present in olive oil: yellow, green (chlorophyll), and brown. The yellow and brown matters tend to be more completely adsorbed by certain agents than the green, so that by prolonged treatment or by the use of excess of decolorising agent, the oil may have a more pronounced greenish shade than before. This was particularly the case when "oleicrystal," fuller's earth, or kieselguhr was used. In the case of "eponite" the selective adsorption was less pronounced for the yellow colouring matter, so that the yellowish tint tended to persist. Quite apart from the influence of mere filtration, "eponite" had a distinct effect in removing acidity. Together with the oxidation, there is a partial loss of saturated glycerides in consequence of the lower adsorption of the unsaturated glycerides, which loss compensates or even exceeds the influence of the oxidation if the time of the refining process be prolonged or the quantity of decolorising agent or the temperature be increased. "Eponite" did not unfavourably affect the flavour, it improved the tint

of the oil, and appears preferable to other decolorising agents for the treatment of olive oil.—J. F. B.

Whale oil; Hydrogenated — P. Battenberg and J. Angerhausen. Z. Unters. Nahr. Genussm., 1919, 38, 199–206.

WHILST the usual analytical values of hydrogenated whale oils do not differ greatly from those of solid animal edible fats, the quantity of cholesterol and of unsaponifiable matters present affords means, to a certain extent, of differentiating the fats. Hydrogenated whale oil yields about 0.9% of unsaponifiable matter and 0.01% of cholesterol, the quantities for animal fats being 0.25% and 0.1% respectively. The unsaponifiable matter of whale oil, after removal of the cholesterol, is distinctly optically active ($[\alpha]_D^{20} = +2^\circ$), whilst that of animal fats is optically inactive. When crystallised from acetone, whale oils yield a larger quantity of insoluble glycerides than do animal fats, and the glycerides remaining soluble in this solvent have a high iodine value. Hydrogenated whale oils give a positive reaction with the test described by Tortelli and Jaffe (this J., 1915, 1102).—W. P. S.

Formation of fat in yeasts. Lindner and Unger. See XVIII.

Hydrogenated whale oil. Thoms and Müller. See XIXa.

Tomato and grape seeds. Shrader. See XIXa.

Fatty acids. Windisch and Dietrich. See XXIII.

Viscometer. Baume and Vigneron. See XXIII.

PATENTS.

Soaps; Process for the manufacture of economical — R. Gans, Berlin, Grünwald. Ger. Pat. 313,526, 25.6.18.

SOAP is mixed with hydrated silicates capable of exchanging their bases, which comprise iron oxides or other bases in addition to aluminium oxide. The silicates are added to the soap in the form of an extremely fine powder or as gels or sols or are produced in such forms in the soap solution or during the saponification of the fat in a state of intimate mixture. These hydrated silicates liberate a portion of their alkali on wetting with water in the same manner as soaps and have a detergent action. The proportion of such silicates may be varied within wide limits and may amount to 82% or more. The economy in the consumption of soap thus effected is estimated at 30–60%, according to the hardness of the water and the quantity of dirt or grease to be removed.—J. F. B.

Emulsions; Method of making — J. A. De Cew, Montreal, Canada. U.S. Pat. 1,317,617, 30.9.19. Appl., 25.2.18.

OIL is subdivided into fine particles by means of pressure within an atomising device and at the same time is brought into intimate contact with a warm alkaline solution, in which it is violently agitated. The emulsion thus produced is immediately discharged into an aqueous solution at a lower temperature, whereby a stable emulsion is formed.

—J. F. B.

Glycerin from liquids containing it; Process for obtaining pure — Farbentabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. (A) 303,805, 17.6.17, and (B) 305,174, 6.7.17.

(A) THE liquid is treated with lead compounds, such as litharge or lead hydroxide in order to precipitate an insoluble lead-glycerin compound. This is separated and decomposed by any suitable means, e.g., with hydrogen sulphide, sulphuric acid, or carbon dioxide. (B) The lead-glycerin compound

may be decomposed by heating with water under pressure. A solution containing 16% of glycerin is thus obtained; the lead oxide is precipitated in a heavy form and can be easily removed to serve for the precipitation of a further quantity of glycerin from crude glycerin or spent-wash.—J. F. B.

Glycerin; Process for obtaining pure — from glycerin-containing liquids. Farbentabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 305,175, 25.9.17. Addition to Ger. Pat. 303,805 (see preceding abstract).

LIQUIDS containing glycerin are treated with lead compounds in the presence of a small quantity of alkali either with or without the aid of heat.

—J. H. J.

Residues containing carbohydrates. Eng. Pat. 122,405. See XVIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Pigments for galvanised iron primers. H. A. Gardner. Paint Manufacturers' Assoc., U.S.A. Circ. No. 80. Dec., 1919. 2 pp.

EXPERIMENTS have shown that basic pigments in the presence of moisture rapidly etch the smooth surface of galvanised iron. This would indicate that much of the peeling trouble caused by non-adherence of paints to galvanised iron could be overcome by application of a priming paint containing substantial amounts of basic pigments, e.g., red lead and zinc oxide, ground in an oleaginous liquid containing sufficient water to render reactive the basic properties of such pigments.—A. de W.

[Drying] oils; Thickening of — F. Fritz. Chem. Umschau, 1919, 26, 199–200.

ON dissolving magnesium linolate in linseed oil at a temperature just sufficient to overcome foaming due to contained moisture, the solution remains liquid on cooling. If heated for a long time at 200°–250° C., or if the magnesium soap be produced by dissolving the metal in the oil at a necessarily high temperature, solidification takes place on cooling. Thus, for solidification to take place by the aid of magnesium soap, the polymerisation factor is also necessary. The increased coherence and accelerated solidification imparted to oxidised oil by previous solution of magnesium soap in the oil is advantageous to the properties of the end product used in the linoleum industry. For the purpose of increasing the thickness of heat-treated oils, the soap may be added either previous or subsequent to the heat treatment. The thickening properties given to linseed oil by magnesium soaps may be applied to the explanation of the "feeding" or "livering" of paints consisting of strongly heated oils and pigments containing basic constituents.—A. de W.

Gum shellac. C. H. Jones. Chem. and Met. Eng., 1919, 24, 715–721.

THE origin and native Indian method of preparation of shellac are described in some detail. In recent years machine methods have been introduced by one firm. The stick lac and grain lac are fed into mechanical grinders, and steam is used in the washing and melting processes, with the result that the time of production is greatly reduced and a more uniform shellac is obtained. In the native product the insoluble matter ranges from 14% in the finest grades to 5% in the lower grades, whilst in the machine-made product the insoluble matter never exceeds 0.5%, and may be as low as 0.2%. The qualities of orange shellac are determined princi-

pally by colour, and to a less extent by freedom from dirt, insoluble matter, and rosin. Average analyses of high-grade native shellacs gave the following results: Lac resin, 93–94; lac wax, 3–4.5; moisture, 1.5–2; and insol. matter (about two-thirds organic matter), 1–2%. Low-grade native shellacs contained: Lac resin, 91–93; lac wax, 3.5–4.5; moisture, 1.75–2.25; and insol. matter (about two-thirds organic matter), 2.5–4 or 5%. Average analyses of machine-made orange shellac gave: Lac resin, 95–96; lac wax, 3–4; moisture, 1.5–1.6; and insol. matter, 0.2–0.3%. Garnet lac contained: Lac resin, 97.7; lac wax, 0.5; moisture, 1.5–1.6; and insol. matter, 0.2–0.3%. A commercial sample of the best grade (D.C.) of shellac when treated with caustic alkali solution left a residual wax containing 82.18% C. and 14.25% H. This contained: Myricyl alcohol, 45; uncondensable acids (not oxy-acids) and impurities, 8.0; soluble and crystallisable oxy-acids, 27.0; and oily oxy-acids, only slightly soluble, 60.5%. Dry shellac for use in coating has been purchased for a long time by the U.S. Navy on the following general specifications:—*Orange gum shellac. Grade A quality.* (1) *For pattern and varnish work:* Must be equal in quality to the commercial brand "Double Triangle G," and capable of separation by hand. When treated with hot 95% alcohol the residue must not exceed 13%, and the shellac must be free from rosin and other adulterants. *Grade B quality.* (2) *For coating linoleum covered decks:* Must be of good quality and capable of separation by hand without the use of tools. The residue on treatment with hot 95% alcohol must not exceed 3%. It must be one of the following qualities: (1) Free from rosin and have an iodine value of less than 18 as determined by the method of the sub-committee on shellac analysis (this J., 1907, 1020); or (2) the grade known commercially as "U.S.A. A Standard TN" containing not more than 3% rosin. The grade known as "New York Standard 3%" will not be accepted. *Grade C quality.* (3) *Garnet lac for coating linoleum covered decks:* Must be of good quality and sufficiently finely ground to be readily soluble in alcohol. The residue from hot 95% alcohol must not exceed 1%. Must be free from rosin and have an iodine value of less than 18, as determined by the above-mentioned method. Somewhat different specifications are required when the shellac is to be subjected to electrical stress, or used for coating the interior of shells containing explosives. Certain samples which had been passed as suitable for the latter purpose were found to be quite unsuitable, as they showed a distinct acid test within 1 min., adhered badly to steel, and contained various metals, e.g., manganese, zinc, iron, aluminium, and calcium, in their ash (0.3 to 0.6%). They consisted of a poor grade of shellac, heavily loaded with rosin. Tests were made with various samples by coating the interior of experimental shells with a layer of the lacquer, charging the shells with nine typical explosives (including TNT, nitro-starch, picric acid, and ammonium nitrate) and leaving them for a week in an oven at 50° C. In each case with a good sample of shellac the film remained glossy and in good condition. The same results were obtained in experiments in which the shells were charged while hot. The following specifications have been worked out for pure shellac varnish: The varnish must be quite free from alkali, mineral acids, or volatile organic acids. The test is applied by shaking continuously for 10 mins. 20 grms. of the sample with 50 c.c. of neutral distilled water: if no change occurs in 3 mins. the amount of acid or alkali is negligible. The varnish must be free from heavy metallic oxides except traces of iron oxide, lime, or silica, and must contain no nitrocellulose or other nitrated material. It must adhere firmly, and when dry show a heavy, firm, tough, glossy coating, which will not crack,

peel, or flake. The varnish shall be mixed as follows: Shellac, 4lb. 1oz.; alcohol, 1 gall.; and turpentine, 1 pint. Shellac or garnet lac used in the above formula shall be finely ground so as to dissolve readily in alcohol. It shall not yield more than 1.75% of insoluble residue when treated with hot 95% alcohol. It must not contain rosin, and its iodine value must not exceed 18. The moisture must not exceed 1.75%, and the ash 1.5%. The alcohol in the formula must be either 188 proof completely denatured, or 190 proof specially denatured. The turpentine may be either a distillate from pine oleo-resins, or wood turpentine extracted from resinous woods. It must be colourless, have sp. gr. within the limits of 0.860–0.875 at 15.5° C., and initial b. pt. 150°–160° C. 90% must distil below 170° C., the polymerisation residue shall not exceed 2%, and its refractive index shall not be less than 1.500 at 15.5° C.—C. A. M.

Oil and gas storage tanks. Gardner. See I.

Aluminium corrosion. Gardner. See X.

PATENTS.

Condensation products; Production of resinous — from phenols and formaldehyde. Chem. Fabr. K. Albert and L. Berend, Amöneburg. Ger. Pat. 301,374, 26.3.13.

FORMALDEHYDE, or compounds yielding formaldehyde, are treated with *m*-cresol or its higher homologues, either in presence or absence of condensing agents. *m*-Cresol has a reaction velocity equal to five times that of *o*- and *p*-cresol. The products are characterised by their bright colour, hardness, and freedom from smell. Both soluble and fusible, as well as insoluble and infusible resins are obtainable; the soluble variety most nearly resembles natural resins and is suitable for the manufacture of varnishes. The insoluble products are neutral and are therefore specially adapted for electro-technical purposes, and they are superior to condensation products hitherto prepared by means of acids and alkalis.—W. J. W.

Varnishes for printing ink for copper or zinc plates, lithographic stones or books. A. Kronstein, Karlsruhe. Ger. Pat. 302,544, 8.2.16.

Fish oil is subjected to a vacuum distillation, the amount which is distilled being governed by the special requirements. Thus for a thin varnish for copper plates 10% is sufficient, but for thicker varnishes the amount is 45%. A mixture of $\frac{1}{4}$ of thick varnish with $\frac{3}{4}$ of the thin quality gives good results. For lithographic and zinc printing 35–40% is distilled. The varnishes dry rapidly even without the use of siccatives.—W. J. W.

Nitrocellulose; Precipitation of — from organic solvents. Vereinigte Cöln-Rottweiler Pulverfabriken, Berlin. Ger. Pats. (A) 314,317, 6.6.18, and (B) 314,318, 21.8.18.

(A) Solutions of nitrocellulose in acetone or other solvent are diluted with water, after which an electrolyte, e.g., alum, is added, and the solutions are then intimately mixed with organic compounds which are insoluble in water and have a slight gelatinising action on nitrocellulose. Suitable compounds are toluol, xylol, aniline, methylaniline, dimethylaniline, carbon tetrachloride, pine needle oil, chloroform, and carbon bisulphide. The nitrocellulose is precipitated as a dense mass which can be readily mixed with the organic compounds, the resultant product being employed for the manufacture of varnishes or lacquers. (B) The boiling solvent is diluted with water, an electrolyte is then added, and heating is continued until the nitrocellulose settles out in the form of grains, which can easily be separated from the supernatant liquid. The yield may exceed 90% of the dissolved nitrocellulose. The

exploding point of the precipitated material is 180° C. or above.—W. J. W.

Pigment zinc oxide; Preparing —. American Zinc, Lead, and Smelting Co., Boston, Mass., Assignor to L. E. Wemple, St. Louis, Mo., U.S.A. Eng. Pat. 122,170, 5.12.18. (Appl. 20,208/18.) Int. Conv., 9.1.18.

SEE U.S. Pat. 1,292,976 of 1919; this J., 1919, 330 A.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Sulphite-cellulose waste liquors. Philippsthal. See V.

PATENTS.

Rubber [vulcanised] and method of obtaining the same. L. E. Barton, Niagara Falls, N.Y., Assignor to Titanium Alloy Manufacturing Co., New York. U.S. Pat. 1,322,518, 25.11.19. Appl., 27.2.17.

RUBBER is incorporated with sulphur and titanite oxide, and the mixture is vulcanised.—E. W. L.

Rubber article; Flexible, heat-resistant —. H. E. Smith, Cleveland Heights, Ohio. U.S. Pat. 1,322,731, 25.11.19. Appl., 7.3.18.

AS INFLATABLE, expansible body for use in the heat-curing of sulphurised rubber tyres, is composed of rubber containing a substance of the sulphur group having an atomic weight higher than that of sulphur.—E. W. L.

Rubber or like substances; Process of synthetically producing —. L. Gottschalk, Rahway, N.J. E. Gottschalk, administratrix. U.S. Pat. 1,323,589, 2.12.19. Appl., 18.8.15.

LIMONENE is converted into a rubber-like substance by bringing it into contact with metallic sodium. —E. W. L.

Caoutchouc substances; Preparing — and vulcanisation product therefrom. C. W. Bedford, Assignor to Goodyear Tire and Rubber Co., Akron, Ohio. U.S. Pat. 1,323,951, 2.12.19. Appl., 29.5.17.

A MIXTURE of rubber and a product of hydrolytic decomposition of a protein is vulcanised by heating with a vulcanising agent.—E. W. L.

XV.—LEATHER; BONE; HORN; GLUE.

Tanning liquors; Direct conversion of — into pulverulent solid extracts containing very little water. Smaic and Wladika. Gerber, 1919, 45, 189—191, 204—205.

TANNING liquors suffer decomposition when submitted to prolonged high temperatures even *in vacuo*. It is necessary to reduce the temperature and time of concentration to a minimum, and even then there is some alteration in the tannins themselves shown by the dark colour of the liquors and increased amount of insoluble matter. These difficulties are avoided in a process recently introduced for drying albumin, milk, gelatin, and other materials easily decomposed by heat. By means of compressed air the liquid to be concentrated is forced through fine orifices as a spray into a closed chamber from which the water vapour is continuously exhausted. The dried product falls on to the floor of the chamber and is automatically removed. A clarified oakwood extract containing tans 9.12%, non-tans 6.99%, water 83.45%, insoluble matter 0.44%, and ash 0.66%, when thus treated yielded a dry granular extract containing tans 50.51%, non-tans 39.91%, water 7.58%, insoluble

matter 1.97%, and ash 3.30%. Calculated according to the same percentage water basis, the liquor and extract are practically identical in composition. The extract dissolves very readily to a liquor of the same colour as the original, and the liquor does not give any sediment on standing. It is usually necessary to dry the solid extracts further.—D. W.

Tannin analysis; Modified method of —. Baldracco. J. Soc. Leather Trades Chem., 1919, 3, 197—198.

THE tannin solution is prepared according to the rules of the S.L.T.C. 30–35 c.c. of this solution is passed through a filter bell containing 8–10 grms. of lightly chromed hide powder. The wet powder is blown into a shaking bottle and shaken for 15 mins. with 70 c.c. of the tannin solution. The detanned solution is filtered first through cloth and then through folded filter paper without the customary addition of kaolin. 50 c.c. of the filtrate is evaporated as usual to determine non-tans. This method requires less time than the official method of analysis and gives identical results.—D. W.

Tannin analysis; Baldracco's modified method of —. J. G. Parker. J. Soc. Leather Trades Chem., 1919, 3, 199—200.

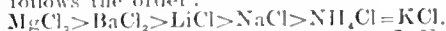
A NUMBER of tannin extracts have been analysed according to the S.L.T.C. shake method and also by Baldracco's modified method (see preceding abstract). A comparison of the results shows differences in the tannin determinations of 2%. The agreement is not so good as Baldracco claimed, and the new method does not appear to be more simple nor does it save time. It combines all the disadvantages of the present shake method and some of those of the old filter bell method.—D. W.

Alkali sulphide; Determination of — in dilute solution [lime liquors]. H. G. and W. G. Bennett. J. Soc. Leather Trades Chem., 1919, 3, 190—193.

A MEASURED quantity of the solution containing not more than 0.04% of hydrogen sulphide is distilled with an excess of boiling magnesium chloride solution in an atmosphere of carbon dioxide. A current of this gas carries the liberated hydrogen sulphide into a receiver containing 25 c.c. of N/10 iodine solution, and a second receiver containing 25 c.c. of N/10 sodium arsenite solution is arranged to trap any iodine vapour carried over. Where lime liquors are under investigation 2 c.c. of glacial acetic acid should previously be added to the iodine solution in the receiver to neutralise the ammonia which distils over. With these precautions and the absence of any rubber connections exposed to the iodine vapour, the method is very accurate.—D. W.

Hydrogen ion concentration of acid solutions [chrome tanning liquors]; Contrasting effects of chlorides and sulphates on the —. A. W. Thomas and M. E. Baldwin. J. Amer. Chem. Soc., 1919, 41, 1981—1990.

THE addition of sodium chloride or ammonium chloride to chrome tanning liquor increases the hydrogen ion concentration, whilst the addition of magnesium sulphate, sodium sulphate, or ammonium sulphate reduces it. Solutions of chromium sulphate, sulphuric acid (0.0005N and 0.1N), and hydrochloric acid (0.001N and 0.1N) behave similarly with both chlorides and sulphates. The power of increasing the hydrogen ion concentration follows the order:



—J. F. S.

Glue; Manufacture of — in the tropics from tannery refuse. K. C. Srinivasan. Dept. Industries, Madras, 1919. 28 pages.

WET fleshings were delimed, washed, and dried in

the sun, soaked for about an hour, transferred to new weak lime liquor, and delimed the next day. The delimed stock was placed in a loosely woven jute bag or rattan basket, and suspended in water in a copper pan over an open fire which was maintained at a low, steady heat. The fleshings were quickly exhausted, and in about an hour were reduced to small bulk. The thin liquor showed excellent adhesive properties. It was filtered and concentrated in the copper pan, being vigorously stirred and just kept at a gentle simmer. When a strength of above 45° Bark. had been attained at a temperature of 50° C., the liquor was poured into shallow galvanised-iron trays lightly coated with linseed oil or a mixture of linseed oil and kerosene. The solution was removed next morning to a refrigerator working at about -7° C. For factory purposes a strongly made hollander is necessary to tear up the soaked fleshings into shreds, a mechanical cleaner to free them from lime and dirt, direct steam digesters, clarifying vats with linen filters, and a vacuum evaporator. The jelly is dried in a roomy building fitted with racks. A powerful fan drives the air through a screen of steam-pipes, thus maintaining a temperature of 31.5° C., and the time required for drying varies from ten days to two months. Experiments showed that undried fleshings were unsuitable for the manufacture of glue, and that there was a deterioration in the yield and quality the longer the fleshings had been stored. The raw material was found to be highly adulterated, and required purification from all foreign matter before being digested. The yield of dry glue from 1000 lb. of raw material varied from 125 to 170 lb. Liming has no good influence on the quality or quantity of the glue, although limed fleshings are more quickly digested than unlimed ones. The total number of hours of soaking necessary is approximately $H = 48 - 4(T_1 - 28^\circ \text{C.}) + 2(28^\circ \text{C.} - T_2)$ where T_1 is the maximum and T_2 the minimum temperature during the day. Particulars are given of the machinery required and the working costs.—D. W.

Casein; Proximate analysis of commercial —. F. L. Browne. J. Ind. Eng. Chem., 1919, 11, 1019—1024.

To ascertain the suitability of commercial casein for making waterproof glues, it is suggested that determinations be made of the fineness, moisture, ash, fat, nitrogen, and acidity; the colour and odour of the sample are also taken into account. Analyses of some 200 samples are recorded. The moisture is determined by drying *in vacuo* at 70°—80° C. To determine acidity, 1 gm. of the sample is dissolved in 25 c.c. of N/10 sodium hydroxide, the solution then diluted with 100 c.c. of water, and titrated with N/10 acid, using phenolphthalein as indicator. The acidity is expressed as c.c. of N/10 alkali used by 1 gm. of dry, fat-free and ash-free substance. U.S. specifications require that all casein used for making waterproof glue for use in aeroplanes should pass the following tests:—Colour, white or light cream; odour, very slight; moisture, not more than 10%; fat, not more than 1%; ash, not more than 4%; nitrogen, not less than 14.25%; acidity, not more than 10.5 c.c. of N/10 alkali solution per gm.—W. P. S.

Red pea gall. Nierenstein. See IV.

PATENTS.

Tanning process. J. M. Brown, Austin, Ark. U.S. Pat. 1,323,956, 2.12.19. Appl., 4.4.19.

In a quantity of water sufficient to cover a hide, cutch is dissolved in the proportion of 1 lb. to 10 lb. of hide. The hide is dipped into the solution at successive intervals, a quantity of white vitriol

(zinc sulphate) equal to one-half the weight of the cutch is added, and the hide is returned to the solution until completely tanned.

Iron-tanned leather; Manufacture of tough and durable —. K. W. Mensing, Freiberg. Ger. Pat. 314,487, 28.11.15.

TANNING with iron salts is carried out in presence of an oxidising agent in excess. During preparation of the iron solution the temperature must not exceed 35° C., and the solution should be rapidly cooled to normal temperature. Treatment with the solution should then proceed at once. The delimed hide may be treated with faintly alkaline salts or with basic aluminium and chromium oxides, and then tanned with slightly acid ferric salts. The deleterious effect of ferrous salts in the tanning bath, and their ultimate appearance in the leather, are thus avoided, and in addition the hydrolytic decomposition of the ferric salts is prevented.—W. J. W.

XVI.—SOILS; FERTILISERS.

Soils; Carbonation of burnt lime in —. W. H. MacIntire. Soil Sci., 1919, 7, 325—453.

NEITHER calcium oxide nor hydroxide when perfectly dry reacts with dry carbon dioxide, but a very slight film of moisture on the containing vessel is sufficient to start the action. The oxide, when exposed in small amounts to a humid atmosphere, undergoes carbonation more rapidly than does the hydroxide prepared from it or from which it is prepared by ignition, under similar conditions of exposure. The formation of carbonate from the hydrated oxide is controlled by the amount of free water available to convert the hydrate from solid to solution phase. The carbonation of quicklime must be preceded by its hydration and solution, but when moist carbon dioxide is present the reactions are so rapid that they may be considered as simultaneous. There is a tendency for a protecting film of calcium carbonate to form round nuclei of the hydrate and thus to check further carbonation during exposure to air. Pot and fields trials indicate that lime is carbonated more rapidly when distributed on the surface of the soil than when in a dry soil mulch or in a moist soil. A sand mulch above the soil tends to retard carbonation. After the application of 2 tons of burnt lime or of hydrated lime per acre to the top six inches of a soil maximum carbonation was reached in six days and complete absorption of the lime by the soil in ten days. The addition of water hastened both the carbonation and the absorption. The time required for maximum carbonation was not affected by the presence of 48 tons of dry farmyard manure per acre. Where, however, heavier dressings (8 tons per acre) of lime were applied maximum carbonation (up to 96% of theory) of the unabsorbed lime occurred after a period of 19—38 days, the time required being unaltered by the presence of dry manure. Whilst with small or medium dressings of lime the carbonation was rapid, with large dressings, such as 32 or 100 tons per acre, carbonation proceeded more slowly, reaching about 80% of theory for the 32-ton dressing and about 50% of theory for the 100-ton dressing by the end of the second year, after which little definite change was recorded. The losses, by leaching, of calcium salts from a soil to which chalk was applied were practically independent of the extent of the treatment, but where quicklime was applied they increased with the weight of lime applied. With a soil *in situ* the presence of the subsoil very considerably checked this loss by leaching. In a comparison of a clay loam and a silt loam, it is shown that, for the former type of soil, the addition of water to

the soil caused a more rapid and greater ultimate carbonation of lime than was attained in the air-dry mixture both in the case of surface applications and when the lime was mixed with the soil. The reverse holds, however, for the silt loam. Experiments in which hydrated lime was applied to the surface of a soil-mulch or in special containers, having open ends, in the soil, the soil being in cylinders with either solid or perforated bottoms, indicate that, whilst the carbon dioxide in the soil atmosphere plays some part in the carbonation, the main action is due to atmospheric carbon dioxide. The principal action of the soil atmosphere is due to its moisture. This was confirmed by experiments in which trap layers of hydrated lime were placed between the moist soil surface and the overlying surface charges of calcium hydroxide. More extensive carbonation occurred in open sandy types of soil than in the heavier types, although the latter were richer in organic matter. This is probably due to less absorption of the lime in the open sandy soils. The practical conclusions drawn from this detailed study of the carbonation of lime are as follows:—Quicklime or hydrated lime, when applied at the rate of 2–4 tons per acre will revert to the carbonate more rapidly when left on the soil surface than when mixed with a dry mulch or the moist soil, the reversion being most rapid during humid atmospheric conditions. If a dressing of the oxide or hydrate is left on the surface of the soil for several days prior to incorporation, the treatment is in effect equivalent to an application of finely divided chalk. If the oxide or hydrate is incorporated with the top layer of soil, prior to a more thorough dissemination throughout the soil, the concentration may be sufficient to effect a temporary, partial sterilisation in that layer, which may have beneficial results. The first action of the quicklime applied to the soil is to fix atmospheric or soil moisture and there is no indication, when this is completed, of any tendency to cause chemical disintegration of soil organic matter. Neither burnt nor hydrated lime, when applied in the amounts commonly used in practice, can be considered as chemically destructive of soil organic matter.—W. G.

Soil acidity, the resultant of chemical phenomena.
H. A. Noyes. *J. Ind. Eng. Chem.*, 1919, 11, 1040–1049.

THE author holds that chemical reactions are the cause of the acidity of soils and of the changes brought about by the addition of substances such as carbon dioxide, calcium carbonate, phosphates, and nitrogenous fertilisers. The reaction of a soil at any time depends on the nature of and the proportions in which the constituents are present with water. Change in the water content, removal of substances from solution, and the addition of other substances alter the reaction in accordance with the law of mass action.—W. P. S.

Potassium in soils; Use of nickel crucibles for the Lawrence Smith fusion in determining —.
S. S. Walker. *J. Ind. Eng. Chem.*, 1919, 11, 1139–1140.

NICKEL crucibles may be substituted for platinum crucibles in carrying out the fusion of the soil with ammonium chloride and calcium carbonate; the same results are obtained but, as the nickel is attacked by the fusion mixture, the crucibles do not last long.—W. P. S.

Humic [peaty] soil; Hysteresis of aqueous solutions of —. H. Puchner. *Kolloid-Zeits.*, 1919, 25, 196–208.

A CLEAR dark yellow solution is obtained by treat-

ing peat with cold water, and at the same time a faintly acid aromatic odour is noted. The solution has an acid reaction and on keeping deposits a slimy brown substance and the solution becomes alkaline. The deposit obtained from the solution obtained from 50 grms. of peat amounts to 0.490 gm. and on incinerating leaves a residue of 0.218 gm. The ignited residue contained alumina, ferric oxide, manganese oxides, lime, magnesia, and alkalis, sulphate, phosphate, and silicate. Microscopical examination of the deposit and the solution showed that on keeping changes occurred in their appearance, notably the formation of crystals. The clear solution after sedimentation contains gels of silicic acid, ferric hydroxide, and alumina.—J. F. S.

Carbon dioxide fertiliser; Use of blast-furnace waste gases as —. F. Ruedel. *Stahl u. Eisen*, 1919, 39, 1497–1506.

CARBON and water form the main constituents of plant substances, and the former is obtained by assimilation of atmospheric carbon dioxide. The partial pressure of this is normally very small—say, 0.03%—and so may prove the limiting factor at periods of rapid growth when the supply of moisture and mineral fertilisers is ample. It has been shown by Kreusler (1885) that the rate of assimilation of carbon dioxide increases extremely rapidly with the partial pressure until this reaches about 0.5%, but afterwards little effect is produced by further increase. This has been examined in horticultural trials in which plants were grown in an atmosphere enriched in carbon dioxide by the exhaust gases of large gas engines. These were driven by blast-furnace gas; the cleaning necessary to render the gas suitable for use in gas engines also removed plant poisons. In 1917 identical crops were grown in three glasshouses 25 m. × 6 m. each, two of them being supplied with exhaust gas, and the third used as a control with a normal atmosphere. Diluted exhaust gas was led in through perforated pipes placed under the roof so as to raise the partial pressure of carbon dioxide to about 0.5%. The experiment began in June, and at once the treated plants obviously grew more vigorously. The deeper shade of green in the foliage of the treated plants pointed to a more rapid production of chlorophyll. The crops were much increased as compared with those of the plants in the control house, e.g., the tomato crop was 2½ times and cucumber crop 1½ times greater. Analogous results were obtained in open-air experiments in which a plot was divided into squares by a network of perforated pipes, through which exhaust gases were discharged at ground level. A variety of vegetables showed increased crops as compared with a control crop—the ratio of increase reaching 2.9 times. In 1918 the trials were repeated with similar results. Three additional glasshouses 40 m. long were used, and the open air trials were made on a field 30,000 sq. m. in area divided up by pipes as before. It was found that the effects of the treatment are more evident when the land is otherwise well fertilised, indicating that the carbon dioxide promotes efficient utilisation of other manures. Analytical determinations on the atmosphere of a glasshouse during the treatment revealed the rapid absorption of carbon dioxide by the plants. In discussing the economic importance of carbon dioxide fertilisation, it is pointed out that a blast-furnace plant of 1000 tons daily output of iron yields carbon dioxide sufficient for 4000 tons of potatoes.—H. J. H.

Arsenious acid and growing tissues. Cobet. *See XX.*

Potash. Keitt and Shiver. *See XXXI.*

PATENT.

Fertiliser and process of making same. G. H. Earp-Thomas, Glen Ridge, N.J. U.S. Pat. 1,309,723, 15.7.19. Appl., 10.4.17.

Finely divided raw phosphate rock is mixed with a humus material containing nitro-bacteria, which exert a decomposing action on the rock.—J. H. J.

XVII.—SUGARS; STARCHES; GUMS.

Clarification [of sugar juice]; Some temperature experiments in —. H. S. Walker. Int. Sugar J., 1919, 21, 611—613.

DURING the suspension of work at the week-end in a factory in Hawaii it was observed that the limed juice in the settling tanks decreased in purity to a marked extent (1° per hour) at 190° — 212° F. (88° — 100° C.), whereas at a lower temperature, viz., about 180° F. (82° C.), the value remained practically constant. The loss of sucrose at the higher temperature is due, not to the presence of heat-resisting micro-organisms, as was at first presumed, but to the effect of heat under the conditions prevailing.—J. P. O.

Scums; Influence of the duration of contact of the — with the first saturated juice in beet sugar manufacture. V. Stanek. Z. Zuckerind. Böhm., 1918, 42, 417—423.

LABORATORY and factory experiments are described demonstrating that instead of immediately filtering off the juice after its first carbonation it is advantageous to stir it for about 30 mins. at 85° C. A marked improvement in the rate of filtration results, and the purity of the juice is raised (e.g., from 93.3 to 94.1), while no increase in calcium oxide, mineral matter, alkalinity, or colour could be observed. It is suggested that during the time of contact amorphous calcium carbonate is converted into the crystalline form, and that tricalcium saccharate present is decomposed with the liberation of sugar.—J. P. O.

Sugar dryers; Method of increasing the capacity of —. E. V. Anthonijs. Archief Suikerind. Nederl.-Indië, 1919, 27, 1125. Int. Sugar J., 1919, 21, 622.

AFTER centrifuging the sugar, and washing with water, it is treated with superheated steam at about 250° C. while still in the machines. This hot sugar may subsequently be passed much more rapidly through the dryers than in the usual mode of operating; moreover, it is unnecessary to circulate a current of heated air through the drying drum.—J. P. O.

Cane sugar; Sucrose content of the molasses as an index of the inversion occurring during the manufacture of —. F. I. Seard. Int. Sugar J., 1919, 21, 604—605.

It is characteristic of the juice produced from cane in British Guiana that its reducing sugars are optically inactive, though this is seldom so in the case of juice obtained in other countries. In order, therefore, to obtain an indication of the amount of reducing sugars resulting from the inversion of sucrose during manufacture, it is suggested that a comparison should be made between the sucrose determined by direct polarisation and that found by the reduction of Fehling's solution before and after hydrolysis. The first figure should be lower than the second in proportion to the amount of levo-rotatory reducing sugars which may have formed during clarification, evaporation, and crystallisation. Normal lead acetate must be used for the clarification of the solution for polarisation, since the basic salt precipitates levulose and dex-

trose (compare, however, Davis, this J., 1916, 203). Application of this method has shown that in the manufacture of raw sugar under good conditions in Guiana no indication of inversion is obtained; whilst in the production of yellow Demerara sugar (in which the juice is generally treated with phosphoric acid to slight acidity), it is very small, seldom more than 2% of the sucrose present being hydrolysed.—J. P. O.

Sugar grain in final cane molasses; its origin and determination. H. Kalshoven. Archief Suikerind. Nederl.-Indië, 1919, 27, 1560—1664. Int. Sugar J., 1919, 21, 608—610.

MOST cane factory molasses contains fine sucrose crystals, averaging about 8%, a quantity that represented in the case of the last Java crop about 35,000 tons of commercial sugar. Probably the principal cause of its presence is the formation of "false grain" during boiling in the vacuum pan, though some may result from centrifuging at a high temperature (say 50° C.), further crystallisation occurring on the subsequent cooling of the molasses. Some may also be produced by cooling massecuite in crystallisers in which the entire contents do not come under the influence of the stirrers, "dead corners" being formed. Its amount may be determined by ascertaining the refractive index of the molasses before and after diluting with a sufficient amount of water to dissolve the fine grain, the first reading giving the solids of the molasses alone (the presence of the crystals not affecting the result), and the second that of the molasses plus crystals.—J. P. O.

Syrups and molasses; Standards for differentiating between American table —. W. L. Owen. La. Planter, 1919, 62, No. 15, 235—236. Int. Sugar J., 1919, 21, 622.

It is difficult, if not impossible, to establish analytical standards differentiating between syrup, first molasses, second molasses, and third molasses as produced in Louisiana. Generally the sucrose in the syrup (concentrated cane juice) varies from 49 to 62; in first molasses from 38 to 54; in second molasses from 28 to 45; and in third molasses from 23 to 33%. In the case of the purity coefficient the differences for the four classes of products are 70 to 87, 43 to 73, 35 to 60, 30 to 50 respectively; while the maximum figures for ash are 2.5, 9.0, 10.6, and 12.5%.—J. P. O.

Reducing sugars; Ost's copper potassium carbonate solution as a reagent for the determination of —. P. Beyersdorfer. Z. Ver. deut. Zuckerind., 1919, 403—437.

COMPARED with Fehling's solution, the author regards Ost's solution (this J., 1895, 993) as superior in most respects for the determination of reducing sugars. It is more sensitive, being capable of detecting 1 mgrm. of invert sugar in the presence of 10 grms. of sucrose, against 5 mgrms. by Fehling's solution. Its effect upon sucrose in producing reducing substances is appreciably less, 3.8 mgrms. of copper being obtained as against 30 mgrms. Moreover, 1 equivalent of invert sugar reduces 3 of copper compared with 2 in the case of Fehling's solution. Ost's solution is prepared by dissolving 250 grms. of potassium carbonate and 100 grms. of potassium bicarbonate in about 700 c.c. of water in a litre flask, which is heated in a water-bath at 60° — 80° C. After cooling, a solution containing the desired amount of pure copper sulphate is added in small successive amounts while well agitating, the liquid being then made up to the mark, mixed, and filtered. Two solutions should be prepared—the first containing 4 grms. of copper (15.71 grms. of crystallised copper sulphate) per litre, which is used for general purposes; and the second with only

0.8 grm. of copper (3.14 grms. of copper sulphate), which serves for the examination of products, as those of the refinery, in which a small amount of invert sugar is present with a large proportion of sucrose. This weaker solution is more sensitive than one of higher copper content, and attacks sucrose less. It may be prepared from the stronger liquor by mixing one volume of the latter with 4 of a filtered solution containing 250 grms. of potassium carbonate and 100 grms. of potassium bicarbonate per litre. For the determination of the cupric-reducing power, 75 c.c. of Ost's solution is mixed with 50 c.c. of the sugar solution in a 125 c.c. flask, and the volume adjusted to the mark. Heating is carried out in a boiling water bath for 15 and 20 minutes for the strong and the weak liquors respectively, and at the end of the period the amount of cuprous oxide precipitated is determined by Bertrand's method (this J., 1907, 60) of dissolving the precipitate in an acid solution of a ferric salt and titrating with permanganate, a procedure which is considered to be capable of yielding very reliable results (*cf.* this J., 1919, 381 A). The amount of invert sugar corresponding to the amount of copper reduced is calculated from two tables. The first is for use with the "fifth normal" liquor, when the 50 c.c. of sugar solution taken for the determination contains less than 20 mgrms. of invert sugar in the presence of 10 grms. of sucrose (for application in the case of refinery products); while the second is applied when the "normal" solution is used, the limit of invert sugar present in the 50 c.c. of sugar solution being 2 to 100 mgrms., and that of sucrose 0 to 10 grms.—J. P. O.

Sugars; Titration of —. E. Schowalter. Z. Unters. Nahr. Genussm., 1919, 38, 221—227.

THE method described by Rupp and Lehmann (this J., 1919, 691 A) is criticised and considered to be untrustworthy under the conditions prescribed.
—W. P. S.

Dextrose and levulose; Separation of —. F. Lucius. Z. Unters. Nahr. Genussm., 1919, 38, 177—185.

WHEN a 2—4% dextrose or levulose solution is heated for seven hours with the addition of hydrochloric acid, the optical rotation of the dextrose is not affected, whilst that of the levulose is destroyed completely. The levulose is converted into other reducing substances, and, consequently, the remaining dextrose cannot be determined by finding the reducing power of the solution after treatment, but polarisation gives an accurate measure of the latter sugar. The details of the determination are as follows: 50 c.c. of the sugar solution is mixed with 10 c.c. of 5N hydrochloric acid and heated for seven hours in a boiling water bath; the mixture is then nearly neutralised with 5N sodium hydroxide solution, diluted to 100 c.c., and polarised at 20° C. in a 200 mm. tube. If necessary, the solution is treated with animal charcoal before it is polarised. The method may be used for the determination of dextrose in invert sugar etc. If the heating is prolonged for 10 to 12 hrs. the rotation of the dextrose itself begins to decrease slightly.—W. P. S.

Sugars; Conversion of the simple — into their enolic and ethylene oxide forms. E. F. Armstrong and T. P. Hilditch. Chem. Soc. Trans., 1919, 115, 1410—1428.

ALTHOUGH α - and β -glucose (dextrose) are practically unaffected by permanganate in pure neutral solution, on the addition of acid or alkali, reduction sets in at once at a definite rate under definite conditions. The change with acids is instantaneous, but with alkalis it is more gradual as the reducing power increases within limits the longer the alkali has acted. The same facts are true as regards fructose (levulose) and mannose, though the rates of reduc-

tion differ, being greater than that of glucose in acid solution; in alkaline solution the order is fructose, glucose, mannose. It is considered that the active agent in acid solution is the ethylene oxide modification of the sugar, whilst in alkaline solution it is most probably the enol which is gradually produced. Parallel reducing actions are observed with methylene blue and indigo, and it is noteworthy that in similar experiments with simple aldehydes, ethylene oxide, and epichlorhydrin, the ethylene oxides behave similarly to the sugars in acid solutions, and the aldehydes in alkaline solutions both towards methylene blue and permanganate.—G. F. M.

Effect of oil in exhaust steam on efficiency of evaporators. Claassen. See I.

Decolorising carbon. Coates. See IIb.

Starch in paper. Kamm and Tendick. See V.

Carbohydrates of vegetables. Falk. See XIXa.

PATENT.

Residues containing carbohydrates. Eng. Pat. 122,105. See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Moisture in malt; Determination of —. T. J. Ward. J. Inst. Brew., 1919, 25, 335—352.

IS the Institute of Brewing method for determining moisture in malt (this J., 1906, 236) the conditions are not defined with sufficient completeness to ensure concordant results. The method recently adopted for moisture determinations in barley (this J., 1918, 523 A) is much more satisfactory in this respect, and may be applied to malt in the following modified form:—15—20 grms. of the well-mixed sample is finely ground in a coffee-mill; the first third of the grist is rejected, and from the remainder 5 grms. is immediately weighed out into a counterpoised flat-bottomed dish provided with a well-fitting lid, and placed on the floor of a boiling-water oven, with the lid beside it or on the shelf. After drying for 3—3½ hours, the dish is stoppered, transferred to a desiccator, and weighed when quite cold. The author describes an exhaustive investigation of the influence of various factors on the results, the experiments being made with the usual type of cubical water-jacketed oven having a shutter ventilator in the lower part of the door and an open ventilator at the top. The chief conclusions are as follows:—Accurate results may be obtained with ovens of any capacity between 3½ and 33 litres. The amount of water in the jacket is immaterial, provided its level remains higher than the interior floor of the oven, and it is kept boiling briskly throughout the drying process. Unsuspected leaks through which water or steam may escape from the jacket into the oven may cause consistently low results. Such leaks are detected by allowing the oven to stand overnight with the jacket full of water, and afterwards examining the interior for drops of water. Both ventilators must remain open during determinations; the total area of the openings in the lower ventilator, in sq. cm., may vary from 1/850 to 1/19000 of the capacity of the oven, in c.c., without affecting the results. There is no objection to the presence of a shelf in the oven, provided it is perforated with at least four holes of diam. about 1 cm.; but as the temperature on the shelf is always considerably below 100° C. (*e.g.*, 90° when the lower ventilator is open and 94° C. when it is closed), the dishes containing malt must always be placed on the floor, and not within a radius of 5 cm. from the ventilator. The dishes

used may be of glass, porcelain, aluminium, tin-plate, etc., but should be flat-bottomed, with vertical sides, and with well-fitting lids, so that a closed dish containing dried malt may be left exposed to the open air of a laboratory for an hour without gaining more than 1 mgrm. in weight. Ordinary shallow tins with well-fitting lids have proved very satisfactory. The average depth of malt in the dishes should not exceed 5 mm. The same results are obtained whether the malt is finely or coarsely ground. A delay of 5 mins., however, between grinding and weighing out may cause a perceptible error (e.g., 0.1%) owing to absorption of moisture. No error is caused by weighing out incorrect proportions of the husk and endosperm fractions of the grist, for these do not differ much in moisture-content and affinity for moisture. Any number of samples may be dried on the floor of the oven at the same time, but no other material should be present, and the oven door should not be opened, especially after the first hour. The same results are obtained at 100° C. whether drying is continued for three hours or longer—e.g., 18 hours; but at 105° constant weight cannot be attained within a reasonable time. The relative humidity of the atmosphere, unless above 96%, does not affect the results; but the entry of moist fumes from the gas-burner into the ventilator must be prevented—e.g., by means of a guard-plate extending downwards from the bottom of the front of the oven. By observing the precautions indicated above, the experimental error in determining the moisture in malt may be reduced to $\pm 0.02\%$.—J. H. L.

Malts; Some analyses of barleys and of the —
made from them. A. R. Ling. J. Inst. Brew.,
1919, 25, 288-296.

ANALYSES (see table) were carried out on various barleys and malts prepared therefrom in 1912-13.

a fair sample, but contained an abnormal proportion of damaged and extraneous corns. The malts from all the foreign barleys showed uneven germination, particularly that from the Polish barley, which was probably a mixture derived from more than one harvest. For the determination of starch in the samples Ewers' method (this J., 1908, 238) was employed, and the results are therefore probably high, owing to the presence of pentosans and other substances which yield sugars on hydrolysis with acid.—J. H. L.

Acidity of worts, beers, and other similar liquids; New methods for determining the —.
W. Windisch and W. Dietrich. Wech. Bran.,
1919, 36, 189-190, 201-203, 209-211.

SOME preliminary experiments were made to explore the practicability of determining the acidity of worts by Traube and Somogyi's method (this J. 1915, 511)—viz., by measuring the surface tension of the wort relative to that of water with a viscostagonometer, then adding a capillary inactive salt of a capillary active fatty acid, whereby the surface tension of the wort is diminished owing to the liberation of free fatty acid equivalent to the wort acidity, and finally determining the volume of standard alkali required to restore the original surface tension—i.e., to neutralise the liberated acid. In studying the change of surface tension accompanying the progressive neutralisation of solutions of various fatty acids, it was found that with nonylic, decylic, and undecylic acids the value of the surface tension constitutes as sensitive an indicator as litmus or neutral red in the region of their colour change. In applying the method to worts, the acidity of which is largely due to primary phosphates, salts of undecylic acid appear far preferable to valerates

	Californian.		Damascus.		Oxford.		Norfolk.			Polish.		
	Barley.	Malt.*	Barley.	Malt.†	Barley.	Malt.	Barley.	Pale Malt.	Amber. Malt.	Barley.	Malt.	Malt.
Extract, lb. per 336 lb.	—	92.5	—	86.5	—	95.2	—	97.9	96.7	—	93.9	93.8
Diastatic power (Lintner)	9	20	16	30	23	16	19	27	14	—	23	28
Tint, N. Lovibond (10% solution in 1" cell)	—	6.0	—	3.5	—	9.5	—	3.5	13.5	—	4.5	4.5
Moisture, per cent.	9.68	1.10	10.94	2.08	12.63	6.55	10.88	1.30	0.48	12.60	2.3	3.2
1,000 corn wt., grms.	45.65	35.85	46.55	39.95	43.47	34.56	31.67	31.93	31.76	43.04	35.99	35.94

Results calculated on the dry samples.

Starch	57.05	50.65	54.64	50.28	61.05	55.16	61.56	56.18	55.66	58.19	51.34	49.98
Albuminoids, insol. ..	6.96	5.25	7.21	5.86	7.74	6.06	7.63	5.85	5.58	7.53	6.61	5.81
.. sol.	2.14	3.47	2.52	3.47	2.53	4.01	1.65	3.59	3.35	2.93	4.37	4.67
Ash, insol.	1.86	1.80	2.07	1.86	1.84	1.99	1.93	1.65	1.62	1.79	1.54	1.49
.. sol.	0.90	0.66	1.00	0.61	0.98	0.66	0.72	0.80	0.71	0.73	0.80	0.84
Fat	1.99	1.94	2.02	1.93	2.51	1.99	2.36	1.78	2.07	2.47	1.87	2.07
Sugar, invert	0.67	3.84	0.56	3.14	0.96	3.40	1.27	4.63	4.30	1.43	6.04	6.34
.. cane	2.31	7.18	1.90	5.53	1.09	6.40	1.01	5.36	5.68	1.27	5.12	6.72
Gums, etc.	1.52	2.59	1.72	2.83	1.48	3.23	0.95	3.35	3.36	1.84	4.06	3.42
Fibre	7.86	8.41	6.02	6.78	4.99	5.71	5.63	5.69	5.45	5.86	5.41	5.65
1,000 corn weight (dry) ..	41.23	35.46	41.46	39.11	37.98	34.37	35.46	31.51	31.61	37.62	35.17	34.79

Malt results calculated on that weight of the corresponding dry barleys required to yield 100 parts of dry malt.

Starch	57.05	43.56	54.64	47.42	61.05	49.91	61.56	49.90	49.61	58.19	47.98	46.21
Albuminoids, insol. ..	6.96	4.52	7.21	5.53	7.74	5.48	7.63	5.19	4.97	7.53	6.18	5.37
.. sol.	2.14	2.98	2.52	3.27	2.53	3.63	1.65	3.19	2.98	2.93	4.08	4.32
Ash, insol.	1.86	1.55	2.07	1.75	1.84	1.80	1.93	1.47	1.44	1.79	1.44	1.38
.. sol.	0.90	0.57	1.00	0.58	0.98	0.60	0.72	0.71	0.63	0.73	0.75	0.78
Fat	1.99	1.67	2.02	1.82	2.51	1.80	2.36	1.58	1.85	2.47	1.75	1.91
Sugar, invert	0.67	3.30	0.56	2.96	0.96	3.08	1.27	4.11	3.83	1.43	5.65	5.86
.. cane	2.31	6.17	1.90	5.22	1.09	5.79	1.01	4.76	5.06	1.27	4.79	6.21
Gums, etc.	1.52	2.23	1.72	2.67	1.48	2.92	0.95	2.97	3.00	1.84	3.80	2.16
Fibre	7.86	7.23	6.02	6.39	4.99	5.17	5.63	5.05	4.86	5.86	5.06	5.22

* Contained 5% of damaged and idle corns and 3% of extraneous corns. † Contained 4% of damaged and idle corns.

With the exception of the Californian and Damascus barleys, all the barleys were of first grade, and required no special treatment in the malt-house. The English barleys were of the 1912 harvest, and gave excellent malts, showing very uniform germination. The Californian barley was

(loc. cit.), as the latter are scarcely affected by primary phosphate acidity. The method would have the advantage of being independent of the colour of a wort, but its accuracy becomes less the higher the surface tension of the wort.

—J. H. L.

Beer; Concentration of hydrogen ions in —. F. Ensländer. Z. ges. Brauw., 1919, 42, 127—130, 135—137. (See also this J., 1914, 212.)

The optimal hydriion concentration for the fermentative action of brewers' yeast is $p_H=4.76-4.16$, whilst for other yeasts investigated by the author the optimal reactions were found to be much more alkaline. In unfermented brewery worts the hydriion concentration is about $p_H=6.5-5.5$, but owing to the formation of acids during fermentation the average value for dark beers made with waters fairly rich in carbonates is $p_H=4.5$ and that for pale beers made with gypscous waters is $p_H=4.2$. Under normal circumstances, therefore, the yeast raises the hydriion concentration of wort to approximately the value most favourable to its own fermentative activity. Any circumstance which tends to prevent or delay the attainment of this optimal hydriion concentration in the fermenting wort, e.g., the use of abnormally alkaline waters, low fermentation temperatures, or too little yeast, must accordingly favour the activities of foreign yeasts, since these thrive best in presence of relatively low hydriion concentrations.—J. H. L.

Fermentations; Production of bouquet in primary and secondary —. P. Lindner. Woch. Brau., 1919, 36, 223—224.

In wines naturally lacking in *bouquet* it appears possible to remedy this defect by the use of suitable kinds of pressed yeast in the secondary fermentation. Certain distillery yeasts, beer yeasts, and mycoderma yeasts, which produce fragrant odours during fermentation might be used. The production of *bouquet* depends more on the yeast employed than on the nutritive substances present, but it is intensified under certain conditions, e.g., when nitrogenous nutriment is lacking and growth is stimulated by high temperatures or other means. Lack of assimilable nitrogen leads to the degradation of yeast proteins, and the amino-acids formed are in part converted into higher alcohols which contribute to the production of *bouquet*. This accounts for the fragrant aroma frequently observed when brewing yeast is regenerated by growth in sugar solution, accompanied by aeration, the nitrogen-content of the yeast undergoing a considerable reduction. The removal of assimilable nitrogen by the yeast in the primary fermentation of wines, no doubt contributes to the production of *bouquet* during the secondary fermentation.—J. H. L.

Yeast as it occurs in nature. A. Jørgensen. J. Inst. Brew., 1919, 25, 353—355.

YEAST as it occurs in nature possesses the flocculating character, the cells tending to remain attached to one another in chains or aggregates. The contrary behaviour of certain yeasts employed in industry, the cells of which tend to separate and remain suspended in the surrounding liquid, thus causing difficulties in clarification, is a character acquired as the result of continued cultivation under artificial conditions. This acquired character can be entirely eliminated, whether in brewers', distillers', bakers', or wine yeasts, by "methodically cultivating the growth under the same conditions as those which it met during the greater part of the year while living in the state of nature." Further particulars will be published after the completion of more extended experiments.—J. H. L.

Yeasts; Formation of fat in — on solid media. P. Lindner and T. Unger. Z. techn. Biol., 1919, No. 1. Woch. Brau., 1919, 36, 188.

SUSPENSIONS of various brewery and distillery yeasts, spread over glass plates, were exposed to vapours of ethyl alcohol. An abnormally copious production of fat globules ensued in the yeast cells, and it is suggested that the formation of similar

globules, often observed in the superficial cells of yeast cultures on solid media, are due to the influence of alcohol vapour produced by the underlying cells. The fat is probably not a reserve material, for it is found in cells of very old cultures. Fat globules are found in largest numbers in cultures of bottom fermentation beer yeasts, whilst in cultures of top fermentation yeasts and wine yeasts the cell-contents tend rather to assume a granular appearance. The original paper contains a tabular record of the microscopical appearance of cultures of numerous yeasts in the V.L.B. collection in Berlin.—J. H. L.

Yeast; Autolysis of — in its dependence on hydrogen and hydroxyl ions. K. G. Dornby. Med. K. Vetensk. Nobelinst., 1918, 3, No. 23, 1—26.

THE simple yeast cell contains enzymes which are capable of degrading albuminous substances and are analogous in type with those present in animal organs. The enzymes identified are yeast pepsin, which degrades albuminous substances to peptones and has a maximum activity at the hydrogen ion concentration $p_H=4-4.15$; yeast trypsin, which does not attack yeast albumin, but attacks gelatin, casein, Witte-peptone, and similar substances, and has a maximum activity at $p_H=7.0$; yeast ereptase, which degrades polypeptides to amino-acids and has a maximum activity at $p_H=7.8$. The autolysis of yeast is brought about by these enzymes and consists in a successive degradation of albumin; it can only proceed when the different enzymes can operate simultaneously. The best hydrogen ion concentration for the process is $p_H=6.1$.—J. F. S.

Lactic acid bacteria; Acidity relations of —. O. Svanberg. Med. K. Vetensk. Nobelinst., 1919, 5, No. 2, 1—10.

Streptococcus lactis will raise the hydrogen ion concentration of milk and whey to $p_H=4.0$ at 16°C . *Bacterium casei* in milk and wort raises the hydrogen ion concentration to $p_H=3.05$. In the presence of sodium lactate the acidity which *Streptococcus lactis* can develop in milk is reduced proportionally to the lactate concentration. The acid tolerance of *Streptococcus lactis* towards sulphuric, hydrochloric, and phosphoric acids is the same, namely $p_H=3.4$ at 18°C ., and for *Bacterium casei* at 35°C . the value is $p_H=3.0$ for hydrochloric and phosphoric acids. The alkali tolerance for *Streptococcus lactis* at 18°C . is $p_H=8.0$.—J. F. S.

Enzyme action. Urease and the radiation theory of —. H. P. Barendrecht. Proc. K. Akad. Wetensch., 1919, 22, 126—138.

THE enzyme urease acts by radiation, which is only absorbed by its substrate, urea, and by hydrogen ions. This action is represented by the formula $nc/0.134 \log \frac{1}{(1-y)+ay} = mt$ in which a is the initial concentration of the urea, y the fraction present at time t , n the absorption coefficient of the hydrogen ions, m a constant, and c the concentration of the hydrogen ions. This explains the kinetics of urease action in both acid and alkaline solutions. In acid solutions a logarithmic curve is obtained and in alkaline solutions a straight line graph. When the urease is decaying the reaction may be reversed and urea synthesised from ammonium carbonate. (See also J. Chem. Soc., 1920, i., 195.) —J. F. S.

Alcohol from coke-oven gas. Bury. See IXa.

Catalytic decomposition of hydrogen peroxide. Phragmén. See VII.

Yeast vitamin. Osborne and Wakeman. See XIX A.

Yeast nucleic acid. Levene. See XX.

PATENTS.

Residues containing carbohydrates; Method of treatment of — J. Effront, Brussels, and A. Boidin, Paris. Eng. Pat. 122,405, 2.1.19. (Appl. 131/19.) Int. Conv., 17.1.18.

AMYLACEOUS residues from the starch, brewing, or oil industries are utilised for the preparation of products which can be employed in brewing and in the manufacture of sweets and syrups. The residues are subjected, in slightly alkaline mashies, to the action of bacterial diastatic enzymes of the kind described in Eng. Pat. 16,198/14 (see Fr. Pat. 471,546; this J., 1915, 444), which are more powerful than vegetable diastase and act best in alkaline media. According to the character of the starch conversion desired, the mash may be held at temperatures ranging from 70° to 85° C. for $\frac{1}{2}$ —1 hour, after which saccharification is arrested, the mash filtered, and the filtrate concentrated to a syrup. From the residues from starch factories syrups may be prepared which crystallise to a mass containing 70% of dextrins (not coloured by iodine) and 30% of sugars, and which have the flavour of honey.

—J. H. L.

Fermented liquor from [ramoon] nuts. R. H. McKee, Ridgefield Park, N.J. U.S. Pat. 1,322,194, 18.11.19. Appl., 20.6.17.

A FERMENTED extract of ramoon nuts.—J. H. L.

Alcohol from wood; Process of making ethyl — H. K. Moore, Berlin, N.H., Assignor to Brown Co., Portland, Me. U.S. Pat. 1,323,540, 2.12.19. Appl., 23.4.14.

Woon is heated with a hydrolysing solution containing phosphoric acid or an acid phosphate, and the resulting liquor is fermented without previous neutralisation.—J. H. L.

Glycerin. Ger. Pats. 303,805 and 305,174—5. See XII.

XIXA.—FOODS.

Milk; Carbonic acid and carbonates in cow's — L. L. Van Slyke and J. C. Baker. J. Biol. Chem., 1919, 40, 335—344.

THE carbon dioxide of milk may be estimated by the process originally proposed for the estimation of carbon dioxide in blood plasma (Van Slyke, this J., 1917, 944). Milk is drawn from the cow's udder into a 100 c.c. cylinder so as to fill the cylinder from the bottom upward, thus avoiding mixing with air or loss of carbon dioxide. 2 c.c. of this milk is transferred with precautions against loss of CO₂ into the Van Slyke CO₂ apparatus and treated with a 20% solution of lactic acid. This is found a more satisfactory method of liberating the carbon dioxide than by employing sulphuric acid, which clots the milk into large lumps. The carbon dioxide content of milk varies over a wide range; a normal value is regarded as about 10% by volume. The degree of acidity tends to decrease with an increase of carbon dioxide. The carbon dioxide may be completely removed by exhaustion from a thin film of milk which is kept in motion. After pasteurisation the carbon dioxide content of the milk falls, but the hydrogen ion concentration remains the same. The suggestion is advanced that the carbon dioxide content of milk might afford a means of distinguishing heated from normal milk. Probably the carbon dioxide of milk is present as a mixture of 1 part of carbonic acid to 2 of sodium bicarbonate.—J. C. D.

Milk; Conditions causing variation in reaction of freshly drawn — L. L. Van Slyke and J. C. Baker. J. Biol. Chem., 1919, 40, 345—355.

In over 300 samples of fresh milk the value of pH

varied from 6.50 to 7.20, being under 6.76 in 80% of the samples. In the case of 20 cows it was found that the reaction of the milk from different quarters of the udder varied greatly, but in most cases the variations were not large. In general a decrease of acidity is associated with a marked tendency towards a decrease in specific gravity, and in percentage of fat, total solids, solids not fat, caseinogen, and lactose, but an increase in proteins other than caseinogen and in ash and chlorine. These changes are such as would be expected if blood serum or lymph were added to normal fresh milk. Abnormal conditions of the udder might cause such additions. Examination of milks of low acidity, pH above 6.80, indicates that the reaction is accompanied by the presence of large numbers of leucocytes, though the reaction in such cases may be neutralised by the presence of large numbers of acid-producing streptococci.—J. C. D.

Milk; Detection of abnormal — based on hydrogen ion concentration. J. C. Baker and L. L. Van Slyke. J. Biol. Chem., 1919, 40, 357—371.

ONE drop of a saturated solution of dibromo-o-cresol-sulphon-phthalein (bromo-cresol purple) in water is added to 3 c.c. of milk and the colour observed. Normal fresh milk gives a greyish-blue colour. The production of a darker or lighter colour serves to awaken suspicion in regard to the normal character of the milk. The colour is made lighter by acids and formaldehyde, and by heating above the usual point of pasteurisation. The colour is of deeper blue in the case of milk from diseased udders, watered milk, skimmed milk, and milk containing alkaline salts. The method has been applied to 570 samples of market milk and has given trustworthy results. Colour standards can be prepared.—J. C. D.

Milk; Determination of keeping quality of — J. C. Baker and L. L. Van Slyke. J. Biol. Chem., 1919, 40, 373—382.

BROMO-CRESOL purple (see preceding abstract) can be used to measure approximately and relatively the keeping quality of milk. The test is applied as given in the preceding paper, with the modification that the pipettes and tubes employed must be sterilised before use, and that the samples under test must be incubated for a given time at 18°—20° C. The milk is examined for change of colour at 24-hour intervals. The main factor shown by this test as related to keeping quality is production of acid, but additional factors to be observed are coagulation of caseinogen, digestion of caseinogen, production of alkali and gas, and development of abnormal odour or taste. In the development of acidity four stages of progress are distinguishable through change of colour. In comparing this test with the bacterial count, it is found that in general large numbers of bacteria and increase of acidity are in fair correlation.—J. C. D.

Milk; Heat coagulation of — H. H. Sommer and E. B. Hart. J. Biol. Chem., 1919, 40, 137—151.

THE coagulation of milk by heat occurs at from 130° to 150° C., with different samples of milk. In the manufacture of condensed milk this coagulation may occur during the sterilisation of the finished product sealed in tins. Manufacturers have attempted to solve the problem by controlling the acidity of the milk, and have set an arbitrary standard, such as 0.18% acid (calculated as lactic acid), above which they reject all milk. Frequently, however, milk, immediately after it is drawn from the cow, has a higher acidity (as determined by titration) than this value. This was the case in approximately half of the samples of freshly drawn milk examined. Study of the factors which govern the heat coagulation of milk showed that in fresh milk

there is no direct relation between the acidity found by titration and coagulation. Acidity measured by titration is, however, no measure of the true reaction, which can only be estimated by a determination of the hydrogen ion concentration. Further experiments showed that the reaction of the milk, as determined by measurement of the hydrogen ion concentration, is not the determining factor in the heat coagulation. The reaction may, however, be a factor, for if the reaction is changed by addition of small amounts of acids the coagulation point is lowered. The concentration of the milk may also be a factor, but neither this nor the hydrogen ion concentration is the chief cause of the coagulation. The main factor was found to be the composition of the salts of milk. Electrolytes have a very marked effect on the stability of the colloids of the milk. Caseinogen requires a definite optimum calcium content for its maximum stability. The calcium content of caseinogen is largely governed by the amounts of magnesium, citrates, and phosphates present. In most cases coagulation can be prevented by the addition of citrates or phosphates, and it appears that the coagulation may be due either to an excess or a deficiency of calcium and magnesium.—J. C. D.

Casein; Solubility of — in dilute solutions of sodium chloride and its dependence on the hydrogen ion concentration. S. Ryd. Arkiv Kem. Min. Geol., 1917-18, 7, No. I, 1-15.

THE solubility of casein at 18°-20° C. increases from 6.4 mgrms. per c.c. in 0.0365N NaCl to a maximum of 3.46 mgrms. per c.c. in 0.1150N NaCl, and then falls to 1.32 mgrms. per c.c. in 0.2725N NaCl. The hydrogen ion concentration of all saturated solutions is the same and has the value 5.13×10^{-8} . The appearance of an opalescence in solutions of casein occurs when the hydrogen ion concentration has the above value, and this point lies very near the iso-electric point.—J. F. S.

Yeast; Extraction and concentration of vitamine of —. T. B. Osborne and A. J. Wakeman. J. Biol. Chem., 1919, 40, 383-391.

THE authors have prepared a highly active growth-promoting fraction from a dilute acetic acid extract of yeast by fractional precipitation with alcohol. No definite substances were isolated.—J. C. D.

Vitamines; Identity of water-soluble growth-promoting and anti-neuritic —. H. H. Mitchell. J. Biol. Chem., 1919, 40, 399-413.

THE author does not consider that there is sufficient experimental evidence available to justify the assumption that the two accessory factors are identical.—J. C. D.

Vitamine; Fat-soluble — in roots and observations on water-soluble vitamine in roots. H. Steenbock, E. G. Gross, and M. T. Sell. J. Biol. Chem., 1919, 40, 501-529.

THE yellow sweet potato and the carrot contain appreciable amounts of the fat-soluble accessory factor, and are much richer sources of this dietary constituent than rutabagas, dasheen, beets, parsnips, potatoes, and mangels. The carrot, rutabaga, and dasheen are richer sources of the water-soluble accessory factor than sweet potatoes, mangol, and sugar beet.—J. C. D.

Tomato seed and grape seed; Possibility of commercial utilisation of —. J. H. Shrader. J. Ind. Eng. Chem., 1919, 11, 1131-1135.

IS making tomato catsup, pulp, soup, etc., the skins, cores, and seeds are separated from the juice and pulp; the waste (skins etc.) amounts to about 5% of the fruit and contains 10% of seeds. Grape pomace is about 20% of the grapes and contains

about 25% of seeds. These two waste products are obtained in factories in many different districts, but not in sufficient quantity in any one to make utilisation profitable. By working out the cost of transport etc., the author comes to the conclusion that a fair profit could be made by bringing the seeds alone to some central point and there pressing them to obtain oil and press-cake, but that it would not be profitable to transport the whole of the wastes.—W. P. S.

Whale (blubber) oil; Use of hydrogenated — as a food. H. Thoms and F. Müller. Z. Unters. Nahr. Genussm., 1919, 38, 191-198.

FEEDING experiments on men showed that hydrogenated whale oil, m. pt. 36°-37° C., is an efficient substitute for butter.—W. P. S.

Food dehydration; Low temperature-vacuum —. K. G. Falk, E. M. Frankel, and R. H. McKee. J. Ind. Eng. Chem., 1919, 11, 1036-1040.

THE dehydration of meat at 50°-70° C. under a pressure of 2 in. of mercury results in the reduction of the water content by 65% in from 3 to 8 hrs., according to the size of the pieces. Subsequent exposure to the atmosphere for 3 days further reduces the water-content, and the meat then contains about 10% of water. Fish, vegetables, and fruits may be dried in a similar way, and all the products are of a satisfactory character.—W. P. S.

Vegetables; Carbohydrates of fresh and dehydrated —. K. G. Falk. J. Ind. Eng. Chem., 1919, 11, 1133.

DETERMINATIONS of reducing sugars, dextrans, and starch (soluble and insoluble) in fresh and dehydrated (air or vacuum) carrots, potatoes, cabbages, and turnips showed that the drying process did not cause any change in the carbohydrate distribution.—W. P. S.

Foodstuffs and feeders; Quantitative micro-analysis of —. W. Herter. Z. Unters. Nahr. Genussm., 1919, 38, 65-89.

A DESCRIPTION of methods of preparation and microscopical examination of various food products and feeding stuffs, including methods of calculating the proportions of various components from the diameters of their particles observed under the microscope.

Lactic acid bacteria. Svanberg. See XVIII.

Pepsin. Northrop. See XX.

Saccharin and benzoic acid. Schowalter. See XX.

PATENTS.

Food; Prepared health — and process of making the same. Fruited Cereal Co., Assignees of J. P. Pursell, Quincy, Ill., U.S.A. Eng. Pat. 121,284, 3.12.18. (Appl. 19,984/18.) Int. Conv., 4.12.17.

A CEREAL is cooked in the form of whole grains, e.g., by direct action of steam under pressure; fruit—preferably consisting of a mixture of dates, figs, and raisins, which may have been previously dried and ground—is then added to the cereal. Cooking is continued for some time further, and the product is then dried and toasted and if necessary granulated.—J. H. L.

Baking compound. W. P. M. Grelek, Assignor to B. H. Grelek, Lincoln, Nebr. U.S. Pat. 1,322,561, 25.11.19. Appl., 4.12.15.

THE product consists of the dried solids of sour milk mixed with an alkaline carbonate.—J. H. L.

Dehydrating fruits, vegetables, etc.; Process of and apparatus for —. C. Shorman, Chicago, Ill. U.S. Pat. 1,321,072, 9.12.19. Appl., 4.6.13.

THE material to be treated is contained in a closed

chamber from which the air is exhausted, the air after withdrawal being compressed, freed from moisture in a condenser (preferably consisting of a coil or coils of pipe in a tank or tanks of water), and then (reheated if necessary in another coil) used again for dehydration.—B. M. V.

XIXB.—WATER PURIFICATION; SANITATION.

Boiler feed water; Lime-barium softener for treatment of —. C. A. Mehring. Chem. and Met. Eng., 1919, 21, 629—632.

THE water to be treated is admitted through a valve controlled by the amount of water drawn from the softening plant to a tank at the top of the apparatus, whence it passes through a siphon tank to the bottom of a lower, outer cone, where it is intimately mixed with slaked lime which has been mixed to a thin paste with a portion of the water under treatment. This removes temporary hardness. As the water rises in the outer cone the precipitate settles and is drawn off at the bottom. The water passes through a pipe leading from the top of the lower, outer cone to the bottom of an inner, upper cone where it is thoroughly mixed with barium carbonate, thus removing permanent hardness. The impurities settle as the water rises in the cone and are drawn off, whilst the water passes through a filter comprising a perforated plate, a brass screen, 6 inches of gravel, and 16 inches of sand, and is collected in a triangular space below the filter bottom, whence it is drawn off as required. The filter is provided with air washing and water washing devices. The operation is controlled by the analysis of drip samples taken through $\frac{3}{8}$ " pipes at various parts of the plant. Details of analyses and results and cost of treatment are given.—S. S. A.

Carbon monoxide; Rapid determination of — in air. A. B. Lamb and A. T. Larson. J. Amer. Chem. Soc., 1919, 41, 1908—1920.

Two methods for the rapid estimation of small quantities of carbon monoxide in air are described. In the first, the gas is passed over a platinum wire heated to redness and the carbon monoxide partially burnt in contact with it. The gas then passes over heated copper oxide where the carbon monoxide is completely burnt, next through a spiral tube contained in a thermostat to bring it to its original temperature, and finally over a second exactly similar platinum wire heated by the same current as the first. The two platinum wires are made arms of a Wheatstone bridge, and since the first wire will necessarily be hotter than the second they will have different resistances. The change in the resistance of the first wire gives a measure of the carbon monoxide concentration. In the second method the mixture is passed through a platinised platinum catalyst when the carbon monoxide is completely burnt. The temperature change is measured by a thermo-element and indicates the concentration of the carbon monoxide. Both methods are rapid and the latter is particularly accurate, easy to instal and operate. The catalyst, with reasonable precautions, remains active for many weeks.—J. F. S.

Mustard gas; Effect of moisture content on the permeability of fabrics to —. G. H. Clowes, G. St. J. Perrott, B. Gordon, and E. L. Greensfelder. J. Ind. Eng. Chem., 1919, 11, 1016—1019.

THE addition of 4—5% of water to heavy duck and a gelatin-impregnated fabric facilitated the passage of mustard gas through the fabrics; with large amounts of water the passage of the gas was partially inhibited. A corresponding but smaller in-

crease in permeability occurred when oiled cloth was treated with water in quantity up to 4%. It appears that at the point of maximum penetration a film of water covering the fabric interferes with surface adsorption of the mustard gas and possibly aids the passage of the gas along the surfaces by capillarity. With more water, and as the interstices of the fabric are filled gradually with water, the passage of the gas would be at first impeded and ultimately almost inhibited, since the rate of passage by solution in water and diffusion would necessarily be slow and associated with considerable hydrolysis of the gas.—W. P. S.

Ammonia respirators. Perrott and others. See VII.

PATENTS.

Softening, treating, and filtering water for steam boilers and other industrial purposes; Automatic method of —, and apparatus therefor. E. Wade-Wilton and W. Hepworth, Bramley, and C. H. Fuessly, Eccles. Eng. Pat. 136,225, 6.12.18. (Appl. 20,216/18.)

WATER to be treated enters a tank through a control valve, and is raised in temperature by the introduction of live steam. A predetermined quantity of reagent is then automatically added and mixed, and the water is discharged from the tank through a protected pipe which prevents the discharge of any scum. From the tank the water passes to a filter, through which it travels in an upward direction, and leaves by a pipe at the top. This pipe is bent downwards and then upwards, and opens in an enclosed space, from which the air escapes by a vent valve, and the water flows away through a pipe in the bottom of the space to the boiler or to a collecting tank.—J. H. J.

Water; Purification of — [with chlorine]. F. P. Candy, London. Eng. Pat. 136,266. (Appls. 20,671, 12.12.18 and 3955, 18.2.19.)

AN apparatus is described specially suitable for the addition of chlorine gas from a cylinder of the liquid substance to water flowing in a closed conduit under pressure. The gas is admitted to a pressure-regulating chamber partly filled with water by a connection to the water conduit. A float or diaphragm in the pressure regulator operates a valve which controls the admission of the gas, so that the pressure of the gas in the regulator is reduced to that of the water. The regulator is placed at a lower level than the conduit, so that the water in the regulator is at a slightly higher pressure than that in the conduit, which enables the gas to be passed from the regulator into the conduit. The pressure regulator may be placed at a higher level than the conduit if desired, in which case the water in the regulator is supplied under the action of an air pressure controlled by the water pressure in the conduit. If the water to be treated is flowing through a suction pipe, the water for the pressure regulator is supplied from a tank at a suitable height above the regulator. In order to prevent the corrosive action of moist chlorine, the regulator may be filled with carbon tetrachloride, and a trap is inserted between the regulator and the water conduit to prevent carbon tetrachloride from entering the latter. The same object may be effected by a layer of paraffin oil on the surface of the water in the regulator.—J. H. J.

Water purification. W. O. Snelling, Pittsburgh, Pa. U.S. Pat. 1,325,213, 16.12.19. Appl., 31.7.15.

PORTLAND cement is hydrated with an excess of a hydrating agent containing a salt of one of the alkali metals which is capable of removing calcium from the cement. The product is a water-softening agent.—J. H. J.

Refuse; Destruction of house and other —, and the recovery therefrom of useful materials. E. Box, Caulfield, Victoria. Eng. Pat. 136,243, 9.12.18. (Appl. 20,427/18.)

REFUSE is treated in a closed and heated retort with air or superheated steam delivered below the charge. The gases given off are collected from the top of the retort, cooled to deposit tar, and passed through a condenser into a scrubber divided into compartments by baffles partly submerged in a liquid. In each compartment a scrubbing-box is mounted crankwise on a common spindle passing through the scrubber. The boxes are wetted as they rotate by liquid obtained from the condenser and supplied from a tank above the scrubber. The gases from the scrubber pass to a blower which also takes in air through a valved inlet. The blower is supplied also with powdered carbonaceous material (obtained from the refuse after burning in the retort), which is discharged with the air and gases through the nozzle of the blower into the furnace of the retort.—J. H. J.

Waste material; Process and apparatus for reduction of organic —. C. G. Darrach, Assignor to G. Darrach, Philadelphia, Pa. U.S. Pat. 1,325,352, 16.12.19. Appl. 29.7.15.

AN apparatus is described consisting of a digester connected at the bottom to a clarifying chamber containing granular material supported between two bodies of liquid. Organic material is treated in the digester, and the liquid from the digester is passed into the clarifier, where a flock-forming reagent is added to it.—J. H. J.

Insecticide. H. A. Richter, Long Island City, N.Y. U.S. Pat. 1,321,818, 16.12.19. Appl. 18.8.17.

ABOUT 9—15 parts of arsenious oxide and 6—10 parts of caustic alkali are ground together in a dry state and mixed with 70% of an inert dry powder containing slaked lime. The product is free from lumps, and is capable of being applied as a dusting powder.—J. H. J.

Purifying gas. Eng. Pat. 135,931. See 11A.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Alkaloids; Use of silicotungstic acid for the determination of —. E. Taigner. Z. anal. Chem., 1919, 58, 346—352.

ATROPINE, strychnine, and cocaine are precipitated completely from their respective hydrochloric acid solutions by 10% silicotungstic acid solution; after the addition of sodium chloride, the precipitate is collected on a filter, washed with 1% hydrochloric acid, dried at 40° C., and weighed. The precipitated compounds have the general composition: $12\text{WO}_3 \cdot \text{SiO}_2 \cdot 2\text{H}_2\text{O} \cdot \text{alkaloid}$, consequently the atropine compound contains 28.61, the strychnine compound 31.69, and the cocaine compound 29.10% of alkaloid.—W. P. S.

Ethylmorphine sulphate. J. L. Thomson. Pharm. J., 1920, 104, 7.

ETHYLMORPHINE sulphate has the formula $(\text{C}_{17}\text{H}_{19}\text{O}_2 \cdot \text{N} \cdot \text{O} \cdot \text{C}_2\text{H}_5)_2 \cdot \text{H}_2\text{SO}_4 \cdot 5\text{H}_2\text{O}$; at 15.5° C. 1 part of the salt is soluble in 9.5 parts of water or 111 parts of 90% alcohol.—W. P. S.

Drugs and poisons; Physical theory of the action of —. I. Traube. Biochem. Zeits., 1919, 98, 177—197.

A THEORETICAL paper. The pharmacological action

of drugs and toxins is attributed mainly to their physical properties. It is urged that more attention ought to be given to the physical side of these principles in the study of chemotherapy.—S. S. Z.

Homologues of hydroquinine and their toxins; Surface action of the —. I. Traube. Biochem. Zeits., 1919, 98, 197—205.

WITH the hydrocupreins and their homologues there is a parallelism between their disinfecting power against many bacilli and cocci, mostly gram positive organisms, and their surface action. An analogy also exists between the susceptibility of bacteria towards basic stains and their power of adsorption of hydrocupreins and their toxins.

—S. S. Z.

Kephalin; Glycerophosphoric acid in —. P. A. Levene and I. P. Roff. J. Biol. Chem., 1919, 40, 1—16.

THE presence of glycerophosphoric acid in kephalin has been established by the isolation and identification of that component. It is apparently identical with the acid found in lecithin. (See further, J. Chem. Soc., 1920, i., 137.)—J. C. D.

Thyroxin; Chemical identification of —. E. C. Kendall and A. E. Osterberg. J. Biol. Chem., 1919, 40, 265—331.

EVIDENCE as to the supposed structure of thyroxin, the physiologically active component of the thyroid gland, is reported. The compound may occur in several isomeric forms, and appears to be a 4,5,6-tri-hydro-4,5,6-tri-iodo-2-oxy- β -indolepropionic acid. (See further, J. Chem. Soc., 1920, i., 180.)—J. C. D.

Yeast nucleic acid; Structure of —. H. Ammonia hydrolysis. P. A. Levene. J. Biol. Chem., 1919, 40, 415—421.

THE author reviews the recent theories of Jones (this J., 1916, 707; 1917, 402) and Thannhauser (compare this J., 1916, 63) regarding the structure of nucleic acid, and points out their weak points. He advances a formula for yeast nucleic acid which satisfies the known facts. (See further, J. Chem. Soc., 1920, i., 193.)—J. C. D.

Pepsin; Quantitative estimation of —. Effect of hydrogen ion concentration. J. H. Northrop. J. Gen. Physiol., 1919, 2, 113—122.

THE change of electrical conductivity during peptic digestion may be employed as a measure of the amount of pepsin present. The combination of pepsin with the substrate and the influence of the reaction of the medium on this combination, and hence on the rate of digestion, is discussed. (See further, J. Chem. Soc., Feb., 1920.)—J. C. D.

Catalysis; Thermal problem in organic contact —. W. J. Huff. Trans. Amer. Electrochem. Soc., 1920, 175—191. [Advance proof.]

A GENERAL review of the heat problems met with in organic catalytic reactions with a more detailed description of specific typical examples. As typical endothermic reactions the cracking of petroleum by the Rittman process, and the dehydration of ethyl alcohol over aluminium oxide, are cited. In these cases momentary local superheating entails locally increased reaction velocity and consequently locally increased absorption of heat, so that the reaction itself serves, figuratively speaking, as a thermal balance wheel. When the product, therefore, is stable enough to use a long reaction tube, as is the case with the catalysis of alcohol to ethylene, an endothermic catalysis presents no great difficulties. In the cracking of petroleum, however, the desired products are themselves liable to further decomposition, and the catalysis must be conducted within very narrow limits of temperature, e.g., 550°—

580° C. for Pennsylvania oil. The problems offered by exothermic reactions are more difficult of solution. As examples of such are cited the partial combustion catalysis of naphthalene to phthalic acid (see Gibbs and Conover, Eng. Pat. 119,518; this J., 1918, 684A) and of benzene to maleic anhydride (U.S. Pat. 1,318,632; this J., 1920, 44A), and chlorination catalyses such as the preparation of carbon tetrachloride from methane and chlorine with the aid of a treated charcoal catalyst. For disposing of the surplus heat various methods are adopted. A well-known expedient consists in increasing the heat capacity of the incoming vapours by dilution with an inert material, or an excess of one ingredient, the method adopted, for example, in the catalytic reduction of acetaldehyde to ethyl alcohol (see Eng. Pat. 120,163; this J., 1919, 28A). The latent heat of vaporisation of an atomised liquid may also be utilised for absorbing surplus heat. For large scale production the author favours the principle of alternate coolings and short catalyst contacts, although the practical application to reactions catalysed only by non-metallic materials present many difficulties.—G. F. M.

Saccharin and benzoic acid; Separation of — E. Schowalter. Z. Unters. Nahr. Genussm., 1919, 38, 185—194.

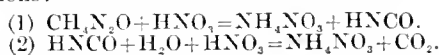
SACCHARIN and benzoic acid are extracted together from foods, etc., by most of the methods employed for the determination of benzoic acid, but the two substances may be separated from one another by means of carbon tetrachloride, in which saccharin is practically insoluble. The ethereal solution obtained as in Von der Heide's method for the determination of benzoic acid (this J., 1910, 367) is evaporated at a low temperature, the residue dried in a vacuum desiccator to remove the last traces of acetic acid, and then treated with carbon tetrachloride. The benzoic acid dissolves and may be titrated. The insoluble residue of saccharin may be identified as such by the usual reactions. Saccharin cannot be separated from benzoic acid by sublimation, since both begin to sublime at about 120° C. Attention is directed to the fact that saccharin, its anhydride and sodium salt, benzoic acid and soluble benzoates are all absorbed to a considerable extent by animal charcoal from their aqueous solutions.—W. P. S.

Acetylsalicylic acid. J. H. Ramsay. Pharm. J., 1920, 101, 7—8.

OF 11 samples of acetylsalicylic acid examined by the author only 3 were free from salicylic acid; the quantity of this acid present in the other 8 samples varied from 3 to 15 parts per million. Ten out of 12 samples of acetylsalicylic acid (aspirin) tablets contained free salicylic acid, the amounts varying from 7 to 50 parts per million.—W. P. S.

Carbamide [urea]; Decomposition of — in the presence of nitric acid. T. W. Price. Chem. Soc. Trans., 1919, 115, 1354—1360.

THE reaction between urea and nitric acid is unimolecular, and as the products consist entirely of carbon dioxide and ammonium nitrate it is analogous to the decomposition of urea by hydrochloric or sulphuric acids and proceeds according to the equations:



As the velocity diminishes with the concentration of nitric acid it appears that only free urea, and not its nitrate, takes part in the reaction (1) above. According to the dissociation theory of the decomposition of urea, whereby ammonia and cyanic acid are supposed to be the primary decomposition products, the addition of a salt containing an ammonium ion should have a retarding influence on

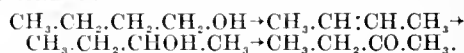
the decomposition, but the reverse was found to be the case, and hence it appears that this theory is not applicable. The decomposition velocity is small below 80° C., and at 30°—40° C. is negligible, and hence the use of urea to remove nitrous acid from nitric acid will not be accompanied by a loss of nitric acid at low temperatures.—G. F. M.

Fatty acids; Volatility with steam of lower — in dilute aqueous solution. E. J. Witzemann. J. Amer. Chem. Soc., 1919, 41, 1946—1951.

THE volatility of the lower fatty acids (formic to butyric) in steam increases with increasing molecular weight. The addition of neutral chlorides to the solution increases the volatility. The amount of increase runs parallel with the electro-affinity of the metallic ion of the salt. The following order is given for metallic chlorides: potassium, sodium, strontium, barium, calcium, magnesium, and aluminium. The chlorides of iron, manganese, and copper are irregular in their action. (See also J. Chem. Soc., 1920, i., 138.)—J. F. S.

Methyl ethyl ketone; Production of — from *n*-butyl alcohol. A. T. King. Chem. Soc. Trans., 1919, 115, 1404—1410.

METHYL ethyl ketone is produced from *n*-butyl alcohol by dehydration to β -butylene, re-hydration of the latter to *sec*-butyl alcohol, and dehydrogenation of the alcohol to the ketone, according to the scheme:



The dehydration of butyl alcohol is effected by passing the vapour over glacial phosphoric acid on pumice contained in a copper tube at a temperature of 280°—400° C. The β -butylene, produced without any appreciable side reactions, is condensed, the liquid shaken under its own pressure with 75% sulphuric acid, and, when absorption is complete, the mixture diluted with an equal volume of water, and distilled. A yield of about 90% of the theoretical quantity of *sec*-butyl alcohol is obtained from the distillate after salting out and drying, and it is then dehydrogenised over a catalyst of reduced copper at 290° C. by the method of Sabatier and Senderens. The yield of the purified ketone amounts to about 70% of the theoretical calculated from the crude butylene employed.—G. F. M.

Chemotherapeutic studies on organic compounds containing mercury and arsenic. G. W. Raiziss, J. A. Kolmer, and J. L. Gavron. J. Biol. Chem., 1919, 40, 533—552.

A NUMBER of new compounds containing both mercury and arsenic in organic combination have been prepared. These compounds are of the type of substituted phenylarsinic acid mercuric acetates. They were not found to possess germicidal properties superior to those of ordinary organic mercury compounds. (See further, J. Chem. Soc., 1920, i., 196.)—J. C. D.

Arsenious acid; Influence of — on growing tissues. R. Cobet. Biochem. Zeits., 1919, 98, 294—314.

IN plants, no improvement in the growth was observed by the application of arsenious acid. Concentrations as low as 1/200,000 were found to be very toxic. Frog spawn and tadpoles were not so susceptible to the toxic effect of arsenious acid, but neither improvement nor retardation in growth of these organisms could be traced to the influence of the arsenic compound.—S. S. Z.

Alcohol from coke-oven gas. Bury. See IIa.

Ignition of electrically charged ether. Holde. See IIa.

Ether-alcohol-air and acetone-alcohol-air mixtures. White and Price. See IIa.

Mercury compounds. Denigès. See VII.

Electrochemical production of organic compounds. Thatcher. See XI.

Fatty acids. Windisch and Dietrich. See XXIII.

PATENTS.

Alcohol; Manufacture of —. Elektrizitätswerk Lonza, Gampel, Switzerland. Eng. Pat. 134,521, 8.8.19. (Appl. 19,573/19.) Int. Conv., 27.10.18.

Is the catalytic reduction of acetaldehyde to ethyl alcohol by means of an excess of hydrogen, which, after the separation of the alcohol formed, is returned to the operation by a circulating device (Eng. Pat. 120,163; this J., 1919, 28a), the production of ether as a by-product and the gradual poisoning of the catalyst by the decomposition products of acetaldehyde, are avoided by circulating with the hydrogen an amount of oxygen not exceeding 0.3. The temperature of the reaction chamber is maintained between 90° and 170° C. by cooling, or preferably by using the hydrogen in such excess that it absorbs and removes the superfluous heat of the reaction. Whereas without the use of oxygen the yield of alcohol fell after 30 hours nearly to zero, an average yield of 95% of the alcohol theoretically possible was obtained in a run extending over 211 hours in which 0.15% of oxygen was used, whilst a content of 3.5% of ether in the condensed products in the former case was reduced to 0.5% in the latter. —G. F. M.

De-toxicated vaccine; Preparation of —. D. Thomson, London. Eng. Pat. 136,036, 11.2.19. (Appl. 3695/19.)

In the preparation of vaccines from bacteria the removal of undesirable toxins is effected by dissolving the centrifuged bacteria in N/10 sodium hydroxide at blood heat and re-precipitating the bacterial substance by the addition of N/5 hydrochloric acid containing 2.5% of sodium chloride. Precipitation is promoted by centrifuging, and as it occurs more readily in concentrated solutions it is important to dissolve a maximum amount of germs in a minimum amount of alkali, e.g., in an equal bulk of N/10 solution. The supernatant fluid contains a proportion of the toxin present, but by resolution and re-precipitation repeated several times until the supernatant liquid ceases to give a precipitate with picric acid, the final germ precipitate is rendered free from toxic substances. As an alternative to repeated solution and re-precipitation the toxin may be removed by washing the first precipitate obtained with a weak acid solution such as 0.5% solution of sodium dihydrogen phosphate. De-toxication is usually complete after 4 or 5 washings. In either case the de-toxicated precipitate is collected and mixed with a solution containing 0.5% each of sodium dihydrogen phosphate and phenol and is standardised to a strength of ten thousand millions of germs per c.c.—G. F. M.

Acetylsalicylic acid; Process for preparing double compounds of caffeine with alkali salts of —. H. Sefton-Jones, London. From Chem. Fabr. J. A. Wülfing, Berlin. Eng. Pat. 136,187, 8.1.18. (Appl. 484/18.)

ACETYSALICYLIC acid (2 mols.), an alkali carbonate (1 mol.), and caffeine (1 mol.) are homogeneously mixed and the mixture is moistened with an alcohol, ester, ketone, or chlorinated hydrocarbon of the aliphatic series to the consistency of a thick paste, which is kneaded until a sample dissolves in water to a clear solution without the liberation of carbon dioxide. The solvent is then removed by evaporation at a low temperature, and the product, con-

sisting of the acetylsalicylate of caffeine and the alkali metal is dried in a vacuum. Alternatively the acetylsalicylate of an alkali metal, such as for example lithium acetylsalicylate, may be brought into reaction with caffeine in the above mentioned proportions in a similar way.—G. F. M.

Camphor; Method of changing isoborneol into —. R. L. Andraeu, Assignor to E. I. du Pont de Nemours & Co., Wilmington, Del. U.S. Pat. 1,324,140, 9.12.19. Appl., 25.5.18.

ISOBORNEOL is oxidised to camphor with moderately concentrated nitric acid by applying momentarily, and then discontinuing the application of, an electric current.—G. F. M.

Photochemical process. W. O. Snelling, Alentown, Pa. U.S. Pat. 1,325,211, 16.12.19. Appl., 6.2.17.

A HALOGENATED carbon compound is exposed to actinic rays in the presence of a substance capable of removing the halogen.—J. H. J.

Desoxycholic acid; Manufacture of products of addition of —. H. Wieland, Munich. Eng. Pat. 105,769, 20.1.17. (Appl. 5589/17.) Int. Conv., 10.2.16.

SEE U.S. Pat. 1,252,212 of 1918; this J., 1918, 108 A.

Ethylene dichloride; Process of preparing —. E. C. R. Marks, London. From Union Carbide Co., New York. Eng. Pat. 136,189, 2.7.19. (Appl. 16,488/19.)

SEE U.S. Pats. 1,315,542 and 1,315,545 of 1919; this J., 1919, 847 A.

Isobutyl ester of oleic acid. E. Preiswerk, Basle, Switzerland, Assignor to the Hoffmann-La Roche Chemical Works, New York. U.S. Pat. 1,318,461, 11.10.19. Appl., 16.8.18.

SEE Eng. Pat. 123,685 of 1918; this J., 1919, 268 A.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Process plate; Emulsion for [photographic] —. R. E. Slade and G. I. Higson. Phot. J., 1919, 59, 260–265.

THE characteristic curve of a photographic plate as determined by Hurter and Driffield's method has been found to depend on the relation of the different sizes of silver halide grains present and their quantities, the thickness and opacity of the film, and the time and method of development, the first of these factors being the most important. Regarding the curve of an emulsion as the sum of the curves due to the various groups of particles of the same size, it follows that, in general, the more uniform the grains of an emulsion the steeper its curve. The actual size of the grains is less important than the degree of uniformity. A plate for process work requires to have a steep curve, and therefore to have uniformity in size of grain. Photomicrographs of a number of commercial process plates, of which several are reproduced, support this view.—B. V. S.

[Photographic] *toning with nickel and cobalt.* J. Dubreton. Bull. Soc. Franç. Phot., 1916, 6, 304.

SOLUTIONS of nickel and cobalt ferrieyanides are both suitable for toning bromide prints. Considerable latitude in the composition of the baths is permissible, the solution recommended for nickel being a mixture of equal parts of solutions containing respectively 1% of potassium ferrieyanide and 2–5% of potassium citrate in water, and 1% of nickel chloride and 2–5% of potassium citrate. The tones obtained vary from a deep, warm brown

to a red similar to that obtained with a copper ferricyanide solution; the intermediate tones, however, are pleasanter than those given by copper, and there is less tinting of the whites if toning is carried to finality. For cobalt toning, the second solution contains 2% of cobalt nitrate or chloride with 6% to 10% of potassium citrate; a violet-rose tint is given, very effective in portraits. Hard prints are preferable, and the strength of the bath in cobalt must be maintained to avoid reduction of the print.—B. V. S.

[Photographic] sulphur toning. R. Namias. 11 Progressio Photographico, July, 1914. Bull. Soc. Franç. Phot., 1919, 6, 305. Selenium toning process of Namias. J. Dubreton. Bull. Soc. Franç. Phot., 1919, 6, 306.

CRYSTALLISED sodium sulphide (60 grms.) and selenium powder (3 grms.), in the commercial form of small brownish-black sticks, are heated together in a porcelain crucible, with thorough stirring. After cooling, the mass is treated with water (1 litre), a deep red transparent solution being obtained which keeps well in full stoppered bottles. This bath gives with bleached bromide prints a fine purple-brown tone similar to that of a gold-toned print-out paper, with no tendency to the disagreeable yellowish appearance often obtained with the usual sulphide toning methods. Pure whites are obtained unless too many prints are treated in one bath.—B. V. S.

PATENTS.

Colour photography. C. Raleigh, Jersey City, N.J., and W. V. D. Kelley, Brooklyn, N.Y., Assignors to Prizma Inc. U.S. Pat. 1,325,204, 16.12.19. Appl., 20.12.16.

In obtaining a colour record, the exposure is made through a moving graded colour screen so that one end of the exposure is made to practically white light, and the other end to the full colour.

—B. V. S.

Coloured cinematograph films; Process of producing —, and apparatus therefor. L. F. Douglass, San Rafael, Cal. U.S. Pat. 1,325,280, 16.12.19. Appl., 13.8.18.

In two-colour cinematography, with alternating red and green taking screens, a portion of the green screen exposure is made through a blue screen, the proportion of blue screen exposure being increased with increase of the distance between the lens and the object.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Trinitrotoluene; Hygroscopicity of —. W. J. Huff. Chem. and Met. Eng., 1919, 21, 570—571.

THE allegation that trinitrotoluene is hygroscopic is denied. Although no measurements of the vapour pressure of its saturated solution are available, calculation from the temperature-solubility curve would indicate it to be 99.997% of that of pure water at 25° C., and this points to trinitrotoluene being practically non-hygroscopic. Laboratory tests by Taylor and Cope's method (this J., 1916, 944) are not suitable for determinations of hygroscopicities below that of potassium perchlorate, but they confirmed, nevertheless, that trinitrotoluene is not hygroscopic and that apparent gain in weight was due to deposition of moisture by adsorption and condensation attending minute temperature fluctuations.—W. J. W.

Nitrotoluenes; Studies on the —. I. Binary systems of a nitrotoluene and symmetrical trinitrotoluenes. J. M. Bell and J. P. Sawyer. J. Ind. Eng. Chem., 1919, 11, 1025—1028.

The freezing points of binary systems in which one

component was 2,4,6-trinitroxylylene and the other a nitrotoluene have been determined by the cooling curve method. Binary mixtures of trinitro-m-xylene (m.pt. 182° C.) with 1,2,4,6-trinitrotoluene (m.pt. 80.5° C.) showed an eutectic point 74.8° C., corresponding (by interpolation) to 8% of the former. The mixtures of trinitro-m-xylene and 1,2,4-dinitrotoluene (m.pt. 69.4° C.) had eutectic point 67.7° C., corresponding to 6% of the former, whilst the eutectic point (50.5° C.) of the mixtures of trinitro-m-xylene and p-mononitrotoluene (m.pt. 51.2° C.) corresponded to 2% of the former. The binary eutectic temperature for mono- and dinitrotoluenes (26.4° C.) was lowered to 25.7° C. by the addition of successive small amounts of trinitro-m-xylene, and analogous reductions of the eutectic points were produced by adding trinitro-m-xylene to mixtures of the other two nitrotoluenes. The formula expressing the relationship between the heat of fusion, m.pt., and composition of ideal solutions is applicable to binary mixtures of trinitro-m-xylene especially when mononitrotoluene is the second constituent.—C. A. M.

Nitrotoluenes; Studies on the — II. Refractive indices of mixtures of p-nitrotoluene, 1,2,4-dinitrotoluene, and 1,2,4,6-trinitrotoluene. J. M. Bell and E. O. Cummings. J. Ind. Eng. Chem., 1919, 11, 1028—1029.

THE refractive indices of mixtures of p-nitrotoluene, 1,2,4-dinitrotoluene, and 1,2,4,6-trinitrotoluene were determined at 38°—39° C., the eutectic temperatures of the mixtures being about 17° C. The results corresponding to the percentage composition by weight of the three components are given in tabular form, and lines of equal refractive index are shown in the conventional triangular diagram. In all but a few instances the observed results agreed with those calculated by means of the formula:

$$n = 1.542m + 1.572d + 1.580t,$$

where m , d , and t are the fractions by weight of mono-, di-, and trinitrotoluene in the mixture. (See also this J., 1920, 84A.)—C. A. M.

Black powder; Hygroscopic properties of —. G. B. Taylor. J. Ind. Eng. Chem., 1919, 11, 1032—1031.

THE relative rates of the absorption of moisture by three samples of ordinary black gunpowder were determined by the methods used in a previous investigation of detonators (this J., 1916, 944). The presence of potassium perchlorate (up to 5%) in mixtures with potassium nitrate has only a slight effect upon the rate of moisture absorption.

—C. A. M.

Ether-alcohol-air and acetone-alcohol-air mixtures. White and Price. See IIa.

Toluene. Lumsden. See III.

Nitro-compounds. Van Duin. See XXIII.

PATENTS.

Mixing materials [nitrocellulose] with fluids; Apparatus for —. E. G. Loomis, Newark, N.J., Assignor to The Arlington Co., Arlington, N.J. U.S. Pat. 1,324,774, 9.12.19. Appl., 12.12.16.

AN apparatus for mixing nitrocellulose with water by imparting motion to it whilst it is suspended in the water, and for removing the mixture through a tangential conduit.—W. J. W.

Chlorate explosives. F. Müller, Frankfurt. Ger. Pat. 307,039, 9.1.18.

BENZOATES may be used as ingredients of chlorate explosives. A typical composition consists of potassium chlorate, 3 parts; charcoal, 1 part; and sodium

benzoate, 14 parts. Such explosives are insensitive to pressure and shock, and detonate at a low temperature, approximately 150°C .—W. J. W.

Incendiary bombs and projectiles; Composition for —. Deutsche Gold- und Silberscheideanstalt vorm. Roessler, Frankfurt. Ger. Pat. 298,957, 9.5.15.

THE filling material consists of an alkali metal, mixed with a substance which readily evolves oxygen, such as sodium peroxide, sodium nitrate, etc. The latter is ignited by easily inflammable mixtures such as aluminium and iron oxide or powdered alkali metal and peroxide, and ignition of these is effected by the usual fuses or igniters or by the action of water or acids. The oxygen-evolving composition and the inflammable mixture are placed in a recess, formed in the alkali metal.

—W. J. W.

Nitrocellulose. Ger. Pats. 314,317–8. See XIII.

XXIII.—ANALYSIS.

Potentiometers for thermo-element work. W. P. White. Bull. No. 153, Amer. Inst. Min. and Met. Eng., Sept., 1919, 1763–1772.

DIRECT deflection methods of measuring temperatures by means of thermo-couples are characterised by lack of relative precision and necessitate the absolute constancy of all resistances. The limit of accuracy of such methods, variously estimated as between 1 part in 500 and 1 part in 2000, is far below that of a simple potentiometer method. This latter method is more costly as well as more complex and necessitates the use of a standard cell and attention to the working cell. Various modifications whereby these and other objections to the potentiometric method are eliminated, at least in part, are detailed. These include the pyro-volter and the Harrison and Foote instrument. In the latter the circuit resistance is adjusted to compensate for any changes or differences in the thermo-element, and the thermo-couple itself is used as a source of temporarily constant current. No battery is required. Both instruments are dependent on the calibration of an ammeter. Higher precision is as yet attainable only by the use of a standard cell. The portable potentiometer of the Leeds and Northrup Co. permits an accuracy of 0.25° with ordinary base-metal thermo-couples. The Diesselhorst-Wolff and the White designs of potentiometer (J. Amer. Chem. Soc., 1914, 36, 1859, 2011) enable readings to be made to within 0.1 microvolt. The latter form is very little affected by corrosive gases. Both are deflection potentiometers enabling part of the readings to be taken direct from the galvanometer deflections, with a gain of speed. Speed is further enhanced by using the instruments as double potentiometers. The precision possible renders these instruments suitable for use in fundamental standardisation work, and in calorimetry.—J. S. G. T.

Thermal expansion; Stretched wire apparatus for measuring —. A. W. Gray. Chem. and Met. Eng., 1919, 21, 667–671.

THE difficulties in measuring small changes in length accurately and without the errors incidental to changes in the apparatus are overcome by this device. Two wires in contact with the ends of the bar under examination are kept stretched by vanes immersed in oil, which by its viscosity prevents swinging of the wires; or, where the bar itself is immersed in a bath, the wires are stretched upwards to another bar, which is rigidly connected to the centre of the test bar, and kept at tension by weights. The ends of the sample must be ground and transverse motion of the wires caused by ex-

pansion of the sample is observed through micrometer microscopes. The apparatus may be used with accuracy at temperatures between -150° and -350°C . A description is given of a dilatometer which embodies the above device and is suitable for industrial purposes. Notes on possible errors and their correction are supplied. The apparatus may also be utilised for measuring magnetostriction in nickel steels and deformation of materials during testing.—W. J. W.

Thermal analysis; Simplification of the inverse-rate method of —. P. D. Merica. U. S. Bureau Standards Sci. Paper, No. 336, 1919.

THE recording of time cooling curves is simplified by the use of two stop watches, which are so arranged in a frame that both stems may be pressed together each time the potentiometer connected to the thermo-couple records a definite temperature. The single action stops one watch, thus recording the desired interval and starts the second watch, which commences recording the next interval. The method saves much time and the use of an expensive automatic recording chronograph.—J. F. S.

Melting point of hygroscopic substances; Apparatus for determining the —. H. J. Backer. Chem. Weekblad, 1919, 16, 1561–1565.

THE apparatus consists of a narrow, thin-walled, glass tube connected by a curved lateral branch to a wider tube. The material under examination is placed in the narrow tube, which is then drawn out and sealed. A desiccating agent, such as phosphorus pentoxide, is placed in the wide tube, which must then be drawn out to a capillary, exhausted by means of a water pump and sealed. The melting point can thus be determined while the substance is virtually contained in a vacuum desiccator.

—W. J. W.

Viscometer. G. Baume and H. Vigneron. Ann. Chim. Analyt., 1919, 1, 379–383.

AN apparatus for determining the viscosity or fluidity of liquids consists of a capillary tube widened at its lower end and provided at the top with a bulb, above which is a short length of tube. The latter passes through a cork closing a test-tube, so that the capillary portion hangs vertically with its lower end immersed to a definite length in 20 c.c. of the liquid to be examined, which has been placed previously in the test-tube. A thermometer is fitted in a tube also passing through the cork, and this tube carries a side tube, to which is attached a rubber pressure ball. The test-tube is heated in a suitable vapour jacket, and when a constant temperature is attained the rubber ball is pressed so as to force the liquid up the capillary tube, filling the bulb on the same; pressure is then released and the time noted for the level of the liquid to fall from a mark just above the bulb to one just below it.—W. P. S.

Electrometric titration; Theory of —. W. D. Treadwell. Helv. Chim. Acta, 1919, 2, 672–680.

THE end point in the titration of silver solutions may be determined by measuring the terminal voltage between a standard silver chloride cell and a silver electrode immersed in the solution being titrated. The end point is that at which the terminal voltage becomes zero. This titration may also be carried out in the presence of foreign substances by means of a method described in the paper.—J. F. S.

Electrometric titration. W. D. Treadwell and L. Weiss. Helv. Chim. Acta, 1919, 2, 680–697.

THE end point in many titrations may be determined electrometrically by measuring the terminal voltage between a metal electrode which is immersed in the solution being titrated, and is

capable of sending the same ions into solution, and a comparison electrode made of the same metal and the titration product. The two terminals are directly connected to a voltmeter, and the point of zero potential is the end point of the titration. It is essential that during the titration the liquid should be rapidly stirred, and the authors describe an apparatus in which the comparison electrode is built up in the stirrer. In the case of silver solutions the comparison electrode is Ag/AgCl (saturated solution), a silver wire is the titration electrode, and the titration is carried out with sodium chloride. With pure silver solutions the results are exceedingly good, but in the presence of foreign substances low results are obtained, which may, however, be corrected from the titration curve. Hydrogen sulphide may be titrated with silver ions, using the combination $\text{Ag}/\text{Ag}_2\text{S}$ dilute NaNO_3 as comparison electrode. Mercurous ions may be titrated with chloride ions, using as comparison electrode $\text{Pt}/\text{Ag}/\text{Hg}_2\text{Cl}_2$ dilute H_2SO_4 , and a similar silver-plated platinum wire as titration electrode. Better results are obtained if the titration is carried out with bromide ions, and the calomel in the comparison electrode replaced by mercurous bromide. Copper ions may be titrated by ammonium thiocyanate, using as comparison electrode Cu/CuCNS dilute NaHSO_3 . Zinc ions may be titrated with ferrocyanide ions, using as titration electrode a platinum gauze, and as comparison electrode $\text{Pt}/\text{Zn}/\text{Fe}(\text{CN})_2\text{HCl}$ (5 c.c. in 300 H_2O). The titration in this case is carried out in warm solution (70° C). Ferrous chloride may be titrated with potassium bichromate, using an already completed titration as comparison electrode, and platinum wires in both solutions. This titration is also carried out in warm solutions. Uranous ions may be titrated with potassium permanganate in warm solutions, using platinum wires and a uranyl solution as comparison electrode. Vanadyl solutions may be similarly titrated with permanganate, using platinum wires and a vanadate solution as comparison electrode.

—J. F. S.

Titration with surface-active substances as indicators. Acid estimations with the homologues of the fatty acid series. W. Windisch and W. Dietrich. *Biochem. Zeits.*, 1919, 97, 135—156.

ADOPTING Traube and Somogyi's method of estimating the reaction by means of the alteration in the surface tension brought about by the displacement of the acid or the base of the indicator by the acid or base of the medium (this J., 1915, 511), the authors tried the homologues of the salts of the fatty acid series up to undecylic acid as indicators. The acids with C_9 — C_{11} have been found to be as sensitive as litmus and neutral red. Free acids could be estimated in the presence of a primary phosphate by using the acids as indicators.—S. S. Z.

Gas analysis; Improved Orsat apparatus for —. G. W. Jones and F. R. Neumeister. *Chem. and Met. Eng.*, 1919, 21, 734—736.

A DESCRIPTION is given of the modified form of Orsat apparatus used by the Bureau of Mines, U.S.A., which embodies the principle of removing the hydrogen and carbon monoxide by Jager's copper oxide method, as in the modification of Burrell and Oberfell (*J. Inst. Eng. Chem.*, 1916, 8, 228). The gas burette is provided with a two-way stop-cock at the top, so as to communicate with either the Orsat pipettes containing the usual reagents for the absorption of carbon dioxide, unsaturated hydrocarbons, and oxygen, or with a compensator of the Petersen type. On raising or lowering the levelling bulb, the mercury in the compensator forms contact with a platinum wire and lights a lamp, which facilitates the reading.

Above the Orsat bulbs is an electrical heater consisting of a helical spiral of nichrome wire coated with alternate layers of a mixture of calcined magnesia and sodium silicate and asbestos cord, and covered with asbestos paper. This heater is brought to 300° C., and lowered over the copper oxide tube so that it rests upon supports. The gas is passed through the heated copper oxide tube about 3 times in each direction and then into the combustion pipette. The heater is then raised, the copper oxide tube cooled by means of a blast of compressed air, the mercury levelled, and the contraction corresponding to the hydrogen measured. The gas is then passed into the potassium hydroxide pipette several times, and again through the copper oxide tube, and the contraction corresponding to the carbon monoxide measured. A measured quantity of oxygen is introduced into the combustion pipette, the wires in which are connected with the electric current through a transformer and nichrome resistance wire and are brought to a high temperature by means of a sliding contact. The gas is slowly introduced into the pipette which is cooled externally by means of compressed air to prevent breakage during the combustion. The figures for the total contraction and amount of carbon dioxide formed give the data for the calculation of the quantities of the two predominating hydrocarbons in the gas by means of the usual formulæ. Nitrogen is obtained by difference.

—C. A. M.

Interferometer; Application of the — to gas analysis. J. D. Edwards. *Chem. and Met. Eng.*, 1919, 21, 560—565.

THE analysis of a mixture of two gases may be effected by the interferometer which measures the difference in their refractivities and the change in the refractivity of the mixture produced by a change in the proportion of one of the components. The author describes a method of calibration, and points out possible sources of error in the use of the instrument. Its application to typical mixtures is indicated and a table shows its relative sensitiveness for different gases. It may be used for the analysis of flue gases and for the determination of helium in mixtures. (See also *J. Chem. Soc.*, Feb., 1920).—W. J. W.

Potash; The De Roode method for determining —. T. E. Keitt and H. E. Shiver. *J. Ind. Eng. Chem.*, 1919, 11, 1049—1052.

FURTHER investigation (see this J., 1918, 253 A) showed that the method is trustworthy in the presence of large or small amounts of ammonium salts, organic matter, sodium nitrate, or phosphates, and that it is more accurate than the Lindo-Gladding method, the results in which vary with the kind and amounts of impurities present.

—W. P. S.

Iron; Application of rotating "reducers" in the determination of —. W. Scott. *J. Ind. Eng. Chem.*, 1919, 11, 1135—1137.

FERRIC salts in sulphuric acid solution are reduced quickly and completely by rapidly rotating cylinders of zinc or aluminium with or without the use of an electric current. The reduction appears to be somewhat more rapid with aluminium than with zinc, but the latter is more readily obtained in a sufficiently pure state. (See also this J., 1918, 544 A).—W. P. S.

Copper; Permanganate method for estimating —. L. F. Clark. *J. Ind. Eng. Chem.*, 1919, 11, 1138—1139.

THE titration of cuprous thiocyanate with permanganate solution is rendered more trustworthy when

modified as follows:—The cuprous thiocyanate is precipitated from a 2% sulphuric acid solution (other acids having been expelled by evaporation) by a solution containing 4% of sodium thiocyanate and 10% of sodium sulphite; the mixture is boiled to coagulate the precipitate, the latter is collected on a filter treated previously with starch solution and is washed with hot water containing a few drops of sulphuric acid. The precipitate and filter are then transferred to a beaker and heated with 25 c.c. of 4% sodium hydroxide solution until the paper is disintegrated and the cuprous thiocyanate decomposed; 25 c.c. of cold 15% sulphuric acid and a quantity of 1% ferric sulphate (anhydrous) solution are added and the mixture is titrated with permanganate solution. Towards the end of the titration the colour of the ferric thiocyanate fades and the mixture becomes white; 5 c.c. of concentrated hydrochloric acid is then introduced and the titration completed. Under these conditions 1 atom of copper should, theoretically, be equivalent to 7 atoms of iron, giving a ratio factor of 0.1626; as the result of actual determinations, this factor is found to be 0.1616.—W. P. S.

Tin; Indirect test for the detection of —. F. Feigl. *Chem.-Zeit.*, 1919, 43, 861.

AN ammoniacal solution of dimethylglyoxime gives a precipitate of ferric hydroxide with ferric salts, and this may mask the red precipitate given by traces of ferrous salts with the reagent. The precipitation of the iron as hydroxide may be prevented by adding an organic hydroxy compound (tartaric or citric acids or potassium sodium tartrate) which will form a complex compound with the iron. Ferrous iron is also no longer precipitated as ferrous dimethylglyoxime, but gives a carmine-red solution. This reaction may be used as a sensitive indirect test for tin. After solution of the sulphides of antimony and tin in warm strong hydrochloric acid a portion of the solution is treated with iron wire as a confirmatory test for antimony. In another portion the tin and antimony are precipitated together by means of zinc (free from lead and iron), and the tin dissolved from the metallic deposit by means of strong hydrochloric acid (the presence of a little antimony is negligible). The solution is poured into a hot dilute solution of ferric chloride, a little citric acid or potassium sodium tartrate added, and the liquid tested for ferrous iron by means of an ammoniacal alcoholic solution of dimethylglyoxime. In the presence of stannous ion the ferrous ion formed by reduction gives a red coloration, the intensity of which is proportional to the amount of stannous chloride.

—C. A. M.

Nitro-compounds; Titration and partial reduction of — with titanous chloride. C. F. van Duin. *Chem. Weekblad*, 1919, 16, 1111—1122.

THE method of estimation of nitrogen in nitro-groups by reduction with titanous chloride and subsequent titration was examined. It was found that the titre of titanous chloride solutions suffered considerable alteration after the solution had been heated and that consequently the analytical results yielded by the method were inaccurate. In presence of acids, especially hydrochloric acid, the error is small. Experiments with various classes of nitro-compounds such as nitramines (trinitrophenylmethyl nitramine, aminotrinitrophenylmethyl nitramine, trinitromethyl nitraminophenol, trinitromethyl nitramino-anisole, trinitrodimethyl dinitraminobenzene), tetranitro-compounds (tetranitrophenol, tetranitrophenylmethyl nitramine, tetranitroaniline), and hexanitro-compounds with two nuclei (hexanitrodiphenyl sulphide and sulphone) showed that the results obtained were of the same order of accuracy as is given by the Dumas nitrogen method. The method can only be considered

superior to the latter when a correction is applied for the change in titre of the titanous chloride solution, and the amount of excess of the latter solution necessary for complete reduction is known. In the reduction of nitramines side reactions may take place in which the nitramino-group is completely removed from the nucleus.—W. S. M.

Anions; Indirect electrolytic estimation of — without platinum electrodes. E. Lasala. *Anal. Fis. Quim.*, 1919, 17, 235—247. (See also this J., 1919, 391 A.)

THE anion is precipitated as insoluble silver salt. This is dissolved in a suitable solvent, e.g., ammonia solution or potassium cyanide, and the solution is electrolysed with a nickel-plated copper cathode and any iron anode. The method gave satisfactory results with carbonate, oxalate, ferro- and ferricyanide, and thiocyanate, but was found unsuitable for the estimation of orthophosphate, arsenate, and chromate. The method may be used in the separation of chloride and iodide. The precipitate of mixed silver salts is washed with ammonia solution (sp. gr. 0.92) and the chloride solution electrolysed directly. The residual silver iodide is dissolved in 20% potassium cyanide solution and electrolysed. A correction must be made for the solubility of silver iodide in ammonia.—W. S. M.

Toluene. Launsden. See III.

Identification of dyes. Hitch and Knapp. See IV.

Dyestuffs. Watkins. See IV.

Starch in paper. Kamm and Tendick. See V.

Ammonia. Allen and Davisson. See VII.

Hydrogens. Reedy. See VII.

Mercury compounds. Denigès. See VII.

Litharge. Morgan. See VII.

Sulphur. Bjerregaard. See VII.

Sulphur and chromium in steel. Goldenberg. See X.

Hydrogenated whale oil. Battenburg and Angerhausen. See XII.

Tannin analysis. Baldracco. See XV.

Tannin analysis. Parker. See XV.

Alkali sulphides. Bennett and Bennett. See XV.

Cascia. Browne. See XV.

Potassium in soils. Walker. See XVI.

Syrups and molasses. Owen. See XVII.

Reducing sugars. Beyersdorfer. See XVII.

Sugars. Schowalter. See XVII.

Dextrose and levulose. Lucius. See XVII.

Moisture in malt. Ward. See XVIII.

Acidity of worts etc. Windisch and Dietrich. See XVIII.

Abnormal milk. Baker and Van Slyke. See XIXa.

Keeping quality of milk. Baker and Van Slyke. See XIXa.

Foodstuffs and fodders. Herter. See XIXa.

Carbon monoxide. Lamb and Larson. See XIXb.

Alkaloids. Taigner. See XX.

Pepsin. Northrop. See XX.

Saccharin and benzoic acid. Schowalter. See XX.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

- Beyer. Ring furnace and drying plants. 1453. Jan. 16.
Broadbridge. Ball mills etc. 2227. Jan. 23.
Bury, and Skinningrove Iron Co. Drying washed mineral matter. 2180. Jan. 23.
Collis Products Co. Apparatus for desiccating liquids. 1800. Jan. 20. (U.S., 13,12,15.)
Conder and Vivian. 2212, 2243, and 2245. See X.
Davis. Apparatus for drying or cooling powdered etc. substances. 2271. Jan. 24.
Dunker. Apparatus for treating air or other gas. 1469. Jan. 16.
Fesca und Sohn. Centrifuges. 978 and 979. Jan. 12. (Ger., 26,6,15 and 18,6,19.)
Frühwacht (Neumann and Neumann). Kibbling or grinding mills. 964. Jan. 12. (Ger., 9,10,16.)
Hirsch. Apparatus for generating gas by action of liquids on solid etc. reagents. 1176. Jan. 14.
Hutton. Process of drying. 940. Jan. 12.
Morshead. Cooling towers. 1606. Jan. 17.
Ten Bosch. Filtering or de-watering presses. 1678. Jan. 19.

COMPLETE SPECIFICATIONS ACCEPTED.

- 1940 (1919). Levy. Collection and recovery of volatile solvents evaporated into the atmosphere. (137,615.) Jan. 28.
2858 (1919). Kruger. Apparatus for drying granular and other substances. (137,631.) Jan. 28.
3582 (1919). Soder. Grinding mills. (137,640.) Jan. 28.
4856 (1919). Savy. Drying apparatus. (123,984.) Jan. 28.
6175 (1919). Tullis. Process and apparatus for drying. (137,427.) Jan. 21.
8797 (1919). Wallace and Tiernan. Packing materials and processes of making the same. (131,871.) Jan. 21.
13,436 (1919). Hellner. Rotary digesters and like boilers. (137,742.) Jan. 28.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

- American Coke and Chemical Co. Coke-ovens etc. 2316—2324, 2326. Jan. 24. (U.S., 3,1, 22,6, 15,9,16; 9,11,17; 11,7 and 8,9,19.)
Burton, and South Metropolitan Gas Co. Gas-producers. 1808. Jan. 20.
Byrnes. 2075. See XX.
Carmichael. 1932. See XII.
Castelli and Corthesy. Means to generate gases from coal etc. 2060. Jan. 22.
Coke Oven Construction Co., and Marr. Coke-ovens. 1512. Jan. 16.
Duckham. Manufacture of mixtures of pulverised fuel with tar etc. 2315. Jan. 24.
Fabry. Purification of coal gas. 2050. Jan. 22.
Gair, and South Metropolitan Gas Co. Incandescent gas mantles. 1392. Jan. 15.
Gledhill. Coal-carbonisation retorts. 1886. Jan. 21.
Grocott. Fuel-gas producer. 1263. Jan. 15.
Grocott. Combined gas-producer and furnace. 1407. Jan. 16.
Hudson. Method of making decolorising-carbon. 1846. Jan. 20. (U.S., 15,2,19.)

International Coal Products Corporation. Treating lignites. 1576. Jan. 17. (U.S., 30,4,19.)
Leadbeater. Manufacture of artificial fuel. 1550. Jan. 17.

Lloyd. Peat fuel. 1950. Jan. 21.
Morris. Gas-generating apparatus. 2333. Jan. 24.

COMPLETE SPECIFICATIONS ACCEPTED.

- 18,984 (1918). Rappaport. Manufacture of acetone by dry distillation of pyrolignite of lime and other acetates. (137,558.) Jan. 28.
261 (1919). Swinburne. Low temperature destructive distillation of coal. (137,572.) Jan. 28.
4010 (1919). West and Wild. Gas-producers. (137,647.) Jan. 28.
5075 (1919). McMinn. See VIII.
9893 (1919). Atkinson, and Powdered Fuel Plant Co. Combustion of powdered fuel. (137,450.) Jan. 21.
19,727 (1919). Szekey. Producing cakes of solid crude paraffin residue. (131,293.) Jan. 28.

IV.—COLOURING MATTERS AND DYES.

APPLICATION.

Oates. Manufacture of dyestuffs. 1393. Jan. 15.

COMPLETE SPECIFICATION ACCEPTED.

11,891 (1919). Imray (Soc. Chem. Ind. in Basle). Manufacture of azo dyestuffs and intermediate products. (137,733.) Jan. 28.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

- Bonwitt. Manufacture of dissolved or gelatinised cellulose esters. 1597. Jan. 17. (Ger., 6,6,17.)
Braun. Manufacture of cellulose. 999. Jan. 12. (Ger., 5,8,16.)
Deutsche Celluloid Fabrik. Manufacture of cellulose compounds. 2120. Jan. 22. (Ger., 9,1,18.)
Fletcher and Ward. Recovering useful products from waste celluloid. 1596. Jan. 17.
Frood. Fibrous fabrics or compositions for frictional and wearing purposes. 1426. Jan. 16.
Great Northern Paper Co. Paper-making. 1125. Jan. 13. (U.S., 25,10,18.)
Hashimoto. Obtaining new fibre from Sugamo seaweed. 1116. Jan. 13.
Jenkins. Machinery for drying, cleansing, or carbonising wool etc. 1279. Jan. 15.
Knibiehler. Treating silk. 1442. Jan. 16.
Naefe. Protecting woollen fabrics from moths. 1355. Jan. 15.
Naefe. Dressing and waterproofing textile fabrics. 1356. Jan. 15.
Stulemeyer. Treatment of viscose. 2056. Jan. 22.
Wells. Testing paper, board, textiles, etc. 1930. Jan. 21.

COMPLETE SPECIFICATION ACCEPTED.

15,007 (1919). Hollis and Woodmansey. Cleansing wool etc. in the raw state or in any stage of manufacture. (137,747.) Jan. 28.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATION.

Schwarzkopf. Bleaching, cleaning, and decolorising by bleaching-earths. 2108. Jan. 22. (Ger., 25,10,18.)

COMPLETE SPECIFICATIONS ACCEPTED.

9532 (1919). Lomax. Finishing or lustring textile fabrics. (137,710.) Jan. 28.
15,611 (1919). Textilmaschinen Fabrik A.-G. Automatic yarn bank-dyeing machine. (128,586.) Jan. 28.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

- American Coke and Chemical Co. Ammonia saturators. 2325. Jan. 21. (U.S., 17,415.)
 Burt (Becker). Manufacture of oxides of phosphorus and phosphoric acid. 1709. Jan. 19.
 Chance and Hunt, Gidden, and Ragg. Manufacture of oxide and carbonate of zinc. 2007. Jan. 21.
 Commis. Production of ammonia. 2002. Jan. 21.
 Dutt and Dutt. Manufacture of sodium carbonate. 2232. Jan. 21.
 Dutt and Dutt. Manufacture of sodium hydrate, alumina, and calcium fluoride. 2233. Jan. 23.
 Dutt and Dutt. Manufacture of soluble potassium salts from feldspar. 2234. Jan. 23.
 Frischer. Process of vaporising dilute nitric acid etc. 1012. Jan. 12. (Austria, 31,816.)
 Head. Manufacture of chromic oxide and sodium sulphide from sodium chromate. 1346. Jan. 15.
 Jones and Kelly. Production of boric acid from sodium pentaborate. 2053. Jan. 22.
 Mehta. Manufacture of potassium chromate and calcium chromate from chromium ores, feldspar, and gypsum. 2025. Jan. 22.
 Mehta. Manufacture of calcium chromate from chromium ores. 2026. Jan. 22.
 Sieurin. Producing aluminium oxide from aluminium chloride. 1711 and 1712. Jan. 19.
 Soc. Ind. de Prod. Chimiques. Isolating or purifying alkali chromates. 1997. Jan. 21. (Fr., 2,817.)
 Wildman. Extraction of alumina and its salts from clay. 1231. Jan. 14.

COMPLETE SPECIFICATIONS ACCEPTED.

- 1383 (1918). Adams and Greenwood. Manufacture of hydrogen. (137,340.) Jan. 21.
 3607 (1918). General Chemical Co. Production of ammonia from atmospheric nitrogen. (120,546.) Jan. 21.
 13,596 (1918). Curphey. Production of ammonium salts. (137,345.) Jan. 21.
 1730 (1919). Matheson. Recovering acetic acid and manufacturing acetates. (137,388.) Jan. 21.
 2042 (1919). Higgins, and United Alkali Co. Manufacture of caustic potash. (137,632.) Jan. 28.
 8552 (1919). Leach, and United Alkali Co. *See XX.*
 16,021 (1919). Norsk Hydro-Elektrisk Kvaestof-aktieselskab. *See XVI.*

VIII.—GLASS; CERAMICS.

APPLICATIONS.

- Corning Glass Works. Heat treatment of articles. 1607. Jan. 17. (U.S., 17,319.)
 Laurie, Ormandy, and Osmosis Co. Refractory articles made from china clay, and method of manufacture. 2077. Jan. 22.

COMPLETE SPECIFICATIONS ACCEPTED.

- 4956 (1919). Mathy. Machine for re-melting glass articles. (137,660.) Jan. 28.
 5075 (1919). McMinn. Glazing-cement for use in fireclay retorts and furnaces. (137,419.) Jan. 21.
 17,560 (1919). Harrison. Abrasive material, and process of making same. (137,490.) Jan. 21.

IX.—BUILDING MATERIALS.

APPLICATIONS.

- Dutt and Dutt. Manufacture of Portland cement from feldspar residues. 2231. Jan. 23.
 Fish. Seasoning, sterilising, and drying wood. 1285. Jan. 15.
 Fish. Manufacture of wood. 1286. Jan. 15.
 Jones. Manufacture of fireproof composition for building slabs, tiles, etc. 2080. Jan. 22.

Norton Co. Manufacture of flooring compositions etc. 2294, 2295. Jan. 21. (U.S., 1 and 14,219.)

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

- Ashcroft. Electrolytic production of magnesium from anhydrous magnesium chloride. 1981. Jan. 21.
 Ballantine. Production of metallic alloys. 1137. Jan. 13.
 Bury, and Skinningrove Iron Co. 2180. *See I.*
 Coles. Coating steel and iron with brass. 1142. Jan. 13.
 Coles. Apparatus for Sherardising. 1143. Jan. 13.
 Conder and Vivian. Crushers or mills for stone or ores. 2242, 2243, 2245. Jan. 21.
 Durant and Vautin. Smelting of zinc. 2310. Jan. 24.
 Goslett, and Metals Extraction Corporation. Extraction of tin from its ores. 2226. Jan. 23.
 Honhorst. Steel alloys, and process of making same. 1829. Jan. 20. (U.S., 8,12,17.)
 International Nickel Co. Manufacture of nickel and copper. 1013. Jan. 12. (U.S., 5,2,19.)
 Macdonald. Solder for aluminium or its alloys. 2272. Jan. 24.
 Picard and Sulman. Cementation agent for copper solutions. 1924. Jan. 21.

COMPLETE SPECIFICATIONS ACCEPTED.

- 278 (1919). Fuller, and Fuller Engineering Co. Steel-smelting furnaces. (137,574.) Jan. 28.
 781 (1919). Weyman. Cleaning blast-furnace and like gases. (137,586.) Jan. 28.
 1150 (1919). Weyman. Apparatus for cooling blast-furnace gases. (137,378.) Jan. 21.
 2566 (1919). Hills and Wheeler. Manufacture of ore briquettes. (137,626.) Jan. 28.
 2705 (1919). Wood and Wood. Cupolas. (137,399.) Jan. 21.
 4665 (1919). Oates and Green, and Ashton. Acid or like baths for treating wire. (137,414.) Jan. 21.
 7617 (1919). Rondelli. Oxidising iron or steel surfaces. (137,436.) Jan. 21.
 8023 (1919). Harris. Coating metal on metal or other conductive flat surfaces. (137,695.) Jan. 28.
 10,131 (1919). Norsk Hydro-Elektrisk Kvaestof-aktieselskab. Metallurgical reduction processes. (126,951.) Jan. 21.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

- Ashcroft. 1981. *See X.*
 Hepburn. Electrolysers. 1475. Jan. 16.
 King. Electric dry battery. 1089. Jan. 13.
 Oldham, and Oldham and Son. Galvanic batteries. 1025. Jan. 12.

COMPLETE SPECIFICATIONS ACCEPTED.

- 12,567 (1918). Benjamin. Electrolytic apparatus. (137,553.) Jan. 28.
 1732 (1919). Matheson and Kaelin. Electrolytic cells. (137,609.) Jan. 28.
 3676 (1919). Van Raden and Co., and Smith. Electric storage cells. (137,641.) Jan. 28.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

- Broadbridge. Treatment of froths containing fatty matter etc. 1708. Jan. 19.
 Brooker and Royle. Manufacture of soap. 1716. Jan. 19.
 Carmichael. Purifying and clarifying oil. 1932. Jan. 21.
 Niessen. 1113. *See XIX.*

Nordiske Fabriker De-No-Fa Akt. Catalysts for hydrogenation of unsaturated organic compounds. 1126. Jan. 13. (Ger., 17.3.19.)

Nordiske Fabriker De-No-Fa Akt. Treatment of fish oils. 1129. Jan. 13. (Ger., 18.3.19.)

Schwarzkopf. 2108. See VI.

Verein. Chem. Werke. 1841, 1974—7. See XVIII.

Warburton. Treatment of oils and fats. 1016. Jan. 12.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Birkby and Birkby. Preparation of phenolic aldehyde condensation products. 1034. Jan. 13.

Chance and Hunt, and others. 2007. See VII.

Dunham. Solubilised Karaya gum composition. 1814. Jan. 20.

Hyslop. Printing, and inks etc. for use therein. 1782. Jan. 20.

Judge and Shapiro. Protecting and preserving composition or paint. 1601. Jan. 17.

Melamid. Manufacture of hard resin-like substances. 1241. Jan. 14. (Ger., 13.5.19.)

COMPLETE SPECIFICATION ACCEPTED.

14,346 (1919). Wright. Paints. (137,475.) Jan. 21.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Davidson. Treatment of rubber latex. 1690, 1691. Jan. 19.

Minton. Rubber-proofed fabrics and articles of india-rubber. 1738. Jan. 20.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Hatschek and Schidrowitz. Liquid glue, and process of making same. 2148. Jan. 23.

Heyl. Manufacture of glazed, varnished, or japanned leather. 2332. Jan. 24. (Ger., 18.9.15.)

Niessen. 1113. See XIX.

XVI.—SOILS; FERTILISERS.

COMPLETE SPECIFICATION ACCEPTED.

16,021 (1919). Norsk Hydro-Elektrisk Kvaelfstof-aktieselskabet. Ammonium nitrate fertilisers. (129,974.) Jan. 21.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

Barbet et Fils et Cie. Manufacture of grape honeys and fruit syrups. 1972. Jan. 21. (Fr., 31.10.19.)

Hudson. 1846. See II.

Kowalski. Purification of syrups and wastes in manufacture of sugar. 1247. Jan. 14. (Ger., 21.4.15.)

Verein. Chem. Werke. 1841, 1974—7. See XVIII.

COMPLETE SPECIFICATIONS ACCEPTED.

16,678 (1917). Daniel. Producing caramel. (137,339.) Jan. 21.

1891 (1919). Stewart and Co., Durham, and Wishart. Handling and storing sugar cane in sugar factories. (137,614.) Jan. 28.

15,766 (1919). Marks (Industrial Apparatus Corporation). Defecation of sugar solutions. (137,750.) Jan. 28.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Goldschmidt and Migeon. Treatment of peat, distillers' wash, etc. 2057. Jan. 22.

Verein. Chem. Werke. Manufacture of glycerol from sugar. 1841, 1974—1977. Jan. 20 and 21. (Ger., 12.4.15; 22.4 and 19.5.16; 18.6.17.)

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

By-Products Recovery Co. Treatment of material. 2211. Jan. 23. (U.S., 19.7.15.)

By-Products Recovery Co. Treatment of milk etc. 2212. Jan. 23. (U.S., 11.4.16.)

Mapleton. Food preparation. 1883. Jan. 21.

Niessen. Boiling and drying offal etc. 980. Jan. 12. (Ger., 11.5.15.)

Niessen. Apparatus for boiling and drying organic substances. 1112, 1114, 1115. Jan. 13. (Ger., 20.7.16; 2.1 and 12.5.17.)

Niessen. Apparatus for treating mixtures of fat and glue-water derived from boiling and drying organic substances. 1113. Jan. 13. (Ger., 2.10.16.)

Thomson. Preparation of non-toxic foods and extracts from vegetable and animal matter. 1941. Jan. 21.

White (Collis Products Co.). Desiccating butter-milk etc. 1135. Jan. 13.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Byrnes. Making aldehyde fatty acids from mineral oils and their distillates. 2075. Jan. 22. (U.S., 22.1.19.)

Ellis (Soc. Chim. des Usines du Rhône). Manufacture of oxaldehydes and their derivatives. 1140. Jan. 13.

Nordiske Fabriker De-No-Fa. 1126. See XII.

Thomson. Preparation of detoxicated vaccines. 1940. Jan. 21.

Thomson. 1941. See XIX.

Thomson. Detoxication of tubercle bacillus etc. for preparation of vaccines. 2196. Jan. 23.

COMPLETE SPECIFICATIONS ACCEPTED.

16,092, 16,094—6 (1918). Rockefeller Institute for Medical Research. Aromatic arsenical compounds. (120,381, 120,383—5.) Jan. 21.

2376 (1919). Jackson (Citro Chemical Co. of America). Manufacture of citric acid. (137,396.) Jan. 21.

8552 (1919). Leach, and United Alkali Co. Manufacture of acetic anhydride. (137,701.) Jan. 28.

14,549 (1919). Soc. Chim. des Usines du Rhône. Manufacture of esters of ethylenic halogenhydrins. (128,911.) Jan. 21.

23,881 (1919). Wilde. Manufacture of aliphatic nitrites. (133,304.) Jan. 21.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Donisthorpe, and Dye Impression Photos, Ltd. Photographic printing. 1245. Jan. 14.

Möller. De-oiling kinematograph etc. films. 961. Jan. 12. (Ger., 2.9.16.)

COMPLETE SPECIFICATIONS ACCEPTED.

8089 (1919). Portass, Portass, and Portass. Development and chemical treatment of photographic films. (137,703.) Jan. 28.

21,263 (1919). Wieland. Manufacture of colour screen plates or films for photography in natural colours. (137,502.) Jan. 21.

XXII.—EXPLOSIVES; MATCHES.

APPLICATION.

Friederich. Manufacture of detonating-caps. 1715. Jan. 19. (Ger., 27.7.18.)

XXIII.—ANALYSIS.

COMPLETE SPECIFICATION ACCEPTED.

14,030 (1917). Adams, Bousfield, and Todd. Method and apparatus for detecting water vapour or either of the gaseous components thereof. (137,547.) Jan. 28.

I.—GENERAL; PLANT; MACHINERY.

Steam boiler plants; Exact data on the running of —. No. III. The amount of steam used by steam jets. D. Brownlie. *Engineering*, 1920, 109, 71—74.

Of 250 boiler plants investigated 93, or 37%, were fitted with steam jet blowers. It was found that the steam used by the blowers varied from as little as 1% to 20% of the total steam produced by the plant, the average being 6.6% for hand-fired and 6.7% for mechanically-stoked boilers. The steam consumption of properly arranged blowers should not exceed 3 to 4%, faulty design or careless manipulation accounting for the balance, though in many cases it would be better to replace the steam jets by mechanical draught, which on an average takes about 2½% of the steam produced.—W. H. C.

Vacuum-evaporator plant; Air-pump capacities and incompressible-gas volumes in industrial —. E. Corner. *Engineering*, 1920, 109, 74—75.

FORMULÆ, tables, and three sets of curves are given to enable the air-pump capacity to be easily computed. The temperature prevailing in the condenser is approximately that of the outgoing or "tail" water, and this temperature governs the vacuum attainable. In general power practice the allowance for ratio of air to steam is much smaller than is practicable with industrial vacuum evaporators, which have a greater joint surface and are often dealing with liquids which give off permanent gases. The following figures for air introduced into the system, though calculated for sugar-juice, are applicable to most chemical liquids:—4 lb. of air per 1000 lb. of steam or vapour; 0.2 lb. of air per 1000 lb. of liquor treated; 0.1 lb. of air per 1000 lb. of injector water. It should be noted that the quantity of air per 1000 lb. of liquor treated varies with the number of vessels in the evaporator system.—W. H. C.

Absorption tower packing; Resistance of — to gas flow. F. C. Zeisberg. *Amer. Inst. Chem. Eng.*, Dec., 1919. *Chem. and Met. Eng.*, 1919, 21, 765—767.

THE resistance of various forms of packing was measured in an experimental tower, 30 ins. in diam., and 15 ft. high, and the superiority of artificial packings (corrugated diaphragm rings, corrugated spiral rings, Hechenbleikner blocks, chemical ware tile, and Raschig rings) over pumice, quartz, and coke was demonstrated. The general law of the flow of fluids was found to hold experimentally, and expressing the frictional resistance as the pressure necessary to produce a given flow, an equation, $p = -\frac{f h v^2}{a^2}$ is given, in which p is the pressure in inches of water, h is the height of packing in feet, v is the volume of gas passing in cub. ft. per min., a is the cross-sectional area of packing in sq. ft., and f is a coefficient differing in value according to the nature of the packing. The characteristics of various packings are shown in tabular form; the rapid increase in resistance with decreasing size of material is marked, as is also the greater resistance due to the wetting of the surface of small-sized packing material. For any given size of packing, manufactured shapes give much more surface and greater free space than do the older forms of dumped coke or quartz.—C. A. K.

Absorption of gases; New method of —. I. Moscieki. *Chim. et Ind.*, 1919, 2, 1303—1316.

AN improved type of absorption plant, especially applicable to the process of manufacture of nitric

acid from atmospheric nitrogen, has been constructed for the Soc. anon. pour l'Industrie de l'Aluminium at Chippis, Switzerland, and a similar plant for experimental purposes has been erected at Fribourg University. A description of the latter is given. It comprises two tower sections, the upper being empty and serving for the oxidation of the nitrous gases, whilst the lower is used for absorption purposes. This latter consists of two concentric tubes placed in an outer vessel, the space between them being filled with quartz fragments of 0.3—0.4 mm. in the upper part and with quartz of larger size in the lower part, which is kept below the level of the liquid. Both tubes have oblique apertures, 1 cm. diam., distributed over their surfaces. There is an outlet for the gas near the top and two outlets near the base, one for drawing off the acid and the other communicating with an air-lift. The absorbent enters a distributor through a pipe at the top, and another opening serves for admission of compressed air and liquid. The column of absorbent liquid is not high, and therefore never impedes the flow of gas. The gases are caused to enter the top segment and, after oxidation, flow into the inner tube of the lower segment, whence they pass in a horizontal direction into the intermediate space and eventually to the outlet. This horizontal flow ensures good absorption even with moderately high columns, the packing material being continuously irrigated. The conditions for the best absorption and the requisite velocity of the liquid were studied, and are summarised as regards the experimental plant.—W. J. W.

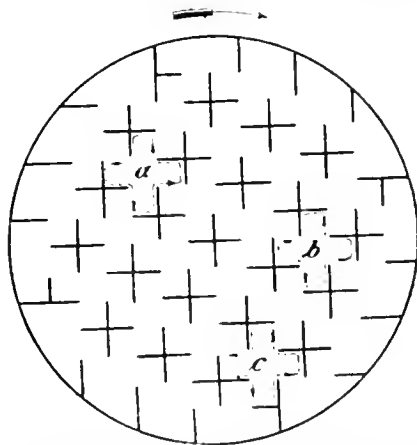
Lubricants. Frank. *See* IIA.

Handling barium products. Zimmer. *See* VII.

PATENTS.

Drying materials; Drums for —. A. Gerlach. Nordhausen, Germany. *Eng. Pat.* 133,327, 3.10.19. (Appl. 21,228/19.) *Int. Conv.*, 10.1.18.

A REVOLVING drum dryer is provided with longitudinal bars of cruciform section (see fig.) placed



in such relative positions that the material to be dried falls freely four times during each revolution, as shown by the arrows at *a*, *b*, *c*.—B. M. V.

Atomizing materials in a melted state; Process and apparatus for —. E. Odum. Paris. *Eng. Pat.* 122,403, 17.12.18. (Appl. 21,085/18.) *Int. Conv.*, 18.1.18.

THE molten material (e.g., metals, chemical products, vitreous substances) is heated by the com-

bustion under pressure, in a suitable chamber, of solid, liquid, or gaseous fuel; the products of combustion are then allowed to flow through a nozzle to the centre of which the molten material is supplied. The sudden reduction in pressure and expansion of the gases causes atomisation of the material under treatment. The combustion chamber may be divided into two communicating parts, one of which forms the combustion chamber proper and the other forms the chamber for heating the material and discharging the gases.—B. M. V.

Drying ovens; Vacuum — J. D. Taylor, Kingston Hill. Eng. Pat. 136,703, 7.2.19. (Appl. 3065/19.)

A HEATING shelf or chest for drying stoves or ovens is constructed of flattened tubular members, spaced apart horizontally and welded or otherwise jointed at each end to vertical tubular supporting members through which the heating medium is supplied and discharged. The major axes of the horizontal members are substantially parallel to the surfaces of the shelves and normal to the supporting members.—W. H. C.

Dryer. E. C. Horst, San Francisco, Cal. U.S. Pat. 1,325,848, 23.12.19. Appl., 23.4.18.

A RECTANGULAR casing is divided by parallel vertical partitions into vertical chambers. The partitions are spaced alternately from the top and bottom of the casing to provide communications between the chambers. Horizontal partitions are provided in each chamber with openings at opposite ends alternately, so as to provide a zigzag passage through the chamber. Air is passed through the chambers in series, and means are provided for reversing the direction of flow when required. Heating devices are provided in the chambers to increase the temperature of the air during its passage.—W. F. F.

Lifting and forcing apparatus for solids and semi-solids; Fluid and/or air direct pressure — S. H. and C. H. Adams, York. Eng. Pat. 136,583, 3.7.18. (Appl. 10,916/18.)

A DISPLACEMENT ejector is provided with inlet and outlet valves and with connections by means of which compressed air or other suitable fluid is delivered into the space above the material in the ejector to force the contents up the rising main and also into the body of the material itself and into the filling and discharge ducts to assist in moving the breeze, sand, or other solid and semi-solid materials which the ejector is designed to deal with. A controllable variable clockwork mechanism or a regulated electric motor allows the pressure fluid to enter the ejector during a predetermined period and then for another predetermined period the pressure fluid is shut off and the exhaust is opened.—W. H. C.

Shaft-kilns for burning or roasting non-sintering substances; Gas-fired — A. and W. W. Steiger, Zurich, Switzerland. Eng. Pat. 136,588, 11.10.18. (Appl. 16,758/18.)

IN a vertical kiln which is provided with one or more sudden increases in diameter below the combustion zone in order to loosen the material so that air for combustion may be forced through the hot burnt charge, provision is made for the continuous withdrawal of the burnt and cooled material by means of a device consisting of sloping reciprocating platforms placed under hopper-shaped guides at the bottom of the furnace.—B. M. V.

Feeding furnaces for boilers and other purposes with solid fuel; Means for — Merz and McLellan, London. A. C. Michie, Newcastle-on-Tyne, and E. G. Weeks, Monkseaton. Eng. Pat. 136,867, 20.11.18. (Appl. 19,082/18.)

THE flue gases from boilers etc., after they have

been deprived of most of their heat by economisers or the like, are passed through the solid fuel immediately before it enters the combustion chamber, the fuel being supported on a perforated grate or conveyor, separate from or combined with the combustion grate.—B. M. V.

Refrigerating systems. E. G. E. Langbert, Stockholm, Sweden. Eng. Pat. 136,602, 10.12.18. (Appl. 20,554/18.)

A VESSEL containing a suitable cold accumulator (e.g., brine) is surrounded by a suitable liquid refrigerating agent that can be vaporised by reduction of pressure (e.g., sulphur dioxide or ethyl chloride), contained in an outer vessel which may be ribbed to increase the cold-radiating surface. During working hours a vacuum is applied to the refrigerating medium, more of the liquid being admitted as required, whereby heat is absorbed from the outer atmosphere, also from the cold accumulator. During idle hours the cold accumulator absorbs heat, the refrigerating liquid serving merely to transmit heat from the outer atmosphere to the inner vessel.—B. M. V.

(A) (c) *Refrigerant and process of making the same*. (b) *Refrigerating gas and process of making the same*. A. G. Crawford, Assignor to H. W. Seaman, Chicago, Ill. U.S. Pats. (A) 1,325,665, (b) 1,325,666, and (c) 1,325,667, 23.12.19. Appl., 5.2.18, 25.2.18, and 14.10.18.

(A) BUTANE and propane are mixed in such proportions that the mixture has a boiling point below 1° C., and the mixture is then liquefied by pressure. (b) The refrigerant is a mixture of liquid sulphur dioxide and a liquefied hydrocarbon gas. (c) A gas of the ethylene series is mixed with propane in such proportions that the boiling point of the mixture is suitable for refrigeration, and the mixture is then liquefied by pressure.—W. F. F.

Evaporating or concentrating liquids; Apparatus for — E. Shaw, London. Eng. Pat. 136,651, 19.12.18. (Appl. 21,286/18.)

TO prevent the accumulation of deposit on the walls of the apparatus, which consists of an inner tube having a helix formed on its exterior and which is rotated concentrically within another tube having an external jacket, scrapers of spring steel are fitted to the edges of the helix. The liquid to be evaporated passes through the spiral passage formed by the helix.—W. H. C.

Mixing, circulating, and agitating materials in a liquid or semi-liquid state; Apparatus for — W. J. Coles, and E. Allen and Co., Ltd., Sheffield. Eng. Pat. 136,758, 19.5.19. (Appl. 12,599/19.)

AN agitator consisting of vertical frames rotates about a vertical shaft within a tank. A system of compressed air or gas supply pipes is carried by the agitator, which extends to the bottom of the tank. The system consists of horizontal pipes secured to the upper part of the agitator by which compressed air is supplied to vertical pipes, spaced at intervals, and having their open lower ends supported by slotted plates carried on the lower part of the framework.—W. H. C.

Chemical reactions; Method of and apparatus for carrying out certain — T. Hughes, London. Eng. Pat. 136,896, 21.12.18. (Appl. 21,452/18.)

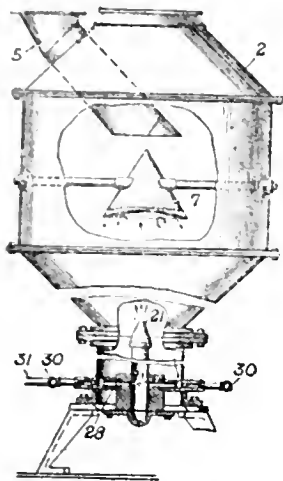
IN reactions in which a solid is treated with a liquid, the solid dissolving with the evolution of gas, the reaction becomes very slow when the dissolving power of the liquid is nearly exhausted. To overcome this difficulty the solid, broken into pieces of a convenient size, is contained in a tall, narrow vessel and the liquid is admitted at the

bottom from an overhead tank. The flow of liquid is so adjusted that while the lower half of the column is bathed in liquid the upper half is filled with a froth of nearly exhausted liquid and gas. The liquid part of the froth comes into intimate contact with fresh solid material and becomes completely exhausted, and is then discharged along with some of the lighter portions of the solid through one or more overflows into a settling tank, where the gas, liquid, and mud are separated.

—W. H. C.

Pulveriser. D. McKelvey, Chicago, Ill. U.S. Pat. 1,325,676, 23.12.19. Appl. 1.4.19.

MATERIAL is fed by the conduit, 5, into the chamber, 2, in which an impact member, 7, having a dish under surface is suspended. Air under



pressure is supplied by the pipes, 30, 31, and passes into the central jet nozzle, 21, so that a jet of air mixed with the material to be pulverised is directed against the impact member, 7. Inlet openings are provided on the upper sides of the tubes, 28, so that air is drawn through them into the main air-jet and a circulation of air is maintained.—W. F. F.

Heating apparatus. W. C. Merrill, Assignor to The Merrill Process Co., Boston, Mass. U.S. Pat. 1,325,952, 23.12.19. Appl. 14.2.16.

HEATING fluid is circulated by a mechanical device operated by an electric motor, and means are provided for heating the fluid during its circulation. The fuel supply to the heater is controlled by a valve which is operated by the same electric circuit that operates the motor of the circulating device, so that the valve automatically closes when the motor is stopped and an alarm is simultaneously operated. The fuel valve is adapted to be opened automatically when the motor is re-started. (See also Eng. Pat. 128,709; this J., 1919, 612 A.)

—W. F. F.

Wrought-iron apparatus and vessels; Method for increasing the resistance of — to chemical action. F. Schüller, Frankfort. Ger. Pat. 314,824, 12.7.18.

FRAGMENTS of natural or artificial stones are worked into the iron by kneading, pressing, or rolling, and the surface is then coated with a thin, acid-resisting layer of the powdered stone. (See also Ger. Pat. 288,572; this J., 1916, 365.)

—J. S. G. T.

Delivering materials in fractions of equal weight; Apparatus for —. F. P. Martin, Paris. Eng. Pat. 136,948, 1.2.19. (Appl. 2588/19.)

Oil-fuel burners for furnaces and the like. J. Simpson and Sons (Bolts and Nuts), Ltd., Darlaston, and G. F. Everton, Wednesbury. Eng. Pat. 137,173, 7.3.19. (Appl. 5661/19.)

Separating oil or grease from water; Apparatus for —. J. H. Carruthers and Co., Ltd., J. Gourlay, C. M. Jackson, G. K. Johnstone, and J. Young, Glasgow. Eng. Pat. 137,213, 30.1.19. (Appl. 10,691/19.)

Drying apparatus. G. D. Harris, New York. Eng. Pat. 136,631, 17.12.18. (Appl. 21,060/18.)

SEE U.S. Pat. 1,282,822 of 1918; this J., 1919, 275 A.

Heat-exchanging apparatus. A. D. Harrison, Marple. U.S. Pat. 1,325,637, 23.12.19. Appl. 9.7.18.

SEE Eng. Pat. 116,786 of 1917; this J., 1918, 398 A.

Ether and alcohol vapours contained in the air of factories; Process and apparatus for the recovery of —. E. A. Barbet, Paris. U.S. Pat. 1,326,432, 30.12.19. Appl. 4.10.16.

SEE Eng. Pat. 101,723 of 1916; this J., 1918, 45 A.

Muffle-furnace. T. W. S. Hutchins, Middleswich. U.S. Pat. 1,325,674, 23.12.19. Appl. 15.10.18.

SEE Eng. Pat. 120,633 of 1917; this J., 1919, 1 A.

Absorbed substances; Process for separation of —. B. Schwerin, Assignor to Ges. für Elektro-Osmose, Frankfurt, Germany. U.S. Pat. 1,326,105, 23.12.19. Appl. 4.1.13. Renewed 17.10.19.

SEE Eng. Pat. 24,666 of 1912; this J., 1913, 432.

Chemical reactions. Ger. Pat. 304,479. See XI.

Electrical purification of gases. Ger. Pat. 314,775. See XI.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Forms in which sulphur occurs in —. A. R. Powell and S. W. Parr. Bull. Eng. Expt. Stat. Illinois Univ. Gas J., 1920, 149, 79.

THE sulphur of coal occurs in four characteristic forms, two organic and two inorganic. The "resinic organic" type is present in the portion of the organic material soluble in phenol. The "humus organic" type of compounds are shown to be closely related to humus substances by their chemical reactions. The pyritic or marcasitic sulphur is present as FeS_2 , as indicated by the iron-sulphur ratio when the pyrites is oxidised and dissolved. The sulphate sulphur is characterised by solubility in dilute hydrochloric acid without preliminary oxidation. Free sulphur was not found in any of the coals tested, and is presumably absent except in unusual cases where it might occur in small quantities as a decomposition product of pyrites. Some of the sulphur, probably the pyritic, is gradually oxidised to the form of sulphate. The experimental data suggest that when coal is coked the sulphate sulphur is retained by the coke, but in some form other than inorganic sulphate, whilst the pyritic sulphur is partially volatilised, a portion being left in the coke, probably as sulphide. All the pyrites is decomposed. The resinic sulphur is left in the coke, but in a form different from that existing in the coal, and the humus sulphur partly volatilises, some remaining in the coke in a changed form. The analytical methods used are described in detail, and the results tabulated for different coals.—W. P.

Anthracite; Burning steam sizes of — with or without admixture of soft coal. W. P. Frey. U.S. Bureau of Mines, Tech. Paper 220, 1919. 8 pp.

A BOILER plant designed for bituminous coal and operating with natural draught is capable of using 20% of steam sizes of anthracite mixed with bituminous coal without material loss in efficiency and without change of equipment provided sufficient grate space is available. Larger proportions of anthracite may be used if a blower is added. The fineness of steam size anthracite necessitates a change of grate when the mixture contains over 40% of anthracite. Proper mixing is essential, and the coal must be fired evenly in small quantities. —W. P.

Arsa coal; Sulphur content of and yield of low-temperature tar from —. F. Fischer. Ges. Abhandl. zur Kenntnis der Kohle, 1918, 3, 98—101. Chem. Zentr., 1919, 96, IV., 977—978.

ESCHKA's method for estimating sulphur was found to be unsuitable for coal having a high sulphur content. For example, Arsa coal was found to contain 6.87% of sulphur by Eschka's method, whereas estimations by decomposition with nitric acid in a sealed tube gave 9.74—9.89%. The coal contained H₂O, 1.6%; ash, 11.4%; C, 65.94—66.14%; H, 4.83—5.02%; gave a coke residue of 55% in a crucible; benzene extract in a Soxhlet apparatus, 0.7%; benzene extract under pressure, 14.2%. In a distillation test with 10 kilos. in the rotary apparatus described previously (this J., 1919, 563A) the coal gave 1.5 kilos. of tar, 6.3 kilos. of coke, and 600—700 litres of inflammable gas. In a distillation test under ordinary conditions the coal (30 grms.) melted and gave 5.3 grms. of tar, of sp. gr. 1.02. The formation of a liquid tar and melting under heating show that Arsa coal is a true coal. The tar contains, however, in contrast to tars from most coals giving a high yield of low-temperature tar, only 10—15% of phenols.—L. A. C.

Gasification; Complete —. H. Strache. J. Gasbeuecht., 1919, 62, 709—713.

THE author discusses the advances in the supply and use of gas made by complete gasification processes during the past 25 years. The properties of water-gas—lower calorific value per cub. ft., lower air requirement, higher speed of inflammation, higher specific gravity, and non-luminous blue flame—render alterations necessary in the burners, and this has stood in the way of its more extended use. On the other hand the water-gas process possesses great advantages in its flexibility, small ground space required, and low cost of plant. Complete gasification may yield one of two kinds of gas. The first class, which includes producer gas, semi-water gas, and Mond gas, has a calorific value of 125—175 B.Th.U., and has a nitrogen content of 50% or over. The second class includes "Doppelgas" (double-gas), which has a calorific value of 360—390 B.Th.U., and contains 3—6% of nitrogen, and "Trigas" (a mixture of coal-gas, water-gas, and producer-gas), which has a lower calorific value and a higher nitrogen content than "Doppelgas." The gases obtained by complete gasification have a lower calorific value than coal gas made under pre-war conditions, but their use has many advantages (see this J., 1919, 707A). The supply of gas of high nitrogen content and low calorific value for long distance transmission is not recommended. The economy or otherwise of the recovery of by-products during complete gasification processes is being investigated by the Institut für Kohlenvergasung und Nebenproduktengewinnung in Vienna. —W. P.

Oil-gas manufacture; Experiments in connection with —. J. D. Ruys. Chem. Weekblad, 1919, 16, 746—780.

A SERIES of investigations has been made on the Rincker-Wolter process in which oil from petroleum residues is sprayed over hot coke, air and steam being admitted into the generators. Preliminary trials were carried out with Mexican Panuca crude oil, and a special method was devised to remove the sulphur from the gas produced, but other oils were found to be more suitable. The trials included investigations on the air-blast, the water-gas production, and the gasification of the oil. Extensive samples were taken of the gases at various stages, and determinations were made of the amount of water evaporated and the quantity of steam required for most effective working. An illuminating gas of 4800—5000 cals. was aimed at. The details of the trial runs and the results obtained are tabulated and plotted as curves. It was found that the yield of gas from the oil is proportional to the rapidity with which the products are removed. To ensure this, water-gas alone is insufficient and steam must be introduced. Hydrogen may be absorbed by the oil from the water-gas under certain conditions. No separation of carbon appears to take place.—W. J. W.

Producer-gas for motor vehicles. D. J. Smith. Inst. Automobile Eng., Jan. 8, 1920. Engineering, 1920, 109, 59—61, 92—95.

THE author has shown that producer-gas can be used to drive motor vehicles, and he claims the following advantages:—Home-produced fuels, some of which are even at present waste products, can be used. There is no fire risk. Producer-gas requires no labour for its production like coal-gas. No gas-bag is required. A vehicle can travel the same distance as on petrol without taking in fresh fuel. The gas is of uniform quality, so that mixture troubles are eliminated. It is the cheapest source of power for motor vehicles known at present, and with coal at 40s. per ton is equal to petrol at 2½d. per gall. when used in the same engine. A gas producer is lighter and smaller than a compressed gas installation. It requires no pressure, and is therefore free from danger. Against the above the following disadvantages must be considered. It takes from 15—20 mins. to start from cold when using a producer. The extra weight of the plant has to be taken into account. Periodical cleaning is necessary. From 3—4 mins. is required to start after a lengthy stop. There is an increase of 75% in weight of fuel, including water, to cover any given distance as compared with petrol. The following comparisons are given for a five-ton commercial lorry:—

	Steam	Petrol	Producer gas
Price of fuel	50s. per ton	3s. per gall.	55s. per ton
Cost in pence per gross ton mile	0.31	0.66	0.048
Cost in pence per net ton mile	0.75	1.20	0.091

The author's producer has been designed to do away with a thick fuel bed and to burn any volatile matter so completely that a bulky water scrubber for the gas will not be needed. The fuel bed is only about 6 in. deep and practically continuous feeding of fuel and discharge of ash are maintained by coupled feed and discharge valves. The fuel ignites at once, and the ash is not allowed to rest long enough to fuse into clinker. The only impurity in the gas is a little of the fine dust of the fuel carried over by the draught, and the gas is purified by passing it through a dry tubular scrubber heater where

the water for the producer is pre-heated. The scrubber is easily cleaned out and the producer itself is jacketed, air being passed through the jacket to the water saturator and thence to the producer. The whole apparatus weighs 2–3 cwt., and is compact enough to be carried on the dash-board of the car. The fuel (anthracite, coke, charcoal, or prepared peat) is carried in a bunker under the hood over the driver's seat, and is fed by gravity; the water is carried under the seat. Both vehicle and engine ought to be specially designed for use with producer-gas, but very fair results have been obtained with a petrol engine; the cost per gross ton mile in the same vehicle and engine was found to be 1.45d. with petrol against 0.0856d. with producer-gas. The producer-gas is better adapted to the larger and heavier commercial class of vehicles, and if generally adopted would set free petrol, benzol, etc. for the lighter pleasure cars. Experiments have shown that no danger is to be apprehended from excessive escape of carbon monoxide, even when the vehicle is standing. Anthracite in the form of "beans" which pass a $\frac{1}{4}$ - or $\frac{1}{2}$ -in. screen is the most satisfactory fuel, but coke may be used, although it contains a good deal more ash and sulphur and does not feed so easily. Charcoal is a good fuel but is bulky. Peat briquettes which have had the objectionable volatile matter driven off by heat are very satisfactory. The best results are obtained with a fuel bed of 6 in. for anthracite, 7 for coke, 4 for charcoal, and 2 or 3 in. for prepared peat.—W. H. C.

Alcohol or ether; Industrial process for the synthetic manufacture of — from the gases obtained in the distillation of coal. E. de Loisy. *Comptes rend.*, 1920, 170, 50–53.

LEBEAU and Damiens have shown that in the presence of catalysts such as vanadic, uranic, tungstic, or molybdic acids, sulphuric acid rapidly and completely absorbs ethylene (this J., 1913, 277), and it has since been shown by Damiens that the ethylene is absorbed as ethyl hydrogen sulphate. The author makes use of these two important facts in his process. The coal gas is bubbled through the acid at a concentration of 66° B., and by suitable subsequent dilution and distillation alcohol or ether may be obtained as required. The diluted acid serves for the preparation of ammonium sulphate. Since, however, twice as much acid is required for the absorption of ethylene as for the production of ammonium sulphate, a portion of the acid is concentrated by means of the waste heat from the gas coming from the coke ovens, and can thus be used either for the absorption of the ethylene or for the preliminary drying of the gases and the absorption of the traces of propylene, butylene, and acetylenic hydrocarbons present. By this method it is only necessary to replace the acid removed in the form of ammonium sulphate. (See also Bury, this J., 1920, 91 A).—W. G.

Carbon monoxide; Production of — in the flames of gases. A. Kling and D. Florentin. *Comptes rend.*, 1919, 169, 1401–1406.

A NUMBER of samples of gas, some industrial and some synthetic, containing from 0 to 60% CO, were burnt in different types of burners in a large chamber, and at the end of an hour the carbon monoxide in the air was estimated. It is shown that the production of carbon monoxide is mainly due to any sudden cooling of the flame, and for a given type of burner is practically independent of the amount originally present in the gas. The Auer burner produced comparatively large amounts of carbon monoxide, and it is shown that this is due mainly to the presence of the incandescent mantle. The hourly production of carbon monoxide with certain common types of burners is sufficiently high to warrant further efforts to improve them.

The amount of carbon monoxide coming into the air by leakage from the piping etc. of an interior supply having 14 taps, the gas containing 20% CO, is much less than the amount produced in the same time by burning the gas in a small furnace or a small Auer burner.—W. G.

Nitrogen contained in oil shales; Recovery of —. L. Simpson. *Chem. and Met. Eng.*, 1920, 22, 20–22.

THE economy of shale distillation in "single" or "dual purpose" retorts under the conditions obtaining in North America is discussed. The "dual purpose" retort, which is designed to recover a maximum of nitrogen from the shale, even at the expense of the oil content, necessitates high capital charges, and, taking into account the high local costs of sulphuric acid, labour, and plant repairs, coupled with the probable fall in value of ammonium sulphate, the view that the successful establishment of the oil shale industry depends on the continued extreme recovery of the nitrogen is not accepted. The success of the industry is held to be dependent on a single purpose retort, erected at low cost, which will yield a maximum quantity of oil, and the recovery of nitrogen is not advisable unless sulphuric acid can be obtained cheaply or unless a good local market for ammonium sulphate exists.—C. A. K.

Mineral oils; Removal of aromatic hydrocarbons from — by means of trioxymethylene-sulphuric acid. J. Tausz. *J. prakt. Chem.*, 1919, 99, 276–280.

THE efficiency of Nastjukoff's formolite process (this J., 1904, 1082) is increased by the use of trioxymethylene in place of formaldehyde solution. In the case of an oil volatile in steam, e.g., refined ligroin, 500 c.c. is added to a solution of trioxymethylene (30 grms.) in sulphuric acid (100 grms.). The mixture is kept cool and well shaken for half an hour, after which ice (500 grms.) is added, and the oil distilled with steam. Salt may be added to the mixture before the addition of ice in order to protect the hydrocarbons from the sulphuric acid. Less volatile oils are first diluted with ten volumes of previously treated petroleum spirit. It is inadvisable to prolong the treatment, because a proportion of the oil, which increases with the duration of the reaction, is always lost. Thus 1,3,4-trimethylcyclohexane soon gave a coloration with the reagent, and after two days a yellow precipitate was obtained by successively adding ice and ammonia. Aromatic hydrocarbons can be detected in gaseous mixtures by the formation of a red precipitate on shaking with 1–2 c.c. of the reagent. The reaction is also shown by diolefines, such as methyl- and dimethyl-butadienes, which contain conjugated double bonds, but in the case of aromatic hydrocarbons is limited to those which contain a hydrogen atom in the nucleus. Thus, hexamethyl- and hexaethyl-benzenes do not respond to it.—J. K.

Fuel value of volatile liquid mixtures; Determination of —. G. Barsky. *J. Ind. Eng. Chem.*, 1920, 12, 77.

THE use of gelatin capsules is recommended in the determination of the fuel value of very volatile liquids in a bomb calorimeter. They are constructed of ordinary capsules by turning in the caps so that the latter fit the menisci when the capsules are filled; all air is thus excluded. The igniting wire is wound round the capsule.—W. P. S.

Oil fractionation; Constant temperature stillhead for light —. F. M. Washburn. *J. Ind. Eng. Chem.*, 1920, 12, 73–77.

THE stillhead described is used in conjunction with

a Hempel column; it consists of a spiral made from a 12 ft. length of $\frac{3}{4}$ in. wrought iron pipe, the diameter of the spiral being about 6 in. This spiral is mounted in an oil-bath which is heated by a resistance coil immersed in the oil, and the required temperature is maintained by means of a thermostat and relay. The oil-bath is provided with a stirrer. The lower end of the spiral is connected to the side tube at the top of the Hempel column, which is 1½ in. in diameter and made of glass, whilst a side tube at the upper end of the spiral is connected to an ordinary condenser. The oil to be distilled is contained in a 1500 c.c. flask attached to the lower end of the Hempel column and heated by a Rose burner with a hemispherical top covered with asbestos-coated gauze. Thermometers are inserted in the top of the Hempel column, in the top of the spiral, and in the oil-bath respectively. The apparatus gives fractions which have a very small b. pt. range, and almost all of each of the components of a mixture (e.g., benzene, toluene, and solvent naphtha) can be distilled in a practically pure state.

—W. P. S.

Lubricants; Technical and scientific examination of —. F. Frank. Z. angew. Chem., 1919, 32, 374—379.

HILLIGER (Z. Ver. deuts. Ing., 1918, 173) estimated that approximately 0.2 gram of cylinder oil was consumed per h.p. per hour, whilst Schmidt found that although this value might be reached it was too high for the general practice of lubrication. From the experiments of Haserick and others the conclusion is drawn that not only can this value be reached, but that with good working it must be reached, since normally there is superheating up to 330° C. The use of an unsuitable oil or of an excessive quantity of a suitable oil is indicated by the appearance of the oil flecks produced on tinfoil or cartridge paper when held at a distance of about 10 cm. from the indicator tap of the cylinder, so as to receive in each test the same number of puffs of vapour. In the case of asphaltogenic oil and normal lubrication the oil fleck is diffused and shows no sign of radiating structure, whilst excessive lubrication with acid and asphaltogenic oil produces a very dark fleck with sharply defined radiations. Excessive lubrication of the cylinders of gas engines results in deposits due to incomplete combustion of the lubricant mechanically separated. Highly refined oil does not necessarily give the best results in interior lubrication. In the case of the cylinders of such engines as Diesel motors the best results were obtained with absolutely ash-free oil, which was also exceedingly pure in other respects, and had a high flash-point. It is possible also to obtain good results with such oils artificially thickened, e.g., with montan wax. The flash-point of an oil is not an absolutely decisive factor, since it may be materially lowered by the motor spirit. Hence, as a safeguard, in the case of large gas engines the flash-point should not be below 180° C., but a viscosity of 3°—4° Engler is sufficiently high. Turbine oils should be as pure as possible, so as not to emulsify readily with water, and have a high flash-point, sp. gr. as low as possible, and a viscosity of not less than 3° Engler. As a rule oils introduced into the circuit of turbines are not effectively cooled, the coolers being too small to effect complete separation of the admixed water. It is important that the oil-hole should be outside the electric field to obviate the effect of the nitric acid formed from the atmosphere upon the oil. For internal lubrication the conditions may be less stringent. Experiments in which spindle oil (viscosity 1.5° at 50° C.) was substituted for castor oil (viscosity 139° Engler) for shaft lubrication showed a reduction in the heat of friction from 34.4° C. to 6.9° C. Comparative results obtained with a good lubricating

grease and a fairly fluid mineral oil showed that the energy consumed by the latter was about 31.9% less than in the case of the grease. Details are given of an experimental plant in Düsseldorf where oils are tested under actual working conditions. Experiments on a large scale on the manufacture of lubricating oils from low-temperature tar have shown that it is not so much a question of the temperature at which the tar is separated from the coal as of removing the tar as rapidly as possible from the influence of the heat. Another important factor is that the time required for the coal to swell up should be independent of the rate at which the retort can produce gas. The oils obtained by the decomposition of tars usually show a strong tendency to form asphalt owing to the presence of unsaturated hydrocarbons, whilst owing to their high paraffin wax content they frequently solidify at a high temperature. The first drawback may be obviated by rapid distillation of the tar with superheated steam, whilst paraffin wax may be removed by the ordinary process of chilling or by Edeleann's sulphur dioxide process. Other solvents, in addition to liquid sulphur dioxide, appear to have an analogous action.—C. A. M.

Combustible matter in rocks. Fieldner and others. See VII.

PATENTS.

Artificial fuel; Manufacture of —. C. M. C. Hughes, Bognor, and T. Hill-Jones, Ltd., London. Eng. Pat. 136,584, 17.8.18. (Appl. 13,414/18.)

A METHOD is described for the manufacture of briquettes containing coke, breeze, furnace ashes, sawdust, peat, coal, or anthracite, together with pitch, filter-cake from sugar refineries, iron oxide, lime, or chalk as a binder.—W. P.

Fuel. J. V. Eyre, Chertsey, Surrey. Eng. Pat. 136,790, 12.9.19. (Appl. 22,433/19.)

THE fuel consists of coal dust 6½—10 parts, pitch 1½—3 parts, and sawdust 2—3½ parts, all measured by bulk. The powdered components are intimately mixed, and are heated to 250° F. (about 120° C.) with agitation. The mixture is then compressed to one-third of its original volume whilst still warm. It is finally sprayed with water to cool and harden.—W. P.

Carbonisation of fuel by vertical coke ovens and the like. W. E. Davies, Penygraig, S. Wales. Eng. Pat. 136,880, 19.12.18. (Appl. 21,239/18.)

THE coking chamber, which is discharged from below, is surrounded by a band of flues. The flues are triangular, heart-shaped, or oval in section, and are in sets of three vertical flues, one flue being a heating flue and the others waste heat flues, the three being so connected together that heating gases having traversed the heating flue pass directly through both the waste heat flues. The waste gases are collected in horizontal flues. By means of horizontal perforated diaphragms, at suitable distances apart, the heating gases are made to travel against the wall of the oven chamber. A sheet of impermeable metal may be placed between double heating walls and between the stages of heating flues to prevent diffusion of the gases. The temperature of the centre of the oven may be controlled, or secondary reactions may be produced by introducing gases or vapours at the top or bottom of the flues for withdrawing volatile products, by partially or completely forcing the gases into the body of the charge and withdrawing the products of reaction by suction, maintained continuously or intermittently. The oven may be operated under a

pressure within the carbonising chamber between 15 inches mercury vacuum and 100 atmospheres pressure, and at any temperature between 200° and 1400° C.—W. P.

Coke ovens. W. P. Thompson, Liverpool. From Fours et Proc. Mathy. Soc. Anon., Liège, Belgium. Eng. Pat. 137,905, 4.6.19. (Appl. 14,105/19.)

The coke ovens are heated by surface combustion methods. Air and gas are mixed in a passage beneath each retort, and then pass into vertical flues packed with porous fireproof material. The hot gases released from the upper part of these flues are brought by other like flues not packed with porous material into a recuperator serving for heating air or gas. Two recuperators of this kind are arranged under each retort.—W. P.

Steaming gas retorts; Appliances for —. A. E. Whitcher, Burgess Hill, Sussex, H. K. Hiller, and The Hewitt Construction Synd., Ltd., London. Eng. Pat. 136,910, 21.12.18. (Appl. 21,637/18.)

A COMBINED superheater and steam distributor is described. In order to bring the steam into contact with the incandescent carbon at the highest possible degree of superheat, the pipe coil conveying the steam is brought through the back or stop end of the retort. The superheater coil is placed in one of the waste gas flues, and the steam on passing from this may be still further heated in an expansion chamber. From this chamber a fan-like series of openings conducts the steam in regular streams into the coal undergoing distillation.

—W. P.

Water-gas; Manufacture of —. Comp. pour la Fabrication des Compteurs et Matériel d'Usines à Gaz, Paris. Eng. Pat. 113,271, 21.1.18. (Appl. 1159/18.) Int. Conv., 31.1.17.

THE producer consists of two parts of equal height, the lower part cylindrical in shape and the upper a double truncated cone, the bottom of which has a materially smaller cross-section than that of the lower cylinder. Air is blown alternately through each part, first from the bottom of the grate, and then into the middle of the producer as soon as carbon monoxide flames appear in the producer throat. In this way a high and practically uniform temperature is maintained throughout the whole of the fuel, and formation of excess of carbon monoxide during the blowing is avoided. The process is more particularly suitable for small installations.—W. P.

Coal; Method of treating caking — before use in the Mond gas producer for the recovery of the nitrogen. Ehrhardt und Schmer Ges.m.b.H., Saarbrück. Ger. Pat. 301,602, 18.11.16.

BEFORE treatment in the Mond producer the coal is freed from tarry matter by preheating to a temperature below that at which the greater part of the nitrogen is removed, i.e., to about 450° C. By this means the caking property of the coal is almost completely destroyed and very little nitrogen is lost, as formation of ammonia begins only at about 500° C.—L. A. C.

Petroleum; Process of and apparatus for cracking —. F. C. Ruff, Los Angeles, Assignor to By-Products Manufacturing Co., San Francisco, Cal. U.S. Pat. 1,325,582, 23.12.19. Appl., 15.1.17.

PETROLEUM preheated to below its cracking temperature is mixed in a closed chamber containing a catalyst with steam heated to above the cracking temperature. The gaseous products are scrubbed by passage through the liquid residue.—L. A. C.

[Hydrocarbon oil] distillates; Process of condensing and treating —. R. Fleming, Westfield, N.J., Assignor to The Richard Fleming Co. U.S. Pat. 1,325,668, 23.12.19. Appl., 25.3.18.

Hydrocarbon oil vapour from a still is passed through a dephlegmator to the jet of an injector, where water is drawn in and mixed with the oil vapour under pressure. The mixture passes to a chamber where gas and liquid are separated, and the bottom of this chamber is connected by a pipe to the top of an oil and water separator at a higher level. The oil collecting at the top of the separator then passes to a storage tank. The mixture of oil vapour and water produced by the injector is subjected to sufficient back pressure due to the head of liquid in the oil and water separator to prevent any material expansion before reaching the gas separator.—W. F. F.

Oils [gasoline]; Improving —. A. M. McAfee, Port Arthur, Tex., Assignor to Gulf Refining Co., Pittsburgh, Pa. U.S. Pat. 1,326,072, 23.12.19. Appl., 26.6.18.

HIGH-GRADE gasoline is prepared by treating cracked gasoline at a temperature below its end boiling point with sufficient aluminium chloride to convert unsaturated compounds into low-boiling saturated compounds.—L. A. C.

[Mineral] oils; Apparatus for separating composite —. F. E. Van Tilburg, Minneapolis, Minn. U.S. Pat. 1,326,230, 30.12.19. Appl., 18.2.18.

A MIXTURE of oils is vaporised, and the vapour is led into an expansion chamber containing a central vertical tube which passes directly through the top wall of the chamber and is connected to the vapour condenser. The vertical tube is provided with a relatively small aperture just below the top of the expansion chamber, and can be sealed at its lower end.—L. A. C.

Briquette presses. K. K. Koffroth, Amsterdam, Holland. Eng. Pat. 122,190, 4.1.19. (Appl. 270/19.) Int. Conv., 14.11.17.

Petroleum products; Manufacture of high-grade low-boiling —. A. M. McAfee, New York, Assignor to Gulf Refining Co., Pittsburgh, Pa. U.S. Pat. 1,326,073, 23.12.19. Appl., 9.1.15.

SEE Eng. Pat. 22,243 of 1914; this J., 1916, 298.

Hydrocarbons etc. Ger. Pats. 314,745—7. See III.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

PATENTS.

Carbonaceous material; Apparatus for the distillation of solid —. C. D. Burney, London. Eng. Pat. 136,585. (Appls. 14,176, 31.8.18, and 19,177, 21.11.18.)

COAL, cannel, lignite, shale, peat, etc., are distilled at a low temperature in a horizontal retort by passing hot gases from a furnace through a conduit surrounded by the material. The raw material is fed continuously into one end of the retort, and is slowly moved through it by means of a rotating helical conveyor, the helical blade of which is hollow, and is mounted on a hollow shaft. The hot gas is passed into the hollow shaft and thence into the hollow blade at one or more points. The material is heated by conduction through the wall of the conveyor, and the hot gas passes through the retort in a direction opposite to that of the material. The variation in temperature along the length of the retort may be controlled by varying the velocity of the hot gas through the conveyor

or by varying the inlet pressure. An archimedean screw is provided with its blade intermeshing with the blade of the conveyor, so that the material in the retort may be broken up by rotating the screw. The gaseous products of distillation are withdrawn at various points along the length of the retort, and are passed to condensers. The waste gas from the conveyor may be used for preheating the air and gas which are supplied to the burners of the furnace.—W. F. F.

Low temperature distillation of fuel. Merz and McLellan, London, A. C. Michie, Newcastle-on-Tyne, and E. G. Weeks, Monkseaton. Eng. Pat. 136,868, 20.11.18. (Appl. 19,083/18.)

MEANS are provided for heating each charge of incoming fuel, before it is fed to the retort, to a temperature above the temperature of condensation of the distillate but not high enough to cause any appreciable decomposition of the fuel. In this way the binding together of the fuel is prevented during the continuous gasification of fuel at comparatively low temperatures (500° C.) when blowing through superheated steam or other heated gases.—W. P.

Decolorizing vegetable carbon; Manufacture of —. C. E. Eastick, South Woodford, Essex, and S. P. Eastick, London. Eng. Pat. 136,873, 16.12.18. (Appl. 20,971/18.)

ANY vegetable matter or carbonaceous matter obtained from vegetable matter is finely comminuted, moistened, and then carbonised at a high temperature out of contact with air and without addition of other substances. The decolorising power of the product may be increased by washing out the ash with hydrochloric or other acid.

—W. F. F.

Arcs employed in searchlights, projection apparatus, and arc lamps; Method of arrangement, formation and coloration of one or more —. R. Mylo, Charlottenburg. Ger. Pat. 301,235, 21.9.16.

CHANNELS are arranged longitudinally at the base of one or more of the several arcs, and these serve for the introduction of gases or gaseous mixtures, chemicals in the form of dust or vapour, etc., under increased or diminished pressure. By these means one or more of the arcs, as desired, can be established and maintained in any definite direction, and, if necessary, coloured. The various gases or gaseous mixtures are caused to have various velocities by providing the channels with different cross-sections. The gases or other chemicals employed for colouring the arc are introduced through the channel lying nearest to the core of the electrode.—J. S. G. T.

Electric arcs; Apparatus for automatic regulation of the velocity with which air or gas or mixtures thereof enter or leave longitudinal channels in electrodes of — under the influence of increased or diminished pressure. R. Mylo, Charlottenburg. Ger. Pat. 301,236, 21.9.16.

IN the case of electrodes such as those referred to in the preceding abstract the supply of air or other gas is controlled by valves which are operated by alterations of voltage or current strength in such manner that the supply of air or gas is dependent upon these variations.—J. S. G. T.

Thermic effect of cathode rays; Method and means for utilisation of the —. A Sinding-Larsen, Vestre Aker, Norway. U.S. Pat. 1,326,794, 30.12.19. Appl. 30.10.18.

SEE Eng. Pat. 121,280 of 1918; this J., 1919, 568 A.

III.—TAR AND TAR PRODUCTS.

Tar; Estimation of "free carbon" in —. H. I. Waterman and F. H. C. Barkhuysen. Chem. Weekblad, 1919, 16, 1251—1257.

ABOUT 10 grms. of tar is weighed out in a hardened filter, which is then placed in a Soxhlet thimble, both filter and thimble having been previously extracted with ether, dried at 100° C., and weighed. The tar is then extracted in a Soxhlet apparatus with a solvent, such as aniline, chloroform, benzene, or glacial acetic acid mixed with toluene. The time of extraction should be 22½ hours. A table is given of results obtained by other methods, which show considerable variations, but the table of the actual results obtained by the authors, although referred to in the text, is omitted.—W. J. W.

Low-temperature coal tars; Properties and uses of the products containing phenols obtained by the direct distillation of —. F. Fischer and W. Glud. Ges. Abhandl. zur Kenntnis der Kohle, 1918, 3, 109—121. Chem. Zentr., 1919, 90, IV., 979—980.

Low-temperature coal tar was distilled over a naked flame, and the following fractions were collected for examination: (1) To 225° C. motor spirit ("Zündöl"); (2) 225°—300° C., "motor oil"; and (3) over 300° C. If the distillation was stopped at 300° C., a soft pitch was obtained containing the whole of the paraffin; by distilling up to 325° C. a hard pitch remained; at 340° C. the pitch began to decompose. The quantity of motor spirit (flash-point below 35° C.) obtained was about 14% of the tar; it was of low viscosity, sp. gr. less than 1, of a brownish-red colour and petroleum-like odour, and, on account of the phenols present, attacked metals somewhat. Tar prepared from a gas-coal from the Lohberg mine gave with good cooling 25—30% of a similar oil. The darker coloured motor oil, 25—30% of the tar, had sp. gr. 0.998, flash-point 60°—65° C., calorific value 8630 cal., viscosity (at 20° C.) 5° Engler, setting point (at rest) 3° C., (with agitation) -12° C. After a second distillation the fraction remaining above 225° C. had a flash-point of 71° C. From the fraction 300°—325° C., 15—20% of the tar, a quantity of paraffin separated equal to about 0.5% of the tar; the viscous residue has but little value as a lubricant on account of the phenol content, but might be added to the motor oil or used for working up the pitch. After separation of the phenols it formed a good lubricant. Pitch from low-temperature tar closely resembles ordinary pitch, but contains less free carbon; it has sp. gr. about 1.2, a normal calorific value, and leaves less coke residue than ordinary pitch.—L. A. C.

Phenols of low-temperature coal tar. I.—Cresols. W. Glud and P. K. Breuer. Ges. Abhandl. zur Kenntnis der Kohle, 1919, 2, 236—256. Chem. Zentr., 1919, 90, IV. 913—915.

LOW-TEMPERATURE tar always contains a large but variable percentage of constituents soluble in alkalis—the so-called "phenols"—the proportion being highest in tars from gas coals and reaching 50% of the tar. Lohberg (Dinslaken) gas coal gave 7—8% of gas liquor and 10% of tar on distillation. The former contained a small quantity of phenolic substances, 0.1% by weight of the coal, but no measurable quantity of ordinary phenol. A trace of catechol could be detected. Phenols appear in the tar fractions distilling above 200° C. and in about the same proportions as the hydrocarbons. The crude "phenols" contain a considerable quantity of water difficult to separate. Partially methylated polyhydroxyphenols were absent, as also ordinary phenol, which is therefore not a product of low-temperature distillation. All three cresols

were identified. In the fractions 220°—245° C. by acetylation and crystallisation of the phenoxycetic acids, it was possible to identify the xylenols and trimethylphenols. By acetylation and fractional crystallisation of the cresoxycetic acids all three cresols were identified. They predominate in fraction 196°—201° C., but in the higher fractions give place to the xylenols which form almost the whole of fraction 215°—220° C. Of the cresols the meta-compound is present in greatest quantity. The yield of cresols was 0.6% of the tar, and the quantity actually present was probably 1—2%. The yield of cresols seems to be just about double that obtained in gasworks carbonisation. That the cresols in the latter process are partly destroyed was confirmed by passing *m*-cresol over coke heated to 850°—900° C. The condensed liquid obtained was about 30% of the quantity taken and contained about one-half phenols and the rest hydrocarbons, mainly benzene. The "phenols" obtained were insoluble in ammonia and ordinary phenol, and *o*- and *p*-cresol could not be identified. It seems therefore that the production of higher phenols rather than the cresol content of low-temperature tar accounts for the formation of carboic acid in gas tar.—H. J. H.

Phenols of low-temperature tar. II.—Catechol. W. Glud. Ges. Abhandl. zur Kenntnis der Kohle, 1919, 3, 66—71. Chem. Zentr., 1919, 9V., 915.

The gas coal (Lohberg) referred to in the preceding abstract was distilled in the absence of steam. From the gas liquor catechol equal to 0.0056% of the coal was recovered and approximately the same quantity was obtained from the tar, which contained about 0.6%. A sample of low-temperature tar obtained from a Saar gas coal in a large scale distillation yielded 1.5% of catechol. By making an experiment on a tar with a known quantity of added catechol it was found that the efficiency of extraction was about 50%. Thus the Lohberg coal can be considered as yielding about 0.02% of catechol, and the Saar coal in large scale practice 0.25%. By the extraction of aqueous salt solutions of catechol with ether or extraction from carbon tetrachloride the loss is small; a 70% yield is also obtained from an ether or tar oil solution by means of calcium chloride and ammonia. Extraction from ether solution with solution of sodium carbonate or ammonia is less satisfactory.—H. J. H.

Thiophene in commercial benzols; Estimation of —. A Meyer. Comptes rend., 1919, 169, 1402—1401.

A MODIFICATION of Denigès' method (this J., 1896, 746). The reagent is prepared by dissolving 50 grms. of mercuric oxide in 200 c.c. of sulphuric acid diluted with 1000 c.c. of water. For the estimation 20 c.c. of this reagent is mixed with 75 c.c. of methyl alcohol and 5 c.c. of the benzol under examination, in a stoppered bottle. The mixture is shaken from time to time, and after 30 minutes is filtered. 10 c.c. of the filtrate is diluted with 20 c.c. of water and then a few drops of nitric acid and 1 c.c. of a 20% solution of iron ammonium alum are added and the whole is titrated with *N*/10 ammonium thiocyanate solution. To allow for the possible presence of acetone in the methyl alcohol a blank estimation is conducted using 5 c.c. of benzene free from thiophene. The two titration readings being *n*, and *n* respectively, the amount of thiophene in one litre of benzol is given by

$$0.01 \times 2000 \times (n - n) \times 0.11. \quad \text{—W. G.}$$

1,2,4-Chlorobenzenedisulphonic acid and its transformation into the symmetrical compound. S. C. J. Olivier. Rec. Trav. Chim., 1919, 38, 351—355.

WHEN the anhydrous barium salt of 1,2,4-chloroben-

zenedisulphonic acid is heated with fuming sulphuric acid for five hours at 300° C. it is for the most part converted into 1,2,5-chlorobenzenedisulphonic acid.—W. G.

Chlorobenzenedisulphonic acid prepared according to the patent of Meister, Lucius, and Bruning; Structure of the —. S. C. J. Olivier. Rec. Trav. Chim., 1919, 38, 356—357.

By preparation and identification of its acid chloride it is shown that the chlorobenzenedisulphonic acid obtained by the sulphonation of *p*-chlorobenzenesulphonyl chloride by the method described in Ger. Pat. 260,563 (this J., 1913, 693) is the 1,2,4-acid.—W. G.

Nitro-derivatives of diphenylamine. C. F. van Duin and B. C. R. van Lempe. Rec. Trav. Chim., 1919, 38, 358—368.

IN an endeavour to establish the position of the two entrant nitro-groups in the preparation of a hexanitrodiphenylamine by nitration of 2,4,6,3'-tetranitrodiphenylamine, the authors have prepared a number of nitro-derivatives of diphenylamine. (See further J. Chem. Soc., 1920, i., 155.) —W. G.

Mineral oils. Tausz. See IIa.

Lubricants. Frank. See IIa.

Naphthylamine and xylidine in flotation. Robie. See X.

Iodination of aromatic amines. Elbs and Volk. See XX.

Oil fractionation. Washburn. See II.

PATENTS.

*1,6-Dihydroxynaphthoyl-*o*-benzoic acid and its salts [sweetening agents]; Preparation of —.* Ges. für Chem. Ind. in Basel, Switzerland. Ger. Pat. 311,213, 15.2.16.

By condensing 1,6-dihydroxynaphthalene with phthalic anhydride or phthalic acid in the presence of boric acid, 1,6-dihydroxynaphthoyl-*o*-benzoic acid, of m. pt. 226°—227° C., is obtained as chief product; in the absence of the boric acid, or using other condensation agents, the product consists almost entirely of the phthalein. The acid and its salts are characterised by a sweet taste and should serve as substitutes for sugar and other sweetening agents; the 1,5-isomeride is tasteless. 1,6-Dihydroxynaphthoylbenzoic acid can also be used for the production of dyes, and in some respects is superior to the 1,5-isomeride for this purpose.

—D. F. T.

Hydrocarbons and alkali salts of high molecular carboxylic acids; Manufacture of —. E. Albrecht, Hamburg-Wallhof, R. Koetschau, Hamburg, and C. Harries, Berlin-Grünwald. Ger. Pats. (a) 311,715, 29.3.16, (b) 311,716 and (c) 314,747, 11.6.16.

HYDROCARBONS such as (a) tar products from lignite, shale, peat, or bituminous asphalt, cracked petroleum oils, products extracted from oils by means of sulphur dioxide, esters of the olefines obtained from lignite tar oil, *e.g.*, by the action of sulphuric acid, acetic acid, chloroacetic acid, or the like, or esters obtained by the action of potassium acetate on halogenated petroleum hydrocarbons; or (c) the highly unsaturated acid resins separated from waste acid from mineral oil refining, or the compounds produced therefrom by treatment with water, steam, or lime, or petroleum pitch, are melted with an alkali or with a highly concentrated alkaline solution. (b) The hydrocarbons are heated with an alkaline liquor so that,

by elimination of oil and water, highly concentrated or molten alkali is formed in the melt. Light hydrocarbons of a high degree of purity are formed by the treatment owing to decomposition of certain impurities, and alkali salts of high molecular carboxylic acids are produced simultaneously. The alkali salts, which are readily separated from unchanged alkali, rather well and may be employed as soap substitutes. The alkali melt may be agitated by a current of a gas, which may also assist the reaction, *e.g.*, oxygen accelerates the salt formation, whereas hydrogen raises the purity of the light hydrocarbons. The action is rapid and is often complete in less than half an hour. Increased or reduced pressure may be employed during the fusion process, inactive solvents may be present, or catalysts may be added to accelerate the reaction. Examples: 100 kilos. of gas-oil from lignite and 100 kilos. of potassium hydroxide gave about 30 kilos. of light oil, b. pt. 150°–220° C., 45 kilos. of alkali salts, 35 kilos. of residual oil, and 90 kilos. of unchanged potassium hydroxide; 100 kilos of paraffin gas-oil, the iodine value of which had been raised to about 150 by cracking, gave about 60 kilos. of alkali salts, 35 kilos. of benzol and light oil, b. pt. 80°–220° C., and 15 kilos. of residual oil.—L. A. C.

Anthracene; Production of pure —. P. Leroux, Gennevilliers, Assignor to Soc. d'Eclairage, Chauffage, et Force Motrice, Paris, France. U.S. Pat. 1,326,515, 30.12.19. Appl., 24.8.18.

SEE Eng. Pat. 119,855 of 1918; this J., 1919, 354 A.

Resin. U.S. Pat. 1,326,579. See XIII.

Resin solutions from tar. Ger. Pat. 305,065. See XIII.

Linseed oil substitute. Ger. Pat. 305,515. See XIII.

Nitro-derivatives of tetrahydronaphthalene. Ger. Pat. 299,014. See XXII.

IV.—COLOURING MATTERS AND DYES.

Hydroxyanthraquinones; Certain metallic derivatives of —. M. L. Crossley. J. Amer. Chem. Soc., 1919, 41, 2081–2083.

COPPER, cadmium, nickel, iron, cobalt, and chromium salts of alizarin, anthrapurpurin or flavopurpurin are formed when the hydroxyanthraquinone is boiled with a salt of the respective metals, anhydrous sodium acetate, and nitrobenzene. The alizarin salts, though slightly soluble in water, dye wool a pale shade of the colour produced when the wool previously mordanted with the metallic salt is dyed with alizarin. The dye is evenly distributed throughout the fibre, and is apparently fast. In view of these facts it is suggested that the formation of an insoluble alizarin lake in a mordanted fibre is accomplished by combined physical and chemical phenomena.—W. G.

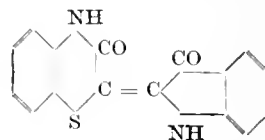
Hydroxydianthraquinonyls; Oxidative formation of — from hydroxyanthraquinones. II. Experiments with erythrohydroxyanthraquinone and quinizarin. R. Scholl, E. Schwinger and O. Dischendorfer. Ber., 1919, 52, 2254–2261.

THE formation of tetrahydroxydianthraquinonyl by the action of hypochlorite on an alkaline solution of alizarin does not appear to be an example of a general reaction, but similar cases are encountered with erythrohydroxyanthraquinone and quinizarin. According to Ger. Pat. 167,461 the former when melted with potash yields the hydro compound of a substance, $C_{28}H_{14}O_6$, of unexplained constitution; this is now shown to be 1.1'-dihydroxy-2.2'-dianthra-

quinonyl, since when distilled with zinc dust in a hydrogen vacuum it gives 2.2'-dianthryl and, when heated in carbon dioxide at 500° C., it yields 2.2'-dianthraquinonylene-1.1'-oxide. Quinizarin, according to Ger. Pat. 146,223, is converted by salts of weak acids into two compounds, $C_{28}H_{14}O_8$ and $C_{28}H_{12}O_8$; further investigation proves that only the first of these is a new substance, the second being a mixture of the first and quinizarin. The new substance is shown to be 1.4.1'.4'-tetrahydroxy-2.2'-dianthraquinonyl since it gives 2.2'-dianthryl when distilled with zinc dust and a furan derivative when melted with zinc chloride. The substance $C_{28}H_{14}O_8$ obtained by A. G. and W. H. Perkin (this J., 1889, 112) by the distillation of sodium anthraquinone-2-sulphonate has been distilled with zinc dust, whereby anthracene and 2.2'-dianthryl were obtained, but the result does not throw much light on the constitution of the substance.—H. W.

Benzoketodihydrothiazine; Dyestuffs derived from —. W. Herzog. Ber., 1919, 52, 2270–2274.

BENZOKETODIHYDRO-1.4-THIAZINE reacts with α -isatinanilide in a solvent of high boiling point, such as ethyl benzoate, to yield 2-(benzodihydro-1.4-thiazine)-2-(indole)-indigo (annexed formula), dull, brownish-violet needles with metallic glance, m. pt. above 300° C., which dyes cotton bluish-violet and wool reddish-violet.



2-(Benzodihydro-1.4-thiazine)-2-(thionaphthen-indigo, yellowish silky needles, m. pt. above 300° C., similarly prepared from thionaphthenquinone-anilide, and 2-(benzodihydro-1.4-thiazine)-3-(indole) indigo, orange-red silky needles, m. pt. about 270° C., have little affinity for the textile fibres. The inferiority of the substances as dyes in comparison with Indigo Blue and Thioindigo Red is readily interpreted according to the Claass hypothesis (this J., 1916, 1150) since they contain only one quinonoid indogen, whilst the latter dyes contain two such groups.—H. W.

Gentian Violet. Crossley. See XIXB.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Fabrics; Determination of the water resistance of —. F. P. Veitch and T. D. Jarrell. J. Ind. Eng. Chem., 1920, 12, 26–30.

THE two following tests are recommended as being the most trustworthy of several investigated. *Funnel test.*—A piece of the fabric, 1 ft. square, is crumpled in the hand, immersed in water at 21°–27° C. for 24 hrs., being taken out and crumpled several times during this period, then dried at 45° C. for 24 hrs., and hung up in the laboratory for 18 hrs. These operations are repeated. The fabric is then once more crumpled, straightened out, placed on a piece of absorbent paper of the same size, the two are folded together in the usual way to form a filter and placed in a 6-in. glass funnel having an angle of 60°. Water at 21°–27° C. is poured into the filter to a depth of exactly 4 in. (this equals 500 c.c.), and this depth is maintained by the ordinary device of an inverted flask filled with water. A graduated cylinder is placed under the stem of the funnel. The time is noted when the paper under the fabric begins to wet, when it is entirely wet, when the first drop passes

into the cylinder, and the quantity of water collected in the latter in 1, 3, 6, and 24 hrs. Note is also made of the time and extent to which the fabric becomes wet above the water level. The water resistance is rated very high when the fabric above the water level and the paper both remain dry after 24 hrs.; high if the paper becomes just wet and the fabric above the water level remains dry; medium if water drips through at the rate of 5–25 c.c. in 6 hrs., or 25–50 c.c. in 24 hrs., and low if the water dripping through exceeds 75–200 c.c. in 6 hrs., or 150–300 c.c. in 24 hrs. *Spray test*.—The piece of fabric used for the funnel test is dried at 45° C. for 24 hrs., and hung up in the laboratory overnight; it is then clamped loosely in a frame which is fixed at an angle of 45° in a trough, and tap water is allowed to fall from a height of 6 ft. on the central portion of the fabric, covering an area of about 8 in. in circumference, for 24 hrs. The water tap has a 2½ in. spray nozzle having 25 holes each 1.9 mm. in diameter, and the water flow is 1000 c.c. per min. The under side of the fabric is examined periodically. The water resistance is rated as follows:—High, if the under surface remains dry for 7 hrs. but is damp in 24 hrs.; medium, if the under surface remains dry for 1 hr. but is wet in 3 hrs.; low if it remains dry for 5 mins. but is wet in 30 mins. The crumpling, wetting, drying, re-wetting, and re-drying of the fabric are done with the object of removing the temporary resistance of new fabric and to imitate, to some extent, the conditions of actual service.—W. P. S.

Sulphite-cellulose waste liquor as fertiliser. Bokorny. See XVI

Nitrogen in celluloid. Nicolaidot and Vourloud. See XXII.

PATENTS.

Wood-pulp digester liquor; Recovering valuable constituents from —. H. K. Moore, Berlin, N. H., Assignor to Brown Co., Portland, Me. U.S. Pat. 1,324,920, 16.12.19. Appl., 14.8.13.

The liquor is concentrated, burnt, extracted with water, and re-causticised.—L. L. L.

Cellulose acetates; Production of solutions of —. F. Steimmig, Hannover. Ger. Pat. 307,075, 3.6.17.

FURFURAL is used as solvent either alone or with other organic solvents in which cellulose acetate is not completely soluble. The acetone-soluble form of cellulose acetate, and also the chloroform-soluble form, yield satisfactory solutions. The solvent action of furfural is not reduced by the admixture of other liquids in which cellulose acetate is only partially soluble, e.g., ethyl acetate, methyl alcohol, ethyl chloride, benzene, and toluene.—H. J. H.

Paper-stock; Process and apparatus for seasoning and rectifying —. G. E. Poggel, Louisville, Ky. U.S. Pat. 1,325,044, 16.12.19. Appl., 24.9.17.

A PROCESS of seasoning and rectifying paper stock consists in blowing currents of heated air upwards between the sheets which are suspended within an enclosure. After passage between the sheets the air is again made to travel in the same cycle.

—L. L. L.

Paper machines; Arrangement for ensuring the movement of the guide rollers of the long sieve of —. Farb. vorm. Meister, Lucius, and Brüning, Höchst. Ger. Pat. 308,372, 12.5.16.

The guide rollers are provided with rings of elastic material such as rubber or felt, which press lightly into the sieve and ensure their sympathetic movement, thereby preventing friction and rapid wear due to the usual tendency of the rollers to remain stationary.—D. F. T.

Paper, pasteboard, cellulose, and similar material; Arrangement for drying — in continuous strips. H. Mallick, Gross-Sachsen. Ger. Pat. 309,543, 16.2.17.

The bands of paper or other material are passed over steam-heated cylinders and at the same time subjected to a blast of air of a different temperature from the cylinder. By arranging the dryer in several sections filled with air at different temperatures it is possible to introduce into any section the air issuing from a previous section.—D. F. T.

Thread, twine, or cord; Manufacture of — from paper or like material. J. C. Grant, Barnes. Eng. Pat. 136,611, 11.12.18. (Appl. 20,894/18.)

Artificial silk; Apparatus employed in manufacture of —. J. Clayton, Coventry. Eng. Pat. 136,784, 5.8.19. (Appl. 19,298/19.) Addition to Eng. Pat. 136,769, 16.6.19.

Drying machine cylinders [for fabrics, paper, etc.]; Dull-head bearings for —. H. Holt, St. Annes-on-Sea, and J. K. Malone, Bury. Eng. Pat. 137,209, 23.4.19. (Appl. 10,098/19.)

Celluloid; Manufacture of unflammable —. H. Dreyfus, Basle, Switzerland. U.S. Pat. 1,325,931, 23.12.19. Appl., 15.2.18.

See Eng. Pat. 111,301 of 1918; this J., 1918, 687 A.

Paper pulp; Treatment of —. J. A. De Cew, Montreal, Canada, and R. J. Marx, London. Eng. Pat. 137,207, 15.4.19. (Appl. 9576/19.)

See U.S. Pat. 1,300,357 of 1919; this J., 1919, 497 A.

Turkey-red oil substitute. Ger. Pat. 310,541. See XII.

Saponin from sulphite-cellulose waste liquors. Ger. Pat. 311,339. See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Nopps; Dyeing of —. A. Reyl. Färber-Zeit., 1919, 30, 252–254.

THE usual method of dyeing nopps in woollen goods consists in topping off the vegetable fibres by means of direct cotton colours, or the dyeing of the material in a neutral bath with neutral wool colours along with direct cotton colours. In the case of several pieces which could not be dyed level the defect was proved to be due to the presence of fatty matter on the cotton. The material requires either wet or dry scouring to remove the saponifiable or unsaponifiable fat prior to dyeing.—L. L. L.

Aniline Black fast to reducing agents. A. Ehrenzweig. Deuts. Baumwollind., 1919, 1, 71. Chem.-Zeit., 1919, 43, Rep., 316.

AN Aniline Black unobtainable by reducing agents is produced by means of compounds of chloric acid, 2–8 parts of aniline glycolate and tartrate, and 1 part of *p*-phenylenediamine, without chromates and salts of heavy metals. A thiocyanate is added to protect the fibre, and this also takes part in the formation of the black. Dry steam at 100°–120° C. must be employed. The elimination of the chromo and soap baths, which are replaced by washing, is advantageous in dyeing articles by the Prud'homme process. The method is applicable to dyeing half-wool and half-silk.—L. A. C.

ERRATUM.—This JOURNAL, Jan. 31, 1920, p. 61 A, col. 1, "*Tinctorial properties of anthocyanins and certain related compounds*," by A. E. Everest and A. J. Hall. In lines 3-4 and 8 of the abstract, for "anthocyanins" read "anthocyanins."

Metallie derivatives of hydroxyanthraquinones. Crossley. See IV.

PATENTS.

Ageing machines. Calico Printers' Assoc., Ltd., G. Turner, and E. A. Fournieux, Manchester. Eng. Pat. 136,507, 28.8.19. (Appl. 21,134/19.)

Noxious gases are often given off in ageing machines which may hinder the proper development of, or otherwise interfere with, the colour. This is more particularly the case with Aniline Black. The usual method of ventilating these machines consists in providing openings generally on the top or at the back of the machine, these openings being connected to a trunk or shaft leading to the open air. By this method there is no ventilation between the folds or laps of the fabric, and the removal of gases is thus incomplete. This defect is overcome by providing suction elements between the folds or laps of fabric and removing the vapours or gases as they are generated.—L. L. L.

Paper yarns and fabrics; Method for dyeing — with substantive colours. Chem. Fabr. Coswig-Anhalt. Ger. Pat. 310,965, 15.3.18.

In addition to oxidising agents there are introduced into the dyeing bath such substances as boric acid, acetic acid, ammonium chloride, weak organic acids, ammonium salts of phosphoric acid, etc. These aid the transfer of the colour to the material and the process, which is applicable with all substantive dyes, yields an open, soft material with bright colour.—D. F. T.

Washing cloth made from yarn treated with mineral oil; Process for —. A. Pinagel, and Aachener Chem. Werke für Textilindustrie, Aachen. Ger. Pat. 311,403, 19.7.16.

The cloth is first immersed in an alkaline saponin solution and an alcoholic fulling oil containing only a small percentage of fat is added, and then the cloth is washed in the usual way. Whereas by the existing methods goods treated with mineral oil require 3–4 kilos. of the best fulling soap containing 60% of fat, after preliminary treatment with saponin solution it is only necessary to use $\frac{1}{2}$ –1 kilo. of a fulling material containing 15% of fat to achieve a satisfactory washing.—H. J. H.

Wool fibres; Stripping colour from —. F. Kollm. Berlin. Ger. Pat. 314,852, 21.3.18.

DETERIORATION of wool fibres during the stripping of colour therefrom, by means of, for instance, formic acid and "decolin," is considerably reduced by addition of the decomposition products of proteins, such as protalbinic acid, lysalbinic acid, and the like, to the bath.—L. A. C.

Turkey-red oil substitute. Ger. Pat. 310,541. See XII.

Detergents. Ger. Pat. 314,590. See XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Nitric acid; Formation of — from nitric oxide. A. Sanfourche. Bull. Soc. Chim., 1919, 25, 633–655.

A STUDY of the oxidation of nitric oxide by dry air over the temperature range 50° C. to 600° C. showed that with an insufficient supply of air it is always nitrogen trioxide which is formed, both at temperatures below 0° C. and at the ordinary temperature. With an excess of air the final term of oxidation is the peroxide at temperatures below 250° C., the formation of the peroxide being extremely rapid at temperatures below 0° C., and the

velocity diminishing with rise in temperature. At temperatures above 250° C. some nitrogen trioxide, is always obtained, the trioxide becoming more and more abundant as the temperature rises, until at about 600° C. the trioxide is the only oxide formed. At temperatures below 0° C. the direct combination of nitric oxide with nitrogen peroxide to form nitrogen trioxide is possible. It is not the peroxide but the trioxide which acts as an intermediary in the formation of nitric acid from nitric oxide, at any rate in the usual installations where the contact of nitrous vapours, air, and water is permanent. If, however, means were devised periodically to assure total oxidation to the peroxide before the action of the water occurred, the productiveness of the installation would be increased. This may be done by the use of alternating oxidation towers and absorption towers. On the other hand, the oxidising action of strong nitric acid on nitrogen trioxide shows that it is not possible to prepare by this means an acid stronger than 50%, since above this concentration the nitric acid is reduced with the formation of nitrogen peroxide. A higher concentration could probably be reached if, whilst assuring the total oxidation of the nitrous vapours, nitrogen peroxide and not the trioxide was allowed to act on the aqueous nitric acid.—W. G.

Nitrogen peroxide; Dissociation constant of —. E. Wourzel. Comptes rend., 1919, 169, 1397–1400.

DIRECT measurements at temperatures between 0° C. and 86.5° C. show that the value of the dissociation constant, K, is given by the equation, $\log K/T = 8.9908 - 2810.5/T$, from which the heat of polymerisation is calculated as 12,850 cal., exact to within 0.5%. —W. G.

Ammonium sulphate; Manufacture of — from gypsum. D. H. B. Wride. Chem. Age, 1920, 2, 32–33.

A PROCESS for the utilisation of gypsum for the production of sulphuric acid is described in Eng. Pat. 124,842 of 1919, in which a mixture of gypsum and water is treated with ammonia and carbon dioxide and converted into ammonium sulphate and calcium carbonate. These are separated by filtration, and the ammonium sulphate is recovered by evaporation and heated with ferric oxide, when ammonia gas and ferric sulphate are formed; the former is returned to the gypsum-treating plant and the latter is decomposed in another furnace into sulphur trioxide and ferric oxide. The first part of the process is capable of being utilised for the production of ammonium sulphate, and is specially applicable to the ammonia obtained by the Haber synthetic process, as the carbon dioxide required is obtained as a waste product in the preparation of the hydrogen. Difficulties have arisen in connection with the filtration of the calcium carbonate mud, which in the presence of small quantities of silica and alumina becomes very slimy and difficult to wash, but suction filters of the leaf type such as are used in gold extraction have given good results. There are considerable deposits of gypsum in Westmorland, Cumberland, and Derbyshire, and experiments were carried out early in 1919 in the laboratory of the Research Department of the Ministry of Munitions. The fresh gypsum was brought into contact with the nearly completely converted ammonium carbonate solution on the counter-current principle. No trouble was experienced with the filtration and the chalk mud after washing tested NH_3 0.62%, SO_4 1.28%, CO_2 42.11%, and moisture 40.81%. 95% of the ammonia was recovered, 97% of which was obtained as ammonium sulphate. The reaction is an exothermic one, and arrangements must be made for cooling the apparatus.—W. H. C.

Sulphurous acid; Neutralisation curve of —. I. M. Kolthoff. Chem. Weekblad, 1919, 46, 1154—1163.

A VALUE of 1×10^{-7} is given for the second dissociation constant of sulphurous acid, calculated from the degree of hydrolysis of solutions of sodium sulphite. The hydrogen ion concentration of a 0.1 mol. solution of sodium bisulphite was found to be 3.3×10^{-2} . A curve is given showing the neutralisation of sulphurous acid to sulphite. In titrating sulphurous acid accurate results can only be obtained if, after coloration of the phenolphthalein, an excess of barium nitrate is added and the titration then continued.—W. J. W.

Chlorates; Iodometric estimation of —. I. M. Kolthoff. Pharm. Weekblad, 1919, 56, 460—465.

HIGH results are obtained in the direct iodometric estimation of chlorates owing to oxidation by atmospheric oxygen. Rupp's method (this J., 1918, 205 A) is accurate, but smaller quantities of reagents are sufficient, whilst by the use of ferrous sulphate still smaller quantities may be employed.—W. J. W.

Iodides; Estimation of —. I. M. Kolthoff. Pharm. Weekblad, 1919, 56, 1029—1035.

A COMPARISON of existing methods was made with the same solution of potassium iodide. Winkler's method (this J., 1918, 390 A) gives good results only in absence of bromide. The presence of small quantities of bromide does not affect the results obtained by those methods (Volhard, Vincent, Richard) in which hydrogen iodide is oxidised to iodine and the latter estimated; but after addition of tartaric acid in Richard's method the solution should be allowed to stand for 2 to 3 minutes. By adding benzoic acid and potassium iodate to an iodide solution, boiling to eliminate iodine, and then adding potassium iodide and sulphuric acid, and titrating back with thiosulphate, good results are obtained even in presence of bromide.—W. J. W.

Potassium thiocyanate; Photochemical oxidation of —. D. Ganassini. Boll. Chim. Farm., 1919, 58, 457—463.

UNDER the influence of sunlight solutions of certain concentrations of potassium thiocyanate, especially those of the pure salt free from traces of ferrous salts, are oxidised by atmospheric oxygen with separation of a yellow precipitate of pseudo-cyanogen sulphide (CNS)₂. In addition to this occasional product, hydrocyanic and sulphuric acids, carbon dioxide, and ammonium salts are formed as constant products of the photochemical decomposition of the thiocyanate: $12\text{KCNS} + 12\text{O}_2 + 6\text{H}_2\text{O} = 6\text{K}_2\text{SO}_4 + 3\text{HCN} + 3\text{CO}_2 + 3\text{NH}_3 + 2(\text{CNS})_2$; an unstable peroxygenated compound, possibly analogous to Caro's acid, is also formed as an intermediate product. Normal, decinormal, and centinormal solutions of potassium thiocyanate undergo decomposition; the two last do not, however, deposit pseudo-cyanogen sulphide and do not alter appreciably in titre within certain time limits. (See also J. Chem. Soc., Mar., 1920.)—T. H. P.

Sodium zincate. Equilibria in the system Na₂O-ZnO-H₂O. F. Goudriaan. Proc. K. Akad. Wetensch., 1919, 22, 179—189.

IN the system Na₂O-ZnO-H₂O there appear as solid phases: zinc oxide, sodium zincate Na₂O.ZnO.4H₂O, and the monohydrate of sodium hydroxide. Sodium zincate forms incongruent solutions from which at the concentration 1 part sodium hydroxide to 2 parts of water zinc oxide separates. Gelatinous zinc hydroxide is a phase of varying water content from which it is not possible to remove all adsorbed material.—J. F. S.

Dolomites; Comparison of —. A. Rothrock and J. B. Shumaker. Chem. News, 1920, 120, 29—31.

TYPICAL dolomite, CaCO₃.MgCO₃, probably never occurs in nature, one or other constituent being partially replaced by other substances. Analyses of thirteen different minerals from various sources are given. The content of calcium carbonate ranged from 27.01 to 96.25 and of magnesium carbonate from 1.15 to 67.91. It is concluded that many minerals and rocks which may fairly be classed as dolomites vary greatly in composition and many so-called dolomites are really limestones.—A. B. S.

Combustible matter in silicate and carbonate rocks; Determination of —. A. C. Fieldner, W. A. Selvig, and G. B. Taylor. U.S. Bureau of Mines. Tech. Paper 212, 1919. 18 pages.

LISSNER's method (this J., 1910, 112), viz., digestion of the material with hydrofluoric and hydrochloric acids, and combustion of the insoluble residue, was found to give abnormally high results for organic hydrogen in rocks containing small amounts of combustible matter, owing to the presence of a hydrous acid aluminium fluoride in the dried, insoluble residue. In the following modification of Lissner's process the presence of this compound in the residue is obviated by adding an excess of hydrochloric acid after the silicates are decomposed, and by avoiding evaporation to dryness. 0.2—1.0 gm. of the finely divided (silicate) rock is heated for 10 mins. with 15 c.c. of concentrated hydrochloric acid in a platinum dish, then 20 c.c. of strong hydrofluoric acid is added, and the contents of the dish are maintained at incipient boiling until the inorganic matter appears to be decomposed (30 mins.—2 hrs.); sufficient of the mixed acids (in the same proportions) is added from time to time to maintain the volume of the liquid above 15 c.c. After evaporation of the solution to 15 c.c., 25 c.c. of concentrated hydrochloric acid is added, the liquid being boiled to re-dissolve any acid aluminium fluoride, then diluted with an equal volume of hot water, boiled again, and filtered, while hot, through ignited asbestos or platinum sponge. The residue is washed with hot water until free from chlorides, placed, with the filter, in a porcelain or platinum boat, dried for 2 hours at 105° C., and transferred direct from the drying oven to the tube of the combustion furnace. It is important to avoid evaporation below 15 c.c., and, if the digestion is conducted on a hot plate, the temperature must not exceed that of incipient boiling. Samples composed mainly of organic matter are best treated on a water bath. When these precautions are observed, the loss of organic carbon and hydrogen during digestion is practically negligible, even from pure bituminous coal. Calcareous rocks require preliminary treatment with dilute hydrochloric acid to remove calcium carbonate, &c., before the process is applied; the insoluble residue is collected on ignited asbestos, which is then heated with hydrochloric and hydrofluoric acids, as described. In a series of analyses of shales, clays, and slates from coal mines, the ratios of organic hydrogen to organic carbon found by this method varied between 1:7 and 1:17 and the combined water between 2.5 and 5.7%.

—W. E. F. P.

Minerals; Colloidal colouring substances in —. C. Doelter. Kolloid-Zeits., 1920, 26, 23—27.

THE colour of many minerals is due to small quantities of impurity which may be the same in differently coloured minerals. The nature of the colour depends on the size of the particles of the impurity. Changes in the colour effected by heating or by the action of radium rays are due to change in the dimensions of the particles.—J. F. S.

Metallic oxide sols; Physico-chemical analysis of —. W. Pauli. *Kolloid-Zeits.*, 1920, 26, 20—23.

A POLEMICAL article against Zsigmondy's theory of solutions and suspensions.—J. F. S.

Rhodium; Behaviour of hydrogen towards —. A. Gutbier and O. Maisch. *Ber.*, 1919, 52, 2275—2280.

THE experiments were performed in the same manner as with palladium, iridium, and platinum (this J., 1913, 661; 1919, 679, 680). The deep black modification, rhodium black, is able to absorb far more hydrogen than any other form. The maxima of hydrogen absorption amounted to 180 volumes at 190° C. for the grayish-black form, 165 volumes at 40° C. for the slightly grayish-black variety, and 206 volumes at 0° C. for the deep-black modification. (The varieties are obtained in the order given by the reduction of sodium rhodium chloride by hydrazine in faintly acid, neutral, and ammoniacal solution respectively; the purest preparations, dried in a vacuum over phosphorus pentoxide, contained about 3.0% H₂O and 0.3% O.) The absorption of hydrogen by rhodium is much less than by palladium and corresponds to that observed with platinum. Spongy rhodium, like spongy platinum, only absorbs small amounts of hydrogen.—H. W.

ERRATA.—This J., July 31, 1919, p. 498 A, line 21 from top, for "100" read "100 grms.," and for "25 grms." read "25 c.c."

Barium products; Modern methods of handling —. G. F. Zimmer. *Chem. Age*, 1920, 2, 34—35.

Absorption of gases. Mosciecki. *See* I.

Acid-resisting metals. Matignon. *See* X.

Acid-resisting alloy. Kowalke. *See* X.

Waste liquors from potash industry. Nolte. *See* XVI.

Iodometric estimation of acids. Kolthoff. *See* XXIII.

Carbon dioxide. Constantino. *See* XXIII.

Carbon dioxide and water in minerals. Almström. *See* XXIII.

PATENTS.

Hydrochloric acid; Production of — from sodium bisulphate and salt. E. Teisler, Delna. *Ger. Pat.* 300,723, 23.5.16.

SOLID substances resistant to hydrochloric acid, e.g., metallic sulphates, or various forms of carbon, are added to the reaction mixture. Segregation of the mixture due to the fusion of the sodium bisulphate is thereby checked and hydrogen chloride is evolved freely at 150° C., the residue forming a porous mass practically free from chloride and free sulphuric acid.—D. F. T.

Radium; Production of compounds of —. H. O. Hedström, Djursholm, Sweden. *Eng. Pat.* 136,768, 6.6.19. (Appl. 14,439/19.)

RADIUM compounds are extracted from minerals or the like by means of liquid sulphur dioxide; the solvent is removed by evaporation, and the extract treated for the production of radium-barium compounds. If oil or other carbonaceous material is present in the mineral, e.g., Swedish alum slates (shales) of the Cambrian formation, or "kolm," this is first removed by burning or distillation. —L. A. C.

Ammonium sulphate; Production of —. C. W. Bailey, H. S. Denny, and W. H. H. Norris, Langwith. *U.S. Pat.* 1,325,605, 23.12.19. *Appl.*, 2.12.18.

NITRE-CAKE, ammonium sulphate, and sodium sulphate are mixed in the presence of water; a portion of the sodium sulphate is separated, and the solution is used without the addition of acid for absorbing ammonia from gases containing the same.—L. A. C.

Leucite; Recovering potash from —. *Treatment of felspar, leucite, and the like.* F. A. Rody, Newark, N.J., Assignor to Metallurgical Co. of America, New York. *U.S. Pats.* (A) 1,325,881 and (B) 1,325,882, 23.12.19. *Appl.*, 18.5.15.

LEUCITE or the like is fused and the fused product is digested (A) under pressure with a solution of sodium salts, or (B) with a solution of easily soluble sodium and potassium salts. The strength and constitution of the solutions are so adjusted that the sodium in solution replaces potassium present in the leucite with a corresponding increase of the amount of potassium salts in the solution.—L. A. C.

Cyanates; Production of — soluble in water. J. D. Riedel A.-G., Berlin-Britz. *Ger. Pat.* 314,629, 7.1.19.

CHLORINE or bromine is passed into concentrated alkaline solutions of cyanides. No appreciable quantity of halogen compounds of cyanogen is produced. Sodium cyanate is formed and separates out in a form suitable for technical and pharmaceutical purposes.—H. J. H.

Inert rare gases; Production of — in a state of purity. R. Brandt, Ludwigshafen. *Ger. Pat.* 314,907, 2.11.16.

THE inert gases are separated from gaseous mixtures by the action of calcium, calcium nitride, or alloys of calcium with magnesium or aluminium. The reagent is previously heated to 200°—650° C. to free it from adherent gases and vapours.

—D. F. T.

Potassium compounds; Apparatus for recovering —. H. Fairbrother, London. From Chemical Construction Co., Los Angeles, Cal., U.S.A. *Eng. Pat.* 136,718, 24.2.19. (*Appl.* 4562/19.)

SEE U.S. Pat. 1,296,462 of 1919; this J., 1919, 362A.

Cyanides; Method of manufacturing —. W. J. Mellersh-Jackson, London. From Air Reduction Co., Inc., New York. *Eng. Pat.* 136,750, 23.4.19. (*Appl.* 10,188/19.)

SEE U.S. Pat. 1,322,195 of 1919; this J., 1920, 109 A.

Chlorine and sulphates from chlorides; Method of producing —. F. Bergius, Essen, Germany, Assignor to Chemical Foundation, Inc. U.S. Pat. 1,326,634, 30.12.19. *Appl.*, 28.10.14. Renewed 21.5.19.

SEE *Ger. Pat.* 299,034 of 1913; this J., 1920, 63A.

Acid-resisting alloy. *Ger. Pat.* 301,721. *See* X.

Ammonium compounds from cyanamide. U.S. Pat. 1,326,045. *See* XX.

VIII.—GLASS; CERAMICS.

PATENT.

Casting pottery and like articles. B. J. Allen, Blythe Bridge, Staffs. *Eng. Pat.* 136,701, 6.2.19. (*Appl.* 2956/19.)

INSTEAD of using a separate vacuum chamber for each mould, as described in *Eng. Pats.* 110,649 and 111,643 (this J., 1917, 1274; 1918, 58 A), one iron

chamber is used and into it are placed moulds of different sizes suspended in horizontal rows or carried on rolled steel girders. Each mould is provided with a separate funnel through which it is filled. The air in the interior of the chamber is then exhausted by means of a vacuum pump, as in the earlier patents.—A. B. S.

IX.—BUILDING MATERIALS.

Timber preservatives. Falck. See XIXu.

PATENTS.

Porous building and insulating bodies of "moler" (kieselguhr, diatomaceous earth) and the like; Production of —. I. N. S. Frydenlund, Frederiksberg, Denmark. Eng. Pat. 123,992, 4.3.19. (Appl. 5358/19.) Int. Conv., 5.3.18.

HEAT-INSULATING and sound-deadening materials are made by mixing organic filling materials, such as the pith of *Helianthus annuus* (sunflower) with diatomaceous earth and then heating the material until all the organic matter is destroyed. The swelling and other disadvantages of other organic filling materials are avoided, as the pith is more compressible.—A. B. S.

Artificial stones [abrasive wheels]; Manufacture of —. H. Sefton-Jones, London. From White Heat Products Co., West Chester, Pa., U.S.A. Eng. Pat. 136,580, 18.3.18. (Appl. 4771/18.)

ARTIFICIAL stones and grinding wheels are made by mixing a natural mineral substance, such as silica, corundum, or a mixture of bauxite and rutile which has been fused and allowed to crystallise, or artificial crystalline mineral substances, such as carborundum, or alundum, with a binder composed of sodium silicate and glass and heating rapidly until the binder is fused. It is claimed that by this means the articles produced do not crack so readily and require a much shorter period of heating than when clay or other binders are used.—A. B. S.

Building brick or block, and method of manufacturing same. C. C. Millard and R. A. Robertson, London, and C. J. Cooper, Chorley Wood, Herts. Eng. Pat. 136,742, 28.3.19. (Appl. 7832/19.)

BUILDING bricks or blocks are made of clinker and/or furnace ashes, with or without brick rubble or burnt clay, and with lime and Portland cement as binders. The clinker, ashes, and burnt clay are reduced to a fine state, mixed with Portland cement and then with lime which has previously been slaked with three times its volume of water so as to form a moist but not slimy mass. This material is moulded into the desired shape and the blocks or bricks are then allowed to dry in the air. The burnt clay makes the bricks waterproof and prevents expansion in the material.—A. B. S.

Cement and soluble potassium compounds; Process of obtaining —. C. Ellis, Montclair, N.J., Assignor to Ellis-Foster Co. Reissue 14,773, 23.12.19, of U.S. Pat. 1,186,522, 6.6.16. Appl., 5.10.18.

See this J., 1916, 843.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Rust-proofing iron and steel; Phosphatic coatings for —. L. E. Eckelmann. Chem. and Met. Eng., 1919, 21, 787–789.

RECENT phosphatic rust-proofing processes do not alter appreciably the dimensions or sharpness of

machined articles, and, due to the low temperatures used, the physical properties of tempered steel are in no way affected. In the process known as "Parkerising" Coslett's solution (Eng. Pat. 8667 of 1906; this J., 1907, 207) is modified by adding an oxidising agent, such as manganese dioxide, to a 0.75% solution of orthophosphoric acid containing ferrous phosphate, which becomes partly oxidised to the ferric state after continued agitation and boiling. The coating formed on iron during the boiling in the phosphoric acid solution is of a uniform dark-grey colour, which varies in intensity according to the surface treatment of the metal previous to immersion. The treated metal, after careful drying, is dipped into a paraffin oil mixture, which changes the coating to a deep black tone. Articles treated by this process are very resistant to saline atmospheres, but are not as resistant to wear as is galvanised or sherardised metal.—C. A. K.

Brass; Influence of cadmium on the properties of —. L. Guillet. Rev. Mét., 1919, 16, 105–115.

THREE series of copper-zinc alloys were studied, containing respectively 70% Cu with 0–4% Cd, 60% Cu with 0–1% Cd, and 55% Cu with 2–10% Cd. With the alloys containing 70% Cu the mechanical properties are not affected by cadmium up to 0.74%. An alloy containing 1.67% Cd possesses about the same breaking strain as when the cadmium content is lower, but the elongation is diminished and the metal becomes brittle. The breaking strain, elongation, and resilience of an alloy containing 1.92% Cd show low values, and the hardness (Brinell) increases until with 4.11% Cd the alloy has practically no mechanical strength, and the hardness attains a maximum. Micrographic examination shows clearly that brass containing up to 0.74% Cd possesses a normal structure (α solution); from 1.67% Cd upwards, increasing quantities of cadmium appear in the free state. Similar results were obtained with the other series of alloys, and it is concluded that up to 1% Cd has no deleterious effect on brass containing 60% or 70% Cu, and that a greater quantity than this influences first the resilience and afterwards the tensile strength. The coefficient of equivalence of cadmium in respect to these alloys is about 0.7. —C. A. K.

Monel metal; Rapid method for the analysis of —. P. Covitz. Chem. and Met. Eng., 1920, 22, 31–32.

THE accumulation of large volumes of filtrates, necessitating considerable evaporation, is avoided by using separate samples for the determination of the different constituent elements. Carbon is determined by direct combustion in a Fleming apparatus (Iron Age, Jan. 1, 1914). For the estimation of silica 1 gm. of Monel metal is dissolved in nitric acid and the solution evaporated to about 7 c.c., diluted to 50 c.c., and filtered. Dehydration of silica is not considered essential for most purposes. Copper is deposited by electrolysis from the filtrate made up to 150 c.c. and acidified with 5 c.c. of sulphuric acid (1:1), using a current of 1.5 amps. for 2 hours. A separate solution of 1 gm. of borings in nitric acid is used for the precipitation of iron and aluminium hydroxides, which are re-dissolved in hydrochloric acid and the iron re-precipitated by means of potassium hydroxide. Iron is estimated by reducing the solution of the precipitate in hydrochloric acid by means of stannous chloride and titrating with permanganate, using a "preventive solution" containing 160 grms. of manganous sulphate, 330 c.c. of syrupy phosphoric acid, and 320 c.c. of concentrated sulphuric acid in 1750 c.c. of water. Manganese is determined by the bismuthate method, and nickel by means of di-

methylglyoxime, care being taken to obtain the proper degree of alkalinity in the latter method.

—C. A. K.

Acid-resisting metals. C. Matignon. *Chim. et Ind.*, 1919, 2, 1283—1302.

THE introduction and development of ferrosilicons for acid-resisting plant are described and the properties and characteristics, especially as regards loss in various acids, of special ferrosilicon alloys are reviewed and tabulated (see Matignon, this J., 1918, 375 A; Tungay, this J., 1918, 87 r). Well-known examples are métillure, élianite, ironac, tantiron, and duriron. The influence of variation in the proportions of iron and silicon has been investigated. Some special applications to particular forms of plant and details in regard to their durability are given and methods of manufacture of the alloys, in so far as these are not secret processes, are described. Some investigations with ferroborens led to the conclusion that these are very inferior in acid-resisting qualities to ferrosilicons.

—W. J. W.

Copper-aluminium-iron bronze as an acid-resisting alloy. O. L. Kowalke. *Chem. and Met. Eng.*, 1920, 22, 37—38.

COPPER-ALUMINIUM-IRON bronzes possessing satisfactory mechanical properties were tested by the loss in weight when subjected, at atmospheric temperatures, to the attack of different acids of 10—50% concentration. A bronze having the composition 87.0% Cu, 9.8% Al, 3.14% “iron alloy” is particularly resistant to sulphuric, lactic, phosphoric, tartaric, and acetic acids, but is attacked by hydrochloric acid and ammonia, and at a rapid rate by nitric acid. By increasing the aluminium content of the alloy 1% (85.6% Cu, 10.8% Al, 3.57% “iron alloy”) the resistance to corrosion is increased materially, though nitric and hydrochloric acids still attack it too readily for commercial application.—C. A. K.

Copper-aluminium alloy; Experience with a 91:9 —. A. I. Krynitzky. *Chem. and Met. Eng.*, 1919, 21, 770—771.

THE preparation of a homogeneous alloy is difficult if more than 9% Al is present. Cast rods of an alloy containing 10% Al almost invariably contained blow-holes and showed segregation along the axis of the bar. Chill casting, at a temperature near to the melting-point of the alloy, is recommended, and the purity of the constituent metals is important. Rods of high aluminium content easily split longitudinally during rolling operations, the fracture often showing almost regular “teeth.” Successful working seems to limit the aluminium to 9%, and an alloy of this composition is readily machined at medium speed.—C. A. K.

Aluminium dross; Evaluation of —. F. K. Bezenberger. *J. Ind. Eng. Chem.*, 1920, 12, 78—79.

TO determine metallic aluminium in aluminium dross 1 grm. of the sample is treated with saturated bromine water, and a small quantity of bromine is added from time to time until the red colour of the bromine is permanent; the free bromine is then expelled by heating, the mixture filtered, the aluminium in the filtrate precipitated as hydroxide by ammonia, and the precipitate collected, ignited, and weighed. It is then treated with 10% sulphuric acid, which dissolves the cupric and ferric oxides present, a small portion of the alumina also passing into solution; after the copper has been deposited electrolytically the iron is reduced and titrated with permanganate solution. The amounts of cupric oxide and ferric oxide are deducted from

the weight of the ignited precipitate and the difference calculated as aluminium.—W. P. S.

Aluminium spontaneously oxidisable in the air. E. Kohn-Abrest. *Comptes rend.*, 1919, 169, 1393—1395.

WHEN aluminium is heated in a vacuum at 1100° C. (in boats of pure aluminium, in a soft iron tube) it slowly distils over, and if the heating is stopped after 16—20 hours it is found that the residual aluminium globules when exposed to the air at the ordinary temperature undergo spontaneous oxidation. The aluminium, which oxidises thus spontaneously, contains as impurities only 0.4% Fe and 0.5% Si.—W. G.

Zinc alloys; Study of cast — with special reference to their use as bearing metals. Gieren. *Z. Metallkunde*, 1919, 11, 14—22. *Chem. Zentr.*, 1919, 90, IV., 964—965.

THE applicability of alloys containing zinc as chief constituent as bearing metals is discussed. Schulz's statement that tin has a detrimental effect on all zinc alloys is not confirmed, but in agreement with Schulz iron was found to be detrimental, and the use of aluminium in place of tin to have a good effect. In the binary zinc-tin-alloys the presence of a eutectic can be detected even when only 0.5% Sn. is present. The melting point of zinc is lowered by 2% Mn to a eutectic point at 400° C. •With further addition of manganese other compounds having transformation points at 570° and 510° C. were formed. Between 0.5 and 1% Mn solid solutions are formed. In the ternary system Zn-Sn-Cu up to 4% Cu and 21% Sn, the constitutional properties of the binary systems Zn-Sn and Zn-Cu appear little changed. Experiments on the technological properties (corrosion, shrinkage, mechanical properties, structure, melting points, bearing properties) of some known and more or less complicated zinc alloys are also described in the original.

—T. H. Bu.

Arsenic; Estimation of — in tin and tin coatings. L. Vallery. *Comptes rend.*, 1919, 169, 1400—1402.

THE results obtained by using Marsh's method for the estimation of arsenic in tin are liable to serious error. If, however, the arsenic is first distilled off as arsenic chloride by the method of Hollard and Bertiaux (this J., 1900, 563), and the arsenic estimated colorimetrically in the distillate after reduction to colloidal arsenic by means of hypophosphorous acid the results are accurate to within 3% of the total at a concentration of 1 in 100,000.

—W. G.

Tungsten; Determination of — in ferro-tungsten. L. Löwy. *Z. angew. Chem.*, 1919, 32, 379—380.

FERROTUNGSTEN is completely decomposed when heated in a platinum or porcelain crucible with ammonium sulphate and concentrated sulphuric acid. The reaction takes place more slowly than in Wolter's process (this J., 1910, 157), but without attacking the porcelain or platinum. The sample is finely powdered in an agate mortar, and 0.5 grm. is heated in a porcelain crucible with 4 grms. of pure ammonium sulphate and 4 c.c. of pure sulphuric acid with frequent shaking until the evolution of sulphur dioxide has ceased, and the temperature is then raised but without igniting the mass, and the heating continued until black particles are no longer visible. After cooling, the mass is dissolved in water, the solution treated with 1 c.c. of concentrated nitric acid, heated to boiling, and, after the addition of 25 c.c. of strong hydrochloric acid (sp. gr. 1.19), boiled for a few minutes. When cold the liquid is filtered and the precipitate of pure tungstic acid washed with cold dilute hydro-

chloric acid (1:5). The filtrate is evaporated until fumes of sulphuric acid appear, cooled and diluted with cold water, and strong ammonia solution added drop by drop until the iron is precipitated. The filtrate is acidified with 10 c.c. of hydrochloric acid and evaporated to dryness, the residue treated with dilute hydrochloric acid (1:5), the solution boiled, cooled, and filtered from the small amount of tungstic acid thus separated, and the precipitate washed as before. For very accurate work a third precipitation may be made. The united precipitates are ignited in a platinum crucible until constant in weight, treated with hydrofluoric acid, and again ignited and weighed. To remove any iron still present the residue is fused with 2–3 grms. of anhydrous sodium carbonate, the mass extracted with hot water, the residue ignited, and the ferric oxide weighed.—C. A. M.

Tungsten metal; Manufacture of pure — C. H. Jones. Chem. and Met. Eng., 1920, 22, 9–16.

The process of manufacture of tungsten as carried on by The Faunsteel Products Co., U.S.A., is described. Hand-picked wolframite containing 73% WO₃ is finely ground in a Braun planetary disc crusher, mixed with 15% excess over the equivalent quantity of soda ash, and the mixture ground in a pebble mill so as to pass a screen with 100 meshes per linear inch. The charge is then roasted in a gas-heated reverberatory furnace at about 800° C., i.e., just sufficient to convert all manganese oxide into the dioxide. Magnesite or dolomite should be used as a lining for the furnace. The fritted mass is leached with water, and calcium chloride is added to the hot filtered solution of sodium tungstate. Calcium tungstate is precipitated and is treated with an excess of hydrochloric acid, and boiled to allow the formation of yellow tungstic acid. After repeated washing this product contains on the average 99.5% WO₃, and is the commercial acid known as Grade A. Further purification is effected by dissolving the tungstic acid in ammonia so as to obtain a 7% solution of ammonium paratungstate, which is filtered through a Sperry filter-press provided with filter paper between the filter-leaves. After evaporating to the consistency of mud the salt is thoroughly washed and treated with concentrated nitric acid. The resulting tungstic acid is calcined in silica crucibles to about 1000° C. The product is of a light green colour, due to a mixture of the yellow oxide (WO₃) and lower blue oxides of tungsten. It contains 99.95% WO₃, and any oxide containing more than 0.02% Fe₂O₃ is returned for further purification. More than 90% of the tungsten in the ore is recovered. A new type of furnace for the reduction of tungstic oxide consists of a silica tube, 4 in. diam. and 5 ft. long, wound with nichrome wire, and surrounded by an insulating casing. Dry pre-heated hydrogen is passed over the heated oxide in the furnace tube, and metallic tungsten is withdrawn as a gray powder, which is formed into bars by a hydraulic pressure of about 300 tons per sq. inch. The bars are sintered by placing them between contact points and subjecting them to a current of 2750 amps. at 220 volts, single phase, in an atmosphere of hydrogen, after which treatment they are drawn down to the required size and cut up by an abrasive cutting wheel.—C. A. K.

Metals and alloys; Fluidity of — D. Saito and K. Hayashi. Mem. Coll. Eng. Kyoto Imp. Univ., 1919, 2, 83–100.

For determining quantitatively the fluidity of metals a flat spiral test-piece is cast in a sand mould. The spiral has a cross-section 10 mm. by 7 mm. and is 5000 mm. long, the distance between the whorls being 25 mm., and the diameter of the outermost whorl 450 mm. The length of the test-piece measured when cold is taken as a measure of the fluidity. The temperature of the molten metal is measured

with a Pt-PtRh couple, and the test-piece is cast under a definite head of metal. The temperature of the mould is maintained between 35° and 40° C. Results are given for lead, zinc, tin, antimony, aluminium, copper, bronze, brass, grey and white iron, and crucible steel. For lead and zinc the fluidity is approximately the same; that of tin is higher. The fluidity of antimony is lower, and that of aluminium less still. The value of copper is about half that of lead. When less than 1.4% of tin is added to copper the fluidity diminishes; above that amount the fluidity increases. Phosphorus increases the fluidity of bronze. The fluidity of brass is about the same as that of bronze, and increases with the zinc content. The fluidity of white iron is approximately one-seventh that of grey iron, and is the lowest of the metals examined. Redcar iron, rich in phosphorus, has a greater fluidity than other grey cast irons examined.

—T. H. Bu.

Froth flotation: its commercial application and influence on modern concentration and smelting practice. W. Broadbridge. Inst. Min. Met., Jan. 22, 1920. [Advance copy.] 58 pages.

Froth flotation is now used to treat some 70 million tons of ore annually. All sulphide ores, including those containing native metals and non-metals, are broadly amenable to froth flotation treatment on a commercial scale. Oxides and carbonates of copper, tin oxide, and lead-silver oxides have also been brought within the scope of the process. Considerable advances have recently been made in differential flotation. Machines for mechanical agitation and sub-aeration are described, also for combining these processes, and for restricted or serial agitation. Flotation units are simple in construction, compact, and flexible, their capacity being large in proportion to their dimensions. Substantially improved recovery of mineral values is obtained by modifying the character of the water by addition of an emulsifying agent during the process of grinding. Pulp concentration is a vital consideration; the most effective water:solid ratio is between 3:1 and 4:1. The size of feed depends on the nature of the ore, and is determined experimentally. Oversize must be limited in quantity, and the quantity of material below 200-mesh size should not fall below a certain percentage. The flotation feed from all portions of the mill must be regular in order to maintain the proper proportions of reagents to feed. The grade of concentrates may be raised by re-treatment in the primary plant without further addition of reagents. This is, however, not possible in sub-aeration plants. The mineral froth is strong, persistent, and sometimes difficult to break down. Moisture in concentrates should be retained in excess of 5% to prevent dusting. Circulation of the plant-water results in a considerable saving of reagents. Ore reduction methods usually comprise two stages—primary crushing followed by secondary crushing. Multi-crushing is avoided. The finest slimes are readily amenable to treatment. Smelting difficulties in regard to flotation concentrates have been overcome. The development of the process has tended towards simplification of operations, flow sheets, and plant. There has been considerable development of labour-saving devices. The process has resulted in considerable saving of initial values, recoveries of over 90% being common where 70% was previously considered good work, and has had a far-reaching influence on the smelting of lead, zinc, and copper.—T. H. Bu.

Flotation; Use of naphthylamine and xylidine in — E. H. Robie. Eng. and Min. J., 1919, 108, 730–732. (See also this J., 1920, 28 A.)

Catene α -naphthylamine or "x-cake" forms a good flotation agent, acting both as a collector and a

frother, and in solution in xylidine excellent results are obtained. Solution takes place rapidly on heating, the usual proportions of the mixture being 60% of "x-cake," 40% of xylidine, although those proportions may be varied considerably to suit different conditions. When used on a bornite-chalcocopyrite ore the following results were obtained:—Feed, 4.5 Cu; tailing, 0.35%; concentrate, 10.78%; recovery, 95.53%. More rapid filtering follows as a higher grade concentrate is obtained, and a smaller amount of concentrate dewatered than when other flotation agents are used.—J. W. D.

Ores and metallurgical products; Treatment of complex —. W. Hommel. Metall u. Erz, 1919, 16, 501—511, 559—576.

THE separation of the individual metals from lead-zinc, copper-zinc, and lead-copper-zinc ores presents considerable difficulties. In some cases zinc blende may be mechanically separated from copper and iron pyrites, but magnetic and electrostatic separation generally give better results. Flotation processes are sometimes combined with roasting or magnetic separation. The dry processes comprise smelting the lead and copper with formation of a zinc-rich slag which is subsequently treated to yield zinc oxide; distillation of the ore to recover the zinc, with the production of lead-rich or copper-rich residues; application of direct blast to materials containing zinc and lead, a method which is only applicable to ores containing little precious metals. Owing to high metal loss in retorts, shaft furnaces are perhaps preferable for the recovery of the zinc by volatilisation. The addition of lime is valuable in the blast process. Wet processes may be grouped according to the solvent used. Sulphuric acid may be used either as concentrated or dilute acid. In the latter case it is usual to start with a low temperature roast and combine with electrolysis. A sulphatising roast or, in some cases, treatment with ferric sulphate may be used to produce zinc sulphate. Another method is the application of sulphurous acid and an oxidising agent. Zinc oxide may be used as an absorbent for sulphurous acid. The separation of zinc with hydrochloric acid or chlorine is advantageous for electrolytic purposes. Separation by means of alkalis and ammonia is not of much value. A solvent should be chosen according to the nature of the ores; the sulphurous acid method is probably the simplest. The best results are obtained by combining the various processes. It is recommended that the zinc should be lixiviated as far as possible before smelting, then treated in a shaft furnace and the slag subjected to blast for separating the remaining zinc. The commercial aspect of the various processes is discussed. Wet processes are not so costly as generally supposed.—T. H. Bu.

Rhodium and hydrogen. Gutbier and Maisch. See VII.

Lead as platinum substitute. Hütter. See XXIII.

Carbon dioxide and water in minerals. Ahmström. See XXIII.

PATENTS.

Ferro-alloys; Production of —. H. L. Sulman and W. B. Ballantine, London. Eng. Pat. 137,080. (Appls. 10,819, 2.7.18 and 19,318, 23.11.18.)

IN a thermo-reducing process for producing ferro-alloys containing chromium and tungsten for high-speed steels, compounds for example, oxides, of tungsten and chromium, containing these metals in chemical combination with each other are used, with or without compounds containing one of these metals and another metal required in the alloy, such as iron. The compounds of chromium and

tungsten are formed by precipitation from a solution of a salt of one of the metals by a salt or compound of the other metal.—J. W. D.

Puddling iron. A. E. White, London. From A. M. Byers Co., Pittsburgh, Pa., U.S.A. Eng. Pat. 136,704, 7.2.19. (Appl. 3066/19.)

A FURNACE and mechanical puddler comprises a tool supported on a carriage which draws the tool backwards and forwards, thereby agitating and "combing" the molten metal on horizontal, longitudinal parallel lines and producing a fibrous stratifying action therein. The furnace has a movable door at one end and longitudinal slots in the walls which are covered with a movable curtain or belt travelling with the supporting carriage. —J. W. D.

Iron; Process for treating —. W. J. Diederichs, Ithaca, N.Y. U.S. Pat. 1,325,626, 23.12.19. Appl., 10.4.17.

WHITE cast-iron castings are heated at about 1000° C. for a period of from 30 mins. to 4 hours, then withdrawn from the furnace and cooled gradually and uniformly.—J. W. D.

High-speed steels not containing tungsten. Stahlwerke R. Lindenberg, A.-G., Remscheid-Hasten. Ger. Pat. 312,311, 27.1.16. Addition to Ger. Pat. 309,175 (this J., 1919, 908 A).

THE steel contains C 0.5—0.8%, Mo 6—10%, Cr 3—6%, Mn 0.2—0.4%, Si 0.2—0.4%, and V 0.15—2%. The vanadium may be partly replaced by cobalt (0.5—3.5%). The addition of vanadium promotes deoxidation and the separation of small slag particles. It also refines the grain which results in greater toughness and better retention of cutting properties. The effect of cobalt is to widen the range of temperature at which the steel may be hardened.—T. H. Bu.

Steel; Process for producing all kinds of — in small basic-lined converters. P. Müller, Frankenberg. Ger. Pat. 314,153, 15.9.17.

WHERE the charge does not contain sufficient phosphorus and silicon for the blow, metallic calcium is either added to the iron or introduced with the blast, the heat of combustion of the calcium serving to raise the temperature of the charge. Owing to the absence of large quantities of silicon the addition of an excess of lime is unnecessary, the duration of the blow is diminished, the loss of iron is reduced, and basic slag containing the usual proportion of phosphorus is obtained. The calcium also combines with nitrogen and sulphur present in the steel.—J. H. L.

Iron and steel; Process for removing scale oxide from the surface of —. W. Fennie, Buhl, Minn, and W. W. Rhodes, Assignors to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,326,585, 30.12.19. Appl., 3.4.19.

THE metal is subjected to the action of an unsaturated solution of sodium bisulphate at 100°—200° F. (38°—93° C.). Normal salts are formed, and sodium bisulphate is added to the solution from time to time until it becomes saturated with salts, and no longer exerts a vigorous solvent action on the scale. The ratio of the normal salts to bisulphate is then decreased to restore the scale-dissolving activity of the solution.—J. W. D.

Blast-furnace air; Method of drying — and apparatus therefor. P. A. Mackay, London. Eng. Pat. 136,957, 17.2.19. (Appl. 3915/19.)

AIR from a blower passes into an acid chamber, containing acid (sulphuric or phosphoric) supplied under pressure by a circulatory system so as to keep its concentration approximately constant.

The air enters by means of a perforated crown submerged in the acid, and on leaving the acid container passes through a chamber containing coke to absorb any acid spray, and is then delivered to the blast furnace either direct or through a blast-furnace stove.—J. W. D.

Steel-making and like furnaces or mixers. Wellman, Seaver, and Heald, Ltd., London, and R. Gray, Redear, Eng. Pat., 137,200, 8.1.19. (Appl. 8890/19.)

A cooling device is used in combination with the port structure consisting of a pipe, or pipes, or passage, or passages, for the cooling medium surrounded by or embedded in an inward extension of the trough of the upper water-seal.—J. W. D.

Flotation apparatus. W. O. Borchardt, Austinville, Va., Assignor to New Jersey Zinc Co., New York. U.S. Pat. 1,326,174, 30.12.19. Appl., 24.1.17.

A gas is introduced into a series of shallow box-like compartments through porous bottoms comprising a supporting diaphragm and a layer of metallic spherules thereon. The ore pulp is subjected to a flotation treatment by causing it to flow longitudinally over the series of compartments, the tailings being deposited at the bend and the ore particles removed as an overflow froth.—J. W. D.

Ore-separating device. A. H. Jones, Tonopah, Nev., U.S. Pat. 1,326,453, 30.12.19. Appl., 11.4.16.

Is a flotation apparatus an upright conduit with means for admitting ore pulp at its lower end and a free annular overflow at its upper end is surrounded by an unobstructed annular perforated medium to receive the overflow. Inside the conduit are mechanical means for agitating the ore pulp and discharging it with an outward swirl upon the perforated medium, and means are also provided for forcing air through the perforated medium into the pulp.—J. W. D.

Copper ores; Process of leaching —. J. J. Nelson, Assignor to O. H. Westergaard and B. Mathews, Los Angeles, Cal. U.S. Pat. 1,326,463, 30.12.19. Appl., 26.3.18.

ORES containing copper sulphate are subjected to the action of a solution containing sodium bisulphate and sodium chloride, the gangue is filtered off, sodium chloride is added to the filtrate and the copper precipitated with finely divided iron.—J. W. D.

Copper; Process for hardening —. F. H. Ruth, Buffalo, N.Y. U.S. Pat. 1,326,693, 30.12.19. Appl., 6.3.18.

A QUANTITY of sulphur is added to molten copper, thoroughly mixed, and the mixture cast in the usual manner.—J. W. D.

Hardness and tenacity of metals and the ease of their working with cutting tools; Process for increasing the —. G. van Oordt, Assignor to Soc. Anon. des Usines Giulini, Basle, Switzerland. U.S. Pat. 1,326,775, 30.12.19. Appl., 30.4.19.

THE metal is heated to a temperature below the usual temperature for annealing.—J. W. D.

Antimony; Alloy to serve as substitute for — in hard lead and similar products. W. Zimmer, Giessen. Ger. Pat. 299,769, 9.1.16.

THE alloy claimed contains arsenic and tin, preferably in equal amounts. It forms alloys with the so-called white metals, lead, tin, antimony, etc., and also with copper, brass, bronze, and tombac. With lead it yields a good bearing metal, and with

suitable additions it may be used as a substitute for Britannia metal, and for the production of acid-resisting hard lead and bronze.—J. H. L.

Hard lead; Substitute for — for acid-resisting objects. W. Stockmeyer, Minden, and H. Hanemann, Charlottenburg, Ger. Pat. 301,721, 9.3.16.

A LEAD-SODIUM alloy containing 0.25% Na is as resistant towards water and sulphuric acid as a lead alloy containing antimony.—D. F. T.

Hard lead; Substitute for —. H. Hanemann, Charlottenburg, and W. Stockmeyer, Minden. Ger. Pat. 305,087, 27.2.15.

LEAD is alloyed with less than 4% of magnesium, and a small proportion of aluminium may be added. Alloys of lead with only 1% of magnesium possess greater hardness and tensile strength than ordinary hard lead containing 11% of antimony. They are not affected by air and water, and only slowly attacked by acids. Addition of a small quantity of aluminium increases the hardness still further.—J. H. L.

Metals; Process for working up alloys containing valuable —. Hirsch Kupfer- und Messingwerke A.-G., Halberstadt. Ger. Pat. 301,265, 18.2.17.

THE alloy is melted down in a known manner, and the resulting slag containing metals it is desired to recover is re-melted and reduced to obtain an alloy, which is afterwards subjected to electrolysis. The subsidiary metals in "black copper" can thus be quantitatively recovered and the copper itself obtained as electrolytic metal. The copper content of the electrolytic bath is preferably maintained above a certain value by addition of copper sulphate as required, and some of the liquor is withdrawn from the bath periodically or continuously to maintain the concentration of subsidiary metals and impurities below a certain limit. The liquor thus withdrawn is worked up for other metals, e.g., nickel. The metals insoluble in acid, e.g., tin, accumulate in the anode sludge.—J. H. L.

Alloys of low and high melting metals in all proportions; Production of —. E. Fernholz, Berlin-Tempelhof. Ger. Pat. 309,114, 23.3.18.

To avoid loss by volatilisation the metals are mixed, melted, cast, and cooled under high pressure until below the melting point of the more fusible component. It is thus possible to alloy mercury, potassium, sodium, cadmium, and zinc with iron, nickel, and manganese.—H. J. H.

Alloys of copper, zinc, and lead; Production of —. Vereinigte Hüttenwerke Burbach-Eich-Düdelingen A.-G., Düdelingen, Luxemburg. Ger. Pat. 314,315, 7.3.18.

COPPER and zinc mixtures in the usual proportions of the brasses (15 to 45% Zn with 85 to 55% Cu) are saturated or mixed with an excess of lead, viz., up to 60% reckoned on the finished alloy. The products resemble, in physical properties, a good phosphor bronze, but differ in the absence of tin and the low proportion of copper. Zinc in large proportion increases the mechanical strength and tends to keep the lead in solution. When the latter is in excess, a portion separates out, imparting plasticity to the alloy. Hardness is increased by the addition of a small proportion of aluminium or other metals.—H. J. H.

Alloys of lead with copper, zinc, and tin; Production of —. Vereinigte Hüttenwerke Burbach-Eich-Düdelingen A.-G., Düdelingen, Luxemburg. Ger. Pat. 314,316, 15.11.18.

THE molten metals are emulsified by heating the

mixture in an induction electric furnace. This effect is produced by the rapidly alternating current and is so pronounced that separation of emulsified constituents may not take place before solidification of the alloy.—H. J. H.

Lead coatings on metals; Production of —. Berlin-Burger Eisenwerk A.-G., Berlin. Ger. Pat. 314,789, 5.10.18.

A smooth durable deposit free from sponginess is obtained by the addition of a small quantity, e.g., 0.01%, of catechu to the plating bath.—D. F. T.

Aluminium; Method for welding or melting —. Chem. Fabr. Griesheim-Elektron, Frankfurt. Ger. Pat. 315,231, 2.5.17.

In order to regulate the melting point of the flux a mixture of various halides of the alkali metals is used; for pure aluminium, a mixture of 4 parts of potassium chloride with 3 of sodium chloride, 1 of lithium chloride, 1 of potassium bromide, and 1 of sodium fluoride, with a melting point near 600° C., is very useful.—D. F. T.

Iron and steel turnings, scrap, and the like; Melting of —. R. A. Hadfield, London. U.S. Pat. 1,326,861, 30.12.19. Appl., 16.9.18.

SEE Eng. Pat. 126,369 of 1917; this J., 1919, 503 A.

[Metallurgical] furnaces; Lining of —. W. C. Ely, Terre Haute, Ind., U.S.A. Eng. Pat. 136,860, 9.9.18. (Appl. 14,647/18.)

SEE U.S. Pat. 1,284,880 of 1918; this J., 1919, 78 A.

Metal maulding. W. J. Kohler, Sheboygan, and A. Brotz, Assignors to Kohler Co., Kohler, Wis. U.S. Pat. 1,325,725, 23.12.19. Appl., 22.1.19.

SEE Eng. Pat. 135,893 of 1918; this J., 1920, 117 A.

Flotation of minerals; Selective —. K. Sundberg, Stockholm. U.S. Pat. 1,326,545, 30.12.19. Appl., 19.3.18.

SEE Eng. Pat. 117,781 of 1918; this J., 1918, 550 A.

Ores; Concentration of —. E. Edser and H. L. Sulman, London. U.S. Pat. 1,326,855, 30.12.19. Appl., 12.5.17.

SEE Eng. Pat. 107,401 of 1916; this J., 1917, 968.

Electroplating and electrocleaning apparatus. A. Ness, Glasgow. U.S. Pat. 1,326,891, 30.12.19. Appl., 22.11.19.

SEE Eng. Pat. 133,162 of 1918; this J., 1919, 868 A.

XI.—ELECTRO-CHEMISTRY.

Electrolytic decomposition of organic acids; Apparatus for — at low temperature and with small volumes of liquid. *Electrolytic decomposition of the alkali salts of citraconic acid.* F. Henrich and W. Schenk. Ber., 1919, 52, 2120—2125.

THE apparatus consists of a small inverted flask, the bottom of which has been removed and the neck of which is provided with a cork holding a small inverted tube. The latter serves the dual function of diminishing the size of the apparatus to the required extent and of allowing efficient cooling by the introduction of a stream of cold water into the tube. As anode, a stout platinum wire, or, preferably, platinum gauze, is placed in close proximity to the tube. The cathode consists of a piece of nickel gauze. The apparatus is easily maintained at 25°—40° C. A gas holder of the type described previously by Henrich (Ber., 1908, 41, 4199) is used for collection of the anode gases, and arrangements are also made for the analysis of these gases at any

desired moment. Electrolysis of concentrated, faintly alkaline solutions of potassium citraconate yields a mixture of oxygen, carbon dioxide, and allylene at the anode. The yield of the latter is favoured by low temperature, high concentration of solution, and high current; it is also found to depend on the particular alkali salt used, increasing from lithium through sodium and potassium to rubidium and then declining when the caesium salt is employed. (See also J. Chem. Soc., 1920, i., 142).—H. W.

Electrometric titrations. Pinkhof. See XXIII.

Hydrogen-ion concentration. Pinkhof. See XXIII.

PATENTS.

Chemical reactions between liquids, gases, or liquids and gases; Method for the electrical treatment of the reagents for —. F. H. A. Wielgolaski, Christiania. Ger. Pat. 304,479, 7.10.15.

THE reaction mixture is submitted to an alternating electric discharge of high frequency between massive porous or perforated electrodes, the reacting or activated substances being in direct contact with the electrode surfaces without the interposition of any other dielectric. The reaction mass may simultaneously be exposed to activating radiation such as Röntgen rays or ultraviolet light. If liquids are being treated, suitable contact substances may also be introduced. The hydrogenation of blubber oil is given as an example.—D. F. T.

Magnetisable cores; Process for the production of —. E. F. Huth, G.m.b.H., and S. Loewe, Berlin. Ger. Pat. 305,048, 30.11.16.

THE magnetisable material (iron, cobalt, nickel, or Heusler's alloy etc.) is atomised and incorporated with insulating material, preferably in the form of alternating layers of the two materials. Thus, for example, a very thin layer of iron may be sprayed upon paper which is then lacquered, a second layer of iron is then sprayed thereon, and so on, until a sheet of the desired thickness is obtained.

—J. S. G. T.

High-tension electrodes for use in the electrical purification of gases. Siemens-Schuckertwerke G.m.b.H., Siemensstadt. Ger. Pat. 314,775, 11.10.18.

A NUMBER of high-tension electrodes are arranged in juxtaposition in the precipitation chamber, and are connected at their lower ends by a transverse conductor serving to bind them together. The floor of the precipitation chamber is made conducting and earthed. An electric field is established between the electrodes and the floor, thereby diminishing the dead space in the chamber.

—J. S. G. T.

Electric furnaces. Armour Fertilizer Works, Chicago, Ill., Assignees of M. Shoeld, Winnetka, Ill., U.S.A. Eng. Pat. 122,828, 6.1.19. (Appl. 364/19.) Int. Conv., 28.1.18.

SEE U.S. Pat. 1,274,794 of 1918; this J., 1918, 662 A.

Electric furnace. F. T. Snyder, Oak Park, Assignor to Industrial Electric Furnace Co., Chicago, Ill. U.S. Pat. 1,325,539, 23.12.19. Appl. 1.3.17.

SEE Eng. Pat. 127,131 of 1918; this J., 1919, 542 A.

Electrical charge of diaphragms; Method of modifying the —. B. Schwerin, Assignor to Elektro-Osmose A.-G. (Graf Schwerin Ges.), Frankfurt, Germany. U.S. Pat. 1,326,104, 23.12.19. Appl., 16.11.14. Renewed 17.10.19.

SEE Eng. Pat. 21,189 of 1914; this J., 1915, 1019.

Odourising apparatus. R. R. Bayeaux and J. Richard, Paris. U.S. Pat. 1,326,631, 30.12.19. Appl., 8.6.18.

See Eng. Pat. 113,606 of 1918; this J., 1919, 119 A.

XII.—FATS; OILS; WAXES.

Momordica seeds; Fat of —. C. E. Corfield and E. Card. Pharm. J., 1920, 101, 43—44.

The seeds of *Momordica cochinchinensis*, a cucurbitaceous plant indigenous to Bengal, the Philippine Islands, etc., have been found to contain on the average 63.3% of kernels. On extraction with petroleum spirit the kernels yielded 47.06% of a greenish fat. A good yield was also obtained by coarsely powdering the kernels and expressing them at about 40° C. in a press surrounded by a steam coil. A sample thus expressed had m. pt. 28°—32° C.; refr. index (40° C.), 1.495; acid value, 1.9; saponif. value, 185.2; iodine value, 23.1. *Fatty acids:* M. pt. 46°—51° C.; solidif. pt., 44°—42° C.; neutralisation value, 188.3; and iodine value about 40. The fat consisted chiefly of glycerides of fatty acids, and no wax alcohols were present. When exposed to the atmosphere the fat gradually became pale yellow and ultimately granular in appearance. When slowly heated to 200°—240° C. the fat was converted into a greenish-brown liquid, which remained liquid and of about the consistence of castor oil on cooling. It then behaved as a semi-drying oil, forming a transparent skin after two days' exposure in a thin layer. In this condition it could probably be used in admixture with drying oils for varnishes and paints.—C. A. M.

Catalytic actions at solid surfaces. II. Transference of hydrogen from saturated to unsaturated organic compounds in the liquid state in presence of metallic nickel. E. F. Armstrong and T. P. Hilditch. Proc. Roy. Soc., 1919, 96 A., 322—329.

EVIDENCE is adduced in support of the view that the catalytic action of metallic catalysts, during the hydrogenation process, is reversible. It is shown that certain saturated organic compounds in the liquid state in the presence of nickel and an unsaturated organic compound at 180°—230° C. undergo dehydrogenation, passing on hydrogen to the unsaturated compound. This work affords an explanation of the results obtained by Moore (this J., 1919, 320 T) during the hydrogenation of unsaturated glycerides. It is suggested that, in the hydrogenation of ethyl oleate, a portion of the freshly-produced ethyl stearate in contact with the nickel undergoes dehydrogenation, the hydrogen liberated being transferred to more ethyl oleate, and the "dehydrogenated ethyl stearate" produced being the ethyl $\Delta^{11,12}$ -oleate isolated by Moore (*loc. cit.*). See further J. Chem. Soc., 1920, ii., 102.)—W. G.

Corn [maize]; Effect of mould on the oil in —. F. Rabak. J. Ind. Eng. Chem., 1920, 12, 46—47.

To determine the effect of mould growth on the oil in maize, a quantity of the latter was placed in a stone jar and inoculated with *Penicillium*; portions were removed at intervals for a period of 90 days and the oil extracted and examined. At the end of 90 days the maize was a dirty gray solid mass which had a strong offensive odour. The acid value, acetyl value, and unsaponifiable matter showed increases, and the iodine value decreased. There was a steady decrease (from 5.58 to 2.02%) in the amount of oil obtained from the maize, indicating that the oil was consumed by the mould.—W. P. S.

Soya bean leaves; Ether extract of —. E. M. Nelson. J. Ind. Eng. Chem., 1920, 12, 49—50.

SOYA BEAN leaves do not form an available source of

oil for paint manufacture, since they yield only 3—6% of ether extract, and the most highly unsaturated compounds in the extract are not oils, but probably cyclic alcohols (phytosterol and stigmasterol) and carotin, which do not form a hard film on drying. W. P. S.

Sulphonated oils [;Determination of moisture in —]. C. F. Barber. J. Soc. Leather Trades Chem., 1919, 3, 206—208.

A REPORT on the work of a committee of the Society of Leather Trades Chemists on the determination of moisture. Heating in an open dish with a free flame gave fair results in the hands of one member, whilst others found long heating at 100° C. unsatisfactory owing to slow decomposition of the oil. Distillation with toluene, commercial petrol, and water-saturated xylene gave identical results; petrol has the advantage of complete insolubility in water. Most workers found medicinal petroleum unsatisfactory on account of too high boiling point. —F. C. T.

Ghedda or East Indian wax. II. Its acids. A. Lipp and E. Kovács. J. prakt. Chem., 1919, 99, 243—255. (Compare this J., 1912, 731.)

MARGARIC acid, a previously unknown hydroxymargaric acid, m. pt. 58° C., and an acid sparingly soluble in ether (see following abstract), with small amounts of formic and butyric acids, were obtained from the wax. (See further J. Chem. Soc., 1920, i., 110.)—J. K.

Ghedda or East Indian wax. III. Hydrocarbons and acids. A. Lipp and E. Casimir. J. prakt. Chem., 1919, 99, 256—258.

THE wax contains approximately 48% of ceryl alcohol, 7% of hydrocarbons (5% of heptacosane, 2% of hentriacontane, with traces of a hydrocarbon of low m. pt.), 24—25% of hydroxymargaric acid, m. pt. 55°—56° C. (see preceding abstract), 1.5—2% of hydroxymargaric acid, m. pt. 71°—72° C. (hitherto unknown), 9—10% of margaric acid, 8—9% of palmitic acid, 2% of "ghedda" acid, and 1% of cerotic acid, with traces of formic, acetic, and butyric acids and tarry matter. Cerotic and ghedda acids are present mainly in the free condition, the others in the form of their ceryl esters. "Ghedda" acid, m. pt. 94.5°—95° C., is given the provisional formula $C_{44}H_{88}O_2$. Its separation depends on its sparing solubility in ether and that of its potassium salt in alcohol. (See further J. Chem. Soc., 1920, i., 111.)—J. K.

Melissyl alcohol and melissic acid. A. Heiduschka and M. Garais. J. prakt. Chem., 1919, 99, 293—311.

CARNAUBA melissyl alcohol and beeswax melissyl alcohol have been characterised by a series of derivatives and shown to have the formulae $C_{30}H_{60}OH$ and $C_{31}H_{62}OH$ respectively. Melissic acid from carnauba melissyl alcohol had m. pt. 90° C. (lead salt, m. pt. 118° C.), whilst that from beeswax had m. pt. 88.5° C. (lead salt, m. pt. 115° C.), and was identical with the product of hydrolysis of carnauba melissyl cyanide. (See further J. Chem. Soc., 1920, i., 131.)—J. K.

Butter and fats of coconut group. Gilmour. See XIXA.

PATENTS.

Oils; Extraction of — by volatile solvents. A. W. MacIlwaine and G. F. Holdcroft, North Ferriby, Yorks. Eng. Pat. 136,870, 9.12.18. (Appl. 20,425, 18.)

OIL-YIELDING material, reduced to a mealy condition, is extracted with benzoline (petroleum spirit)

or other solvent in an extractor, washed, the oil-bearing solvent drained off, and the meal transferred to a closed hopper, at the base of which is arranged a feed apparatus comprising a horizontal sliding box. By oscillatory motion the lower opening of the latter is brought directly above a hydraulic press, into which its contents are discharged. The sliding box then recedes to its former position beneath the extractor, whilst a sliding plate, one portion of which is solid and another portion has an opening corresponding with the plunger, closes the upper end of the press. The plunger then rises and compresses the mass, the oil-charged solvent escaping through the sides of the press and being drained away and collected. The sliding plate is then moved to the position in which it allows passage of the plunger, which is then further raised to place the pressed meal on the horizontal edge of a hopper and directly in the path of the sliding box previously referred to. The sliding box, in its passage forward to recharge the press, the plunger of which is then lowered, discharges the pressed meal to the bottom of a hopper leading to a breaking apparatus, *e.g.*, toothed rolls, from which it passes to a perforated steam-jacketed cylinder fitted internally with a conveyor and agitator to recover the solvent, and thence to a similar cylinder acting as a dryer, being finally discharged past a loaded flap, the material passing entirely through the system without exposure to air. A container, intermediate between the extractor and press, may be employed.—A. de W.

Turkey-red oil; Production of a substitute for —.
Chem. Fabr. Flörsheim H. Noerdlinger, Flörsheim. Ger. Pat. 310,541, 19.12.15.

The so-called "liquid resin," obtained as a waste product in the production of cellulose from pine wood, is distilled under reduced pressure. The distillate contains an oil resembling a fatty oil and a solid acid resembling abietic acid. After the separation of any crystallisable constituents, the distillate is sulphonated with sulphuric acid. The resulting sulphonic acids, which are almost completely soluble in water, give solutions which can be rendered almost neutral, but complete neutralisation gives rise to semi-solid or gelatinous soaps. The solutions form convenient media for the emulsification of all kinds of oils, fats, and organic liquids generally, with water; they can also be applied in the textile industry for mordanting, dressing, and other purposes and to the production of emulsified lubricants and of disinfectants.—D. F. T.

Nickel catalysts; Revivifying exhausted — which have been used in hydrogenation of fats. H. Schlinek und Co. A.-G., Hamburg. Ger. Pat. 314,685, 22.1.16.

The spent catalyst, without previous separation of the last traces of fat, is boiled with a dilute alkaline or alcoholic solution, and is then washed completely free from alkali sulphate. The residue is heated in a current of hydrogen so that the temperature increases at a rate not greater than 150° C. in an hour, and reaches the maximum of 650° C. in not less than 4–5 hours. Under these conditions the nickel particles are not coated with carbon.—L. A. C.

Detergent containing active oxygen; Preparation of a —. Kraemer and Flammer, and L. C. Kelber, Heilbronn. Ger. Pat. 314,590, 1.4.15.

The addition of lysalbinic acid, or protalbinic acid (decomposition products of proteins), or their salts, to a mixture of soap, sodium carbonate, and an oxidising agent such as sodium perborate increases the bleaching action of a solution of the same. For example, a 1% solution of the mixture on heating contains about 50% of the original

active oxygen at 90° C., whereas in the absence of protalbinic acid or lysalbinic acid no active oxygen is present under the same conditions.—L. A. C.

Fats [in barrels]; Method of reducing solid — to liquid, and apparatus used therefor. J. Danin, London. Eng. Pat. 137,252, 5.8.19. (Appl. 19,246/19.)

Hydrocarbons etc. Ger. Pats. 314,745–7. See III.

Chemical reactions. Ger. Pat. 304,479. See XI.

Linseed oil substitute. Ger. Pat. 305,515. See XIII.

Fat-formation in yeasts. Ger. Pat. 307,789. See XVIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Colloidal colouring substances in minerals. Doelter. See VII.

Soya bean leaves. Nelson. See XII.

Balsams. Van Itallie. See XX.

PATENTS.

Lead pigment; Method for making —. J. E. Seeley, Los Angeles, Cal. A. R. Seeley, administratrix. U.S. Pat. 1,325,960, 23.12.19. Appl., 5.2.17. Renewed 6.10.19.

A FINE stream of melted lead is poured into water to obtain the metal in a finely divided condition with a coating of oxide, such divided lead being then subjected to the action of ozonised air, water, and carbon dioxide.—A. de W.

Phenol formaldehyde condensation products. F. J. Robinson, Cowley, Middlesex, and Damard Lacquer Co., Ltd., London. Eng. Pat. 136,298, 20.12.18. (Appl. 21,352/18.)

A PHENOL-FORMALDEHYDE condensation product which is flexible while hot, and can be bent or moulded by heating it to about 120° C., is produced by heating cresol 750 parts, formaldehyde (40%), 675 parts, ammonia (sp. gr. 0.880) 90 parts, and castor oil 50 to 80 parts. The oil may be added at first, or after partial condensation has taken place and the aqueous layer that separates has been removed. The final hardening is carried out in the usual manner by heating in an inert atmosphere. The castor oil may be replaced by camphor, linseed, rape, cottonseed, or other oils.

—L. L. L.

Varnishes or dopes; Manufacture of —. Titanine, Ltd., and P. E. Bowles, Radlett. Eng. Pat. 136,927, 7.1.19. (Appl. 467/19.)

A CELLULOSE ester varnish, to which has been added boric acid or a borate sufficient to produce at least 0.2% of free boric acid, can be stored for several months in a galvanised iron drum with only a negligible drop in viscosity.—A. de W.

Resinous condensation product and process of making same. H. V. Dunham, Mount Vernon, N.Y. U.S. Pat. 1,324,649, 9.12.19. Appl., 4.4.19.

RESINOUS condensation products are obtained by passing a mixture of vaporised di-enes over a heated contact mass, the temperature of the latter being substantially above the melting point of the resinous product.—L. L. L.

[Artificial] resin. M. Darrin, Wilksburg, Pa., Assignor to The Koppers Co., Pittsburgh, Pa. U.S. Pat. 1,326,579, 30.12.19. Appl., 27.7.18.

A NEUTRAL resin is prepared by polymerising by heat compounds which occur in crude solvent naphtha; the clear reddish product has m. pt. above 180° C., and contains less than 0.03 ash and less than 0.06 moisture and volatile matter.—L. A. C.

Resin solutions resembling varnish or gum; Production of — from tar. C. Molhardt, Mödling, Austria. Ger. Pat. 305,065, 19.4.17.

CRUDE tar is diluted with the neutral oils which are removable from it by distillation with steam at 150° C.; after removal of any deposited solids the filtrate is freed from all volatile oils, phenol, naphthalene, and similar substances by distillation with steam at a temperature not exceeding 150° C. The residue from the first distillation may alternatively be treated with drying oils or with suitable oxidising agents and be used as a substitute for drying oils; the distillate contains all the constituents which in ordinary distillation would be collected between 270° and 350° C.; on account of the absence of decomposition, however, the liquid may be applied to purposes for which the use of more volatile fractions is customary.—D. F. T.

Linseed oil substitute. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 305,515, 21.3.17.

CLAIM is made to linseed oil substitutes for varnishes etc., consisting of derivatives of indene in which the double linkage of the cyclopentadiene ring is present: e.g., the oily products obtained from indene or tar distillation fractions containing indene, by introduction of alkyl or aralkyl groups, and in particular monobenzylindene (m. pt. 33°–34° C.), a mixture of mono- and dibenzylindene, xylylated indene, and indene oxalic ester.—J. H. L.

Ink; Solid —. K. Akashi, Tokyo, Japan. U.S. Pat. 1,325,971, 23.12.19. Appl., 14.11.17.

SEE Eng. Pat. 114,601 of 1917; this J., 1918, 313A.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Vulcanisation of caoutchouc and of caoutchouc-like substances; Process for accelerating the —. S. J. Peachey, Davenport. Eng. Pat. 136,716, 21.2.19. (Appl. 4330/19.)

THE vulcanisation of rubber with sulphur is accelerated by the addition of approximately 0.5% of nitrosobenzene or the nitroso derivative of any other cyclic hydrocarbon.—D. F. T.

Rubber; Process of reclaiming —. J. Young, Akron, and W. W. Benner, Cuyahoga Falls, Assignors to The Firestone Tire and Rubber Co., Akron, Ohio. U.S. Pat. 1,321,093, 9.12.19. Appl., 4.4.18.

VULCANISED rubber, "in proper condition," is heated under pressure with xylol and aniline, in presence of a substance capable of combining with or absorbing sulphur.—E. W. L.

Synthetic rubber; Production of highly elastic vulcanisates from —. Mitteldutsche Gummiwarenfabrik L. Peter A.-G., Frankfurt. Ger. Pat. 315,321, 23.1.18.

MIDDLE tar oil is incorporated with synthetic rubber prior to vulcanisation, and imparts a high degree of elasticity to the final product. From three to five times the proportion of the customary

"elasticators" is easily incorporated, the saturation limit being 150–200 —D. F. T.

Rubber and method of obtaining the same. L. E. Barton, Niagara Falls, N.Y., and H. A. Gardner, Washington, D.C. Assignors to The Titanium Alloy Manufacturing Co., New York. U.S. Pat. 1,326,319, 30.12.19. Appl., 2.1.17.

SEE Eng. Pat. 111,257 of 1917; this J., 1918, 11A.

Leather substitute. Eng. Pat. 135,921. See XV.

XV.—LEATHER; BONE; HORN; GLUE.

Gelatin solutions; Structure of —. F. C. Thompson, J. Soc. Leather Trades' Chem., 1919, 3, 209–216.

GELATIN solution of from 0.2 to 0.6 concentration are elastic solids, though apparently quite liquid and mobile. They are composed of a tenuous network of solid gelatin with a continuous phase of water in the interstices. The apparent viscosity of such solutions as found by direct methods is considerable, and is due to rupture of the network. This viscosity is to be distinguished from that of the interstitial water, since diffusing ions and molecules can move in the solutions (and even in jellies) with velocities equal to those in pure water, and are unaffected by the apparent viscosity of the solution. If sugar or a similar substance is added to the water from which the jelly or solution is made, then the true viscosity of the interstitial liquid is raised and the motion of ions etc. is retarded, although the apparent viscosity is little affected. Moreover, gelatin solutions of the concentrations specified will resist indefinitely a small but definite shear, whilst simply viscous liquids such as oils or sugar solutions yield continuously. Double refraction (implying structure) is also shown by gelatin solutions under static stress.—F. C. T.

Sulphonated oils. Barber. See XII.

PATENTS.

Hides or skins; Tanning and liming of —. L. and G. Dufour, Genoa, Italy. Eng. Pat. 125,362, 1.3.19. (Appl. 5124/19.) Int. Conv., 6.4.18.

DELIMING is effected by means of sulphurous acid at 30°–35° C., and the goods are then tanned—preferably at 30°–35° C.—in liquors into which sulphur dioxide is passed to saturation from time to time. To economise tanning liquor some of the excess liquor in the tanning vats may be added to the deliming liquors. It is claimed that a very fine grain is produced, and that the resulting leather is of very good colour.—E. W. L.

Hides; Tanning animal —. W. Clark, London. From Chem. Fabr. Worms A.-G., Frankfurt, Germany. Eng. Pat. 136,193, 8.5.18. (Appl. 7720/18.)

PICKLED skins, freed from hair and prepared by any known method, are treated with a tanning liquor, prepared by dissolving sodium formate (6 kilos.) in water (20 litres), adding the solution to a neutral solution of ferric chloride (5 kilos.) in water (20 litres), and then allowing to stand for some time and diluting with an equal volume of water. Alternatively ferric hydroxide, prepared by the action of ammonia or other alkali on ferric chloride, is dissolved in the calculated quantity of formic acid, and solid iron formate is separated by cooling to about 0° C. The formate is dissolved in water to give a solution of 3° B. (sp. gr. 1.021). In 100 litres of this liquor 100 kilos. of pickled skins are tanned for 4–8 hours, drained for 24 hours, "cleaned" with 1% solution of borax for ½ hr., re-tanned with

sulphite-cellulose extract diluted with three times its volume of water (10° – 12° B., sp. gr. 1.074–1.091), drained for 24 hrs., and dressed like bark-tanned leather.—E. W. L.

Leather; Process of treating — A. J. Hawkins, Montreal, Canada. U.S. Pat. 1,326,261, 30.12.19. Appl., 12.11.18.

LEATHER is covered with a thin coating of oil at about 150° F. (66° C.) and cooled to 80° F. (27° C.) in 12 hrs.; the process is then repeated a number of times.—L. A. C.

Leather compositions [substitutes]. W. Drury, London. Eng. Pat. 135,921, 2.12.18. (Appl. 19,902/18.)

RAW rubber is masticated between hot rollers and an intimate mixture of very finely ground leather (leather flour), filling material, and sulphur is added to it, together with a small amount of factis or similar fatty material. Mastication is continued until the mass is homogeneous, and the temperature of the rolls is then raised to about 240° F. (116° C.) for about 15 mins., in order to effect partial vulcanisation. The mass is passed through a refining mill and formed into thin sheets between calender rolls. Sheet of the requisite thickness is obtained by superposing layers of the thin sheet in alternate directions, and vulcanisation is then completed.

—E. W. L.

Leather substitute; Composition of matter for use as a — J. W. Matthaei, Ingram, Pa., Assignor to Armstrong Cork Co., Pittsburgh, Pa. U.S. Pat. 1,326,681, 30.12.19. Appl., 3.12.18.

A LEATHER substitute contains an excess by weight of rubber, an excess by bulk of cork particles, fibrous materials, reclaimed rubber, sulphur, and glue.

—L. A. C.

Varnished (patent) leather; Method of treating — H. Schulz. Assignor to C. Heyl, Worms, Germany. U.S. Pat. 1,325,415, 16.12.19. Appl., 12.12.16.

VARNISHED leather is subjected to the action of light rich in ultra-violet rays in a closed chamber, through which a current of air, previously heated to above 40° R. (50° C.), is continuously forced.

—E. W. L.

XVI.—SOILS; FERTILISERS.

Soils; Effect of variation in moisture content on the water-extractable matter of — J. C. Martin and A. W. Christie. J. Agric. Res., 1919, 18, 139–143.

Pot experiments were made with two soils, a silty clay loam and a sandy loam, under varying conditions of moisture content from air-dry to just above the optimum content. Analyses were made at intervals during six months. The concentration of the soil solution and the amounts of nitrate and potash were found to tend to be slightly less in the samples near the air-dry condition. The concentration was more markedly depressed in the sandy loam when near the saturation point, the nitrates disappearing entirely. It is concluded that the examination of soils by the water extraction method is not invalidated by variations in moisture content, provided the saturation point is not reached.

—J. H. J.

Sulphur in soils; Effect of oxidation of — on the solubility of rock phosphate and on nitrification. O. M. Shedd. J. Agric. Res., 1919, 18, 329–345.

COMPOST experiments were carried out with various soils mixed with rock phosphate, sulphur, and stable manure. Analyses showed that little solution of

phosphate took place until the compost was inoculated with a small proportion of soil rich in sulphate-forming bacteria. After 2 years' time the amount of total phosphorus rendered soluble in water was 17% and in ammonium citrate solution 84%. This was accompanied by a rise in acidity and formation of sulphate. During the same period 20% of the total nitrogen in the compost was converted into nitrate. The conditions most favourable to the oxidation of the sulphur were found to be warmth, aeration and average moisture content.

—J. H. J.

Carbonates of calcium and magnesium; Natural — in relation to the chemical composition, bacterial contents, and crop-producing power of two very acid soils. S. D. Conner and H. A. Noyes. J. Agric. Res., 1919, 18, 119–125.

IN pot experiments on two very acid soils, a yellow clay and a black sand, wheat, clover and beet were grown with applications of calcite, dolomite, and magnesite at the rates of 4000 and 12,000 parts per million. Although calcium and magnesium compounds were present in both soils, yet the further applications made caused a good crop increase in every case except that of the larger quantity of magnesite on the black sand soil when no growth occurred. Beets tended to be benefited more by magnesium carbonate, while wheat and clover were benefited more by calcium carbonate. The ratios of calcium oxide to magnesium oxide which permitted growth varied from 2:1 to 1:5 on the yellow clay soil and from 3:1 to 1:4 on the black sand soil. The magnesium applications increased the salinity of the soil solution more than the calcium applications, and nitrification also was greater with magnesium. The numbers of bacteria were increased more by calcium in the black sand soil and by magnesium in the yellow clay soil.—J. H. J.

Calcium cyanamide and dicyanodiamide as vegetation factors. E. Linter. Diss., Königsberg, 1917. Bied. Zentr., 1919, 48, 414–417.

PREVIOUS work has shown that dicyanodiamide, which often occurs in crude calcium cyanamide or may be formed by decomposition in the soil, is unfitted for the nutrition of plants and is to be regarded as a poison, its presence resulting in the diminution of field crops and usually in the formation of characteristic white-tipped leaves. In the experiments described, mixtures of dicyanodiamide solution with soils of different characters were kept for some days at 30° C. in presence of carbon dioxide, the mixtures being shaken for seven hours each day. With sand or clay no decomposition of the dicyanodiamide occurred, so that the deleterious effect of the compound persisted until the latter was washed out, this naturally requiring longer in heavy, impervious soils than in lighter ones. In applying calcium cyanamide it is, therefore, advisable to avoid any procedure which would lead to increased formation of dicyanodiamide, for instance, spreading on freshly ploughed land in the autumn or top-dressing in the summer. On the other hand, humic soils are capable of absorbing dicyanodiamide so strongly that the poisonous action gradually disappears, the dicyanodiamide being apparently decomposed, possibly with formation of beneficial nitrogen compounds.—T. H. P.

Waste liquors from the potash industry; Action of — on soils and plants. O. Nolte. Landw. Jahrb., 1918, 51, 563. Bied. Zentr., 1919, 48, 417–426.

THE residual liquors left after crystallisation of crude potassium salts contain, in grms. per litre: $MgCl_2$, 390; $MgSO_4$, 35; $CaCl_2$, 10; $NaCl$, 9; and $MgBr_2$, 1. These liquors effect, especially with rich soils, at first an increase in the crop, but later often

a diminution; this diminution is more marked when the soil is poor in nutrient material and it may be mitigated, but not completely counterbalanced, by copious manuring. The amount of nutrient material taken up from the soil appears, indeed, to be slightly increased rather than diminished by the presence of these liquors. The ash-content of the crop is increased, more magnesia being taken up, although 1% of magnesia, calculated on the dry matter, is never reached; on the other hand, the amount of soda extracted from the soil is diminished in presence of the liquor. In the soil profound changes are produced by the liquor, interchange of bases and liberation of fertilising substances taking place. The amount of nitrogen washed out from the soil appears to be diminished and the same is the case with the phosphoric acid. Increased loss of potash does not occur, except with soils very rich in this constituent; with poor soils, indeed, accumulation of potash is observed. Washing-out of the soda is prevented. The most serious effect produced is the marked de-liming of the soil. The behaviour of the crude potash salts exhibits certain similarities to that of the residual liquor; the chemical composition of the crop remains, however, normal, the absorption of fertilising materials being undisturbed.—T. H. P.

Human urine and desaccharified sulphite-cellulose waste liquors: Plant fertilisation by —. T. Bokorny. Mitt. Deuts. Landw.-Ges., 1919, [1]. Bied. Zentr., 1919, 48, 426—430.

FURTHER experiments confirm the applicability of human urine and of sulphite-cellulose waste liquors as fertilisers (see this J., 1918, 436 A and 1919, 218 A). Laboratory tests with wheat, rye, and beans showed that urine is injurious when diluted to the extent 1:50, but harmless if the dilution is 1:100 or 1:200. The sulphite liquors produced no ill effects when diluted 50 times, or, if previously freed from sugar, when diluted 10 times.

—T. H. P.

Hydrogen cyanide; Effect of — on plants. C. Wehmer. Biochem.-Zeits., 1918, 92, 364—376.

GREEN cress is killed within 10 days by 0.24 mgrm. of hydrogen cyanide per litre of air. In order to achieve the same result with seeds placed in water 71.3 mgrms. of hydrogen cyanide per litre of air is required, although 2.375 mgrms. of the poison per litre of air greatly impairs their germination and development. Green leaves submerged in water are also killed by very dilute solutions of hydrocyanic acid.—S. S. Z.

Nitrates; Do seedlings reduce —? J. Davidson. J. Biol. Chem., 1919, 37, 143—148.

SEEDLINGS do not reduce nitrates of external media as part of their metabolic processes.

—J. C. D.

PATENT.

Seeds: Electrification of — and apparatus therefor. J. J. H. Sturmev, Coventry, and J. Hewett, Hampton, Middlesex. Eng. Pat. 136,920, 2.1.19. (Appl. 84/19.)

SEED after undergoing the Wolfryn electrification process (Eng. Pat. 22,151/13; this J., 1914, 1067) is submitted to a preliminary removal of excess moisture in a centrifugal machine, and is then lifted to the top of a tower furnished with a series of inclined planes set alternately towards each other. While the seed is falling from plane to plane it meets an upward current of air which may be heated and dried if necessary. Instead of a tower a rotating drum may be used in which vanes fixed to the periphery lift the seed and a current of air is drawn through, or the drum may be stationary and the vanes fixed to a revolving shaft.—J. H. J.

XVII.—SUGARS; STARCHES; GUMS.

Sugar cane juice; Colour changes of — and the nature of cane tannin. F. W. Zerbau. J. Ind. Eng. Chem., 1919, 11, 1031—1036. (See also this J., 1918, 778 A.)

It has previously been shown that oxydases and iron are concerned in the colour changes of cane juice. The third factor known to be involved is a water-soluble polyphenol which gives a green coloration with ferric salts. This has now been isolated from the juice and is found to be a true tannin derived, like the oak tannins, from catechol. It is precipitated from cane juice by normal lead acetate, but not by lead nitrate. It is soluble in water, alcohol, acetone, and ethyl acetate, and practically insoluble in ether, benzene, and chloroform. Catechol itself is not present in cane juice.

—J. H. L.

Molasses; Water content of true final cane —. H. C. Prinsen Geerligs. Int. Sugar J., 1920, 22, 34—40.

VAN DER LINDEN (Archief, 1919, 1511) has continued the experiments previously commenced by the author (this J., 1918, 385 A) in order to establish the optimum water content for factory conditions. It was concluded from the results obtained that the optimum water content is about 15—16%, corresponding to a ratio of sucrose to water of 100:161—200, a value in reasonably close agreement with that previously found by the author. It was also apparent that the constitution of some molasses hinders their exhaustion to the same extent as others which are more normal in composition, a low ratio between reducing sugars and ash (1.9—3.2), for example, coinciding with a high purity (39°—43°). In practice boiling and crystallisation are frequently not prolonged sufficiently to permit the maximum yield possible of sucrose to deposit upon the crystals already formed, the result being that after centrifuging when subsequently the temperature of the molasses falls a further deposition of sucrose in the form of very fine grain occurs (cf. also Kalshoven, this J., 1920, 126 A). In general, the procedure that should be followed in the factory in order to obtain molasses from which no further sucrose can be obtained on evaporation is to concentrate the final massecuite having an apparent purity of about 60° until its water content is 15—16%. After discharging from the vacuum pan into crystallisers, the product is maintained for at least 3—4 days in slow and constant motion, while cooling very gradually, finally centrifuging at a temperature of about 45° C. As a check on this operation it is advisable that the final molasses should be examined refractometrically directly after separation and also a week later, following the procedure recently described by Kalshoven (*loc. cit.*).—J. P. O.

Lactose; Catalytic hydrogenation of —. J. B. Senderens. Comptes rend., 1920, 170, 47—50.

IN a repetition of Ipatieff's work (this J., 1913, 107) it is shown that in the catalytic hydrogenation of lactose in aqueous alcoholic solution by means of nickel and nickel oxide, if a weak catalyst is used the action stops when the aldehyde group is reduced to a CH_2OH group, and the product lactositol, $\text{C}_{12}\text{H}_{22}\text{O}_{11}$, m. pt., 78° C.; $[\alpha]_D +12.2^\circ$ in aqueous solution, has been isolated. On hydrolysis with dilute sulphuric acid it yields sorbitol and galactose. If a more active form of catalyst is used the hydrogenation proceeds further and the molecule is split up, the products being sorbitol and dulcitol.

—W. G.

Vacuum-evaporator plant. Corner. See I.

PATENTS.

Decolorising carbon. Eng. Pat. 136,873. See IIb.

Sweetening agents. Ger. Pat. 311,213. See III.

Oxalic acid from sugar. Ger. Pat. 310,923. See XX.

XVIII.—FERMENTATION INDUSTRIES.

Acid formation by moulds and yeasts. F. Boas and H. Leberle. *Biochem. Zeits.*, 1918, 92, 170—188.

THE formation of acid by *Mycoderma*, *Oidium*, and *Aspergillus niger* in media containing various carbohydrates and nitrogenous ingredients has been studied. Most of the acid is formed from the carbohydrates. When, however, an ammonium salt of a strong acid forms the source of nitrogen the liberated acid suppresses the formation of acid from the carbohydrates. The ammonia produced in the fermentation process from the various nitrogenous compounds by means of enzymic action greatly influences the acid formation. The extent of this influence depends on the character of the organism and the nature of the carbohydrate employed.

—S. S. Z.

Glycerol formation in alcoholic fermentation; Natural and artificial —. C. Neuberg and E. Reinfurth. *Biochem. Zeits.*, 1918, 92, 234—267.

By employing sodium sulphite as "fixing" agent for the acetaldehyde formed in alcoholic fermentation an equivalent amount of glycerol is also produced. More than 70% of the theoretical amount of acetaldehyde is thus fixed. The amount of acetaldehyde and glycerol produced increases with the amount of sulphite employed. Acetaldehyde and glycerol are therefore not only by-products but are formed in considerable quantities in certain stages of alcoholic fermentation.—S. S. Z.

Wines; Blue "casse" of —. P. Carles. *Comptes rend.*, 1919, 169, 1422—1423.

THE blue or ferric "casse" of wines cannot be entirely prevented by the presence of sulphur dioxide. The best treatment consists in the addition to the wine of tartaric or citric acid, the amount to be added varying with the intensity of the evil, and in order to determine it the best way is to make a preliminary trial by adding 50 grms. per hectolitre and to increase the amount, if necessary, progressively up to 150 or 200 grms. in the case of certain wines.—W. G.

Soluble starch; Formation of — in selective nitrogen assimilation [by moulds]. F. Boas. *Ber. Deut. Botan. Ges.*, 1919, 37, 50—56. *Chem. Zentr.*, 1919, 90, 111., 888—889.

At a certain hydrogen ion concentration soluble starch is formed from many carbohydrates by *Aspergillus niger*. If in a sugar solution ammonium chloride is consumed as well as amino-acids by the fungus, the liberation of the strongly dissociated hydrochloric acid rapidly increases the hydrogen ion concentration giving conditions favourable for the formation of soluble starch, and the iodine test affords a means of following the selective nitrogen assimilation. In mixtures of ammonium chloride with ammonium phosphate and citrate, with amino-acids and peptone, or with urea, it is the ammonium chloride which is attacked, that is, the mould utilises the least favourable source of nitrogen. With mixtures of nitrogenous substances having different degrees of dissociation, the magnitude of the dissociation controls the absorption into the cell; this absorption is not regulated by the mould, and is the result of purely physico-chemical processes.—W. P. S.

Mould spores; Do — contain enzymes? N. and L. Kopeloff. *J. Agric. Res.*, 1919, 18, 195—209.

A suspension of the spores of the mould under investigation was heated to 63° C. to kill the spores, and then shaken with sand to rupture the spore wall. A measured quantity of this suspension was added to a 10% sucrose solution and incubated at 45° C. After 3 hrs. the solution was examined in a polarimeter and tested for reducing sugar; a decrease in rotation and production of reducing sugar were observed. Upon further incubation for 1 and 4 days increasing changes occurred. If double the amount of the spore emulsion was taken, the amount of change was increased, and with a 20% sucrose solution the changes were more marked. The above results were obtained with the spores of *Aspergillus niger* and *sydowi*, *Aspergillus flavus* and *Penicillium expansum* acted similarly but more feebly. The suspension of spores after heating at 100° C. had no effect on the sucrose solution. The cause of the changes was therefore an enzyme identical with invertase. The upper limit of concentration for the sugar solution was found to be 18—24%, using a suspension containing 100,000—400,000 spores per c.c.—J. H. J.

Catalase of bacteria. M. Jacoby. *Biochem. Zeits.*, 1918, 92, 129—139.

THE catalase of bacteria obtained by the author obeys the same reaction laws as the catalases previously studied (this J., 1918, 557 A, 667 A).

—S. S. Z.

Chinese ang-khak [red rice]. Church. See XIXa.

PATENTS.

Yeasts; Process for stimulating fat-formation in —. Kriegsausschuss für pflanzliche und tierische Öle und Fette, G.m.b.H., Berlin. Ger. Pat. 307,789, 11.8.17.

IN the cultivation of yeast incapable of film formation, for the purpose of producing fats, the yeast is maintained at the surface of the nutrient liquid by means of a diaphragm, so as to realise artificially the conditions of film growth. The nutrient liquid may be charged with oxygen, e.g., by aerating it or treating it with hydrogen peroxide or per-salts before use.—J. H. L.

Yeast; Process of manufacture of pressed — from worts containing little or no sugar. Verein der Spiritus-Fabrikanten in Deutschland, Berlin. Ger. Pats. 314,163—314,166, (A) 24.6.13, (B) 25.6.13, (c) 18.7.13, (d) 11.5.15. Additions to Ger. Pat. 310,461 see Fr. Pat. 472,073 of 1914; this J., 1915, 444).

(A) THE worts are pitched with mixed yeasts, in particular with mixtures of distillery yeasts with mycoderma yeasts. (B) In preparing worts by transforming the sugars present into acid (lactic acid), alkalis are added to combine with the latter. When the acidification process is complete the lactic acid is liberated by means of an organic acid, and the worts are suitably diluted. (C) Worts may be prepared by subjecting to the acidification process waste liquors from the manufacture of cereal starch, or liquors actually containing starch in ungelatinised state, the starch being afterwards recovered from the acid liquors and the latter employed for yeast production. (D) The acidified worts are treated with sugar and inorganic ammonium salts in such quantities that the lactic acid destroyed by the yeast is replaced by the mineral acid liberated by the assimilation of ammonia. A mixture of sugar and inorganic ammonium salts with lactates may be employed for yeast production, the mineral acid liberated from the ammonium salts setting free lactic acid from the lactate for the use of the yeast.—J. H. L.

XIXA.—FOODS.

Milk; Influence of the acidity of — on the velocity of inactivation of the peroxidase by heat. A. Bouma and W. van Dam. *Biochem.-Zeits.*, 1918, 92, 385–397.

THE influence of the hydrogen ion concentration on the inactivation of the peroxidase in milk at 70.55° C. was studied. At 70° C. p_H is 2½–3 times higher than at the ordinary temperature. The addition of so much lactic acid to the milk as was just insufficient to curdle it on pasteurisation had no effect on the velocity constant of the inactivation of the enzyme at that temperature. On the other hand, the addition of sodium hydroxide and ammonia greatly accelerated the destruction of the peroxidase. This confirms the observations previously made by Zilva (this J., 1915, 98).

—S. S. Z.

Lactose in altered milk; Estimation of —. E. Hildt. *Bull. Soc. Chim.*, 1919, 23, 617–621.

A MORE detailed account of work already published (see this J., 1919, 51A).—W. G.

Butter; New method for detecting adulteration in — and for estimating fats of the coconut group. G. van B. Gilmour. *Analyst*, 1920, 45, 2–7.

A METHOD for distinguishing between butter fat and fats of the coconut group has been based on a fractionation of the volatile fatty acids into two groups according to their solubility in saturated brine. Twenty grms. of the filtered fat is saponified, and the volatile fatty acids distilled as in Blichfeldt's method and using his apparatus (this J., 1919, 150D). The distillate is neutralised with N/10 sodium hydroxide solution, a correction of 0.4 c.c. being made for the blank determination, and the neutral soap solution is treated with a quantity of N/10 sulphuric acid equal to that of the sodium hydroxide used ($T + 0.4$), and then with 61 grms. of pure, dry sodium chloride which is neutral to phenolphthalein. The flask is closed and shaken, the contents made up to 200 c.c., and filtered, and 190 c.c. of the filtrate titrated with N/10 sodium hydroxide. The number of c.c. required, multiplied by ¾, and 0.4 (for a blank) deducted from the product represents the soluble volatile acids (S), whilst T–S represents the insoluble volatile acids (I). The variations observed in the case of 12 samples of butter fat were:—T from 26.0 to 33.0; S, from 20.0 to 23.5; and I from 5.0 to 9.5. Six samples of coconut fat gave: T, 19.5 to 22.5; S, 1.3 to 1.8; and I, 18.0 to 20.7. Six samples of palm-kernel fat gave: T, 12.0 to 14.0; S, 1.0 to 1.3; and I, 11.0 to 12.7; and a sample of babassu fat gave: T, 14.3; S, 1.2; and I, 13.1. Using average figures for pure butter, coconut, and palm-kernel fats, the following equations enable the percentage of any one of the three fats in a mixture to be calculated directly:—

(1) Per cent. butter fat = $4.678 - 0.351x$.

(2) Per cent. coconut fat = $51 - 0.38x$.

(3) Per cent. palm-kernel fat = $7.691 - 0.59x$.

Results obtained with mixtures containing arachis oil in addition to the above fats are also given.

—C. A. M.

Cereals; [Determination of] moisture content of —. O. A. Nelson and G. A. Hulett. *J. Ind. Eng. Chem.*, 1920, 12, 40–45.

THE method described is a modification of that used previously by Hulett, Mack, and Smith (this J., 1918, 230A) for the determination of moisture in coals (see also Mack and Hulett, this J., 1917, 378), and consists essentially in heating the material in a very high vacuum produced by a Töpler pump for definite periods of time. The

temperatures employed ranged from 100° to 237° C., and the gases other than water vapour (this was frozen in the receiver surrounded with solid carbon dioxide) evolved at the higher temperatures were collected. It was thus possible to ascertain to what temperature and for what length of time a substance could be heated without yielding an appreciable amount of water due to decomposition. For cereals and cellulose the heating may be continued for 5 hrs. at 180° C. without appreciable decomposition.—W. P. S.

Ang-khak [red rice]; Experiments on the manufacture of Chinese — in the United States. M. B. Church. *J. Ind. Eng. Chem.*, 1920, 12, 45–46.

CHINESE red rice, or ang-khak, is obtained by inoculating moist rice grains with the fungus *Monascus purpureus*, Went.; after a short time, the grains are impregnated with mould hyphae which produce the red colour. The red grains are readily crumbled between the fingers and impart a clear carmine colour to any moist food substance to which they are added. *Monascus purpureus*, Went., although utilised in this way only in China, has been obtained in America from ensilage and “freckled” codfish. All strains of the fungus are not adapted to the production of red rice, since each varies in its physiological activity, its action on rice, and the “richness” of the colour it produces.—W. P. S.

Vitamines of green foods. T. B. Osborne and L. B. Mendel. *J. Biol. Chem.*, 1919, 37, 187–200.

SPINACH, cabbage, alfalfa (lucerne), clover, and timothy plants are not very rich in the water-soluble vitamins, but constitute valuable sources of the fat-soluble factor.—J. C. D.

Foods; Influence of high temperatures and dilute alkalis on the anti-neuritic properties of —. A. L. Daniels and N. I. McClurg. *J. Biol. Chem.*, 1919, 37, 201–213.

THE authors do not agree with McCollum and Simmonds (this J., 1918, 134A) and Chick and Hume (this J., 1918, 17A) that the anti-neuritic vitamin is very unstable. They contend that no great depreciation in the value of foods takes place during the usual processes of canning.—J. C. D.

Jams, preserves and marmalade; [Determination of] insoluble solids in —. C. A. Clemens. *J. Ind. Eng. Chem.*, 1920, 12, 48–49.

TWENTY grms. of the sample is mixed with 100 c.c. of water, boiled for 5 mins., and filtered through an alundum crucible to which suction is applied. The insoluble residue is washed with boiling water, then with alcohol and ether, dried, and weighed.

—W. P. S.

Coconut (Cocos nucifera); Globulin of —. C. O. Johns, A. J. Finks, and C. E. F. Gersdorff. *J. Biol. Chem.*, 1919, 37, 149–153.

A DETAILED method for the preparation of the globulin from the coconut is described. This protein contains a fairly high percentage (15.92) of arginine.—J. C. D.

Jack bean; Globulin of —. J. B. Sumner. *J. Biol. Chem.*, 1919, 37, 137–142.

THE author describes three globulins which he has isolated from this bean. Two are obtainable in crystalline condition. (See further, *J. Chem. Soc.*, 1920, i., 210.)—J. C. D.

Alfalfa [lucerne]; Losses of organic matter in making brown and black —. C. O. Swanson, L. E. Call, and S. C. Salmon. *J. Agric. Res.*, 1919, 18, 299–301.

WHEN partially wilted lucerne is stacked without

curing it undergoes fermentation producing brown and black hay. Analyses of samples from a stack of this nature were made for ash, protein, fibre, and extract, and compared with similar analyses of the original lucerne. It was found that about two-fifths of the organic matter had been lost, and that further losses took place on further standing. Feeding experiments were made with the brown and black material, which showed the black lucerne to be very inferior to the brown and to sound lucerne as a food for cattle.—J. H. J.

Sunflower silage. R. E. Neidig and L. E. Vance. *J. Agric. Res.*, 1919, 18, 325—327.

A CROP of sunflowers was made into silage and examined after four months' storage. At a depth of 9 ft. the colour and odour of the silage were very good, and the amount of volatile acids, chiefly acetic acid, was 3.79%, and of lactic acid 3.45% of the dried sample. The average amount of moisture was 78.5%, ash 2.4%, protein 2.4%, fibre 5.8%, nitrogen-free extract 9.8%, and ether extract 1.1%. —J. H. J.

Pentosans; Modification of the phenylhydrazine method of determining —. P. Menual and C. T. Dowell. *J. Ind. Eng. Chem.*, 1919, 11, 1024—1025.

PHENYLHYDRAZINE may be determined, like hydrazine (see Ebler, this J., 1905, 1257), by interaction with ammoniacal copper sulphate solution and measurement of the volume of nitrogen evolved. The authors applied this reaction to the determination of pentosans, to avoid the use of phloroglucinol, which is now difficult to obtain; the amount of furfural produced was found by precipitating with a known excess of phenylhydrazine, filtering, and determining the excess of phenylhydrazine in the filtrate. As chlorides interfere with the action of the copper reagent the original material was distilled in presence of sulphuric acid instead of hydrochloric acid, the volume of liquid in the flask being kept practically constant by addition of water from time to time. An aliquot part of the distillate was treated with a known excess of phenylhydrazine and agitated for the required period by bubbling carbon dioxide through it. After filtration, an aliquot part of the filtrate (50 c.c.) was introduced by means of a dropping funnel into a 250 c.c. Fresenius nitrogen bulb, which had been previously filled with a 10% solution of ammoniacal copper sulphate and heated to expel the air. A 100 c.c. burette was connected to the tube of the Fresenius bulb, and a glass tube provided with a stopcock connected the neck of the bulb to a Schiff's nitrometer containing sulphuric acid. The 50 c.c. of filtrate was introduced into the bulb through the dropping funnel by closing the cock leading to the nitrometer and lowering the burette. The bulb was then heated to keep the reaction mixture near the boiling point until the reaction was complete. From the volume of nitrogen obtained the excess of phenylhydrazine used was calculated. In 1 grm. of a sample of sorghum 0.1526 grm. of pentosans was found by this method and 0.1523 grm. by the phloroglucinol method; with two other samples still smaller differences were obtained. The time occupied in making a determination of the excess of phenylhydrazine in the filtrate from the hydrazone was 20 mins.—J. H. J.

Citric acid. Broeksmitt. See XX.

PATENTS.

Feeding stuffs from marine plants [seaweed] and processes for manufacturing the same. D. Cronstoe and H. F. Warnecke, Stockholm, Sweden. Eng. Pat. 121,295, 5.12.18. (Appl. 20,197/18.)

SEAWEED is washed, treated with steam under pressure, pressed, dried under reduced pressure, and

ground; the expressed liquor is concentrated at a low temperature under reduced pressure, the crystallised salts are separated in a centrifuge, the liquor added to the ground cake, and the mixture again dried under reduced pressure and pressed into cakes. The product contains, approximately, water 5, proteins 13, digestible carbohydrates 67, fat 1, cellulose 9, and mineral substances 5%.

—W. P. S.

Margarine or the like; Apparatus for use in the manufacture of —. S. H. Blichfeldt, Southall, Middlesex. Eng. Pat. 135,939, 4.12.18. (Appl. 20,079/18.)

IN the manufacture of margarine according to the methods described in Eng. Pats. 4278 and 17,616 of 1913 (this J., 1913, 921; 1914, 883), the heating or working of the material may be carried out in a rotating drum mounted on an inclined axis, the outlet being at the lower end. Means are provided for adjusting the degree of inclination of the axis.

—W. P. S.

Food product containing cocoa. P. Kestner, Paris. Eng. Pat. 136,205, 14.9.18. (Appl. 14,970/18.)

A MIXTURE of cocoa and non-caramelised concentrated sugar-beet juice from which substances having an unpleasant taste and odour have been removed (see Eng. Pat. 135,235; this J., 1920, 76A); the mixture may be flavoured with vanilla, cinnamon, etc.—W. P. S.

Foodstuffs; Animal — [from garbage]. F. W. Gee, Harrow. Eng. Pat. 136,237, 7.12.18. (Appl. 20,363/18.)

A MIXTURE of vegetable and animal or fish offal is heated at 250°—260° F. (121°—127° C.) in a steam-jacketed pan until the moisture content is reduced to about 30%; the mass is then mixed with 20—25% of its weight of farinaceous material (e.g., bakers' sweepings), and the mixture is baked.—W. P. S.

Animal waste; Process for treating nitrogenous —. E. C. R. Marks, London. From C. F. Hildebrandt, Hamburg, Germany. Eng. Pats. (A) 136,853 and (B) 136,854, 14.11.17. (Apples. 16,735 and 16,736/17.)

(A) A MIXTURE of 300 parts of water and 80 parts of hydrochloric acid (sp. gr. 1.15) is heated in a digester, and 100 parts of hair, cartilage, etc., is gradually added. The mass is agitated at 100°—110° C. for three hours. It is then neutralised with soda and concentrated in a vacuum. (B) In order to effect the removal of any substances which would impart a bad smell to the finished product, the raw material is extracted with ether or other fat solvent, or it may be subjected to the action of live steam. The same effect may be produced by submitting the solution obtained by treatment of the waste with acid in the digester to a thorough boiling. The products are similar to meat extracts.

—J. H. J.

Fruit juices; Process for treating —. G. F. Hieber, Spokane, Wash. U.S. Pat. 1,325,094, 16.12.19. Appl., 21.10.18.

THE juice is passed through a preheater into a sterilising vessel provided with a stirrer; on leaving the sterilising vessel the hot juice circulates through a jacket surrounding the preheater, thus heating a fresh quantity of juice in the latter, and is then drawn off, cooled, and stored.—W. P. S.

Bread; Manufacture of leavened —. H. A. Kobman, R. Irvin, and R. J. Cross, Pittsburgh, Pa., Assignors to The Ward Baking Co., New York. U.S. Pat. 1,325,327, 16.12.19. Appl., 11.2.19.

A SUBSTANCE containing diastatic and proteolytic enzymes, produced by the action of a fungus on

carbohydrates and proteins, is added to the yeast, flour, and other ingredients of the dough.

—W. P. S.

Meat extract and the like; Preparation of —. Chem. Ges. Rhenania, Wevelinghoven. Ger. Pat. 315,365, 6.2.17.

CARBON dioxide, under pressure if necessary, is led into the autoclave or other vessel during the extraction. By this procedure the formation of ammonia and decomposition of degradation products of the proteins is reduced, and an extract is obtained with an increased content of amino-acids; this has an important influence on the taste and smell of the extract.—D. F. T.

Bean flour; Process of deodorising and decoloring —. Y. Yamamoto, I. Mizusawa, and T. Kano, Kobe, Japan. Eng. Pat. 136,775, 24.6.19. (Appl. 15,904/19.)

SEE U.S. Pat. 1,314,298 of 1919; this J., 1919, 842 A.

Cheese; Process of sterilising — and an improved product produced by such process. J. L. Kraft, Chicago, Ill., Assignor to J. L. Kraft and Bros. Co. Reissue 14,777, 23.12.19, of U.S. Pat. 1,186,524, 6.6.16. Appl., 18.10.19.

SEE this J., 1916, 861.

XIXb.—WATER PURIFICATION; SANITATION

Bacterium coli; Detection of — in drinking water. J. W. de Waal. Pharm. Weekblad, 1919, 56, 1065—1070.

DIVERSITY of opinion exists in regard to the universal distribution of coli bacilli in drinking water. To some extent the varying experiences may be explained by the different methods of examination employed, as also by the amounts of the samples tested. Further, there is not complete uniformity of opinion in regard to some subsidiary characteristics of *B. coli*, such as indole formation. The chief property distinguishing this bacillus is the decomposition of lactose with formation of gas and acid. The breaking up of the dextrose molecule at 37° C. is not confined to *B. coli*. Tests were carried out with a culture in which ox gall was used instead of sodium taurocholate, and a solution was made of peptone 1%, lactose 0.5%, ox gall 3%, and litmus solution 2.5%. The isolation of the bacilli was effected by means of the same preparation, with the addition of agar, and the test was completed in 96 hours.—W. J. W.

Sewage purified by the activated sludge process; Bacterial flora of —. P. Courmont and A. Rochaix. Comptes rend., 1920, 170, 75—78.

THERE is a considerable reduction of the microbial flora in the activated sludge process of sewage purification. One sample examined before purification contained 202,500,000 microbes per c.c., and only 67,587 after purification. The other sample only examined after purification contained then 58,600 per c.c. All the microbial species left after purification were aerobic, no strictly anaerobic organisms being left, and thus apparently all the putrefactive organisms are destroyed. In both cases the flora remaining was very simple, only seven species being isolated and, of these, five were chromogenic. The purified effluent does not appear to contain either *B. coli* or any other known pathogenic organism.—W. G.

Gentian Violet: its selective bactericidal action. M. L. Crossley. J. Amer. Chem. Soc., 1919, 41, 2083—2090.

COMMERCIAL Gentian Violet is a mixture of equal parts of dextrin and dye, which latter consists

chiefly of hexamethyl- and pentamethyl-*p*-rosaniline hydrochlorides with a small amount of the tetramethyl compound and lower homologues. The selective bactericidal action of Gentian Violet is no greater than that of its component dyes and Gentian Violet has no advantage over these in selective or differentiating power. Organisms which were gentian positive were also gram positive and *vice versa*, the structure both of the dye and the organism being important factors in the selective action. The concentration of the dye is an important factor in determining its power to kill an organism. Not one of the dyes examined was effective against *B. subtilis* in dilutions greater than 1 in 100,000. *B. mycoides* was killed, however, by Malachite Green at a dilution of 1 in 100,000, by resaniline at a dilution of 1 in 200,000, and by Gentian Violet or either of its two principal components at a dilution of 1 in 300,000. The basic nitrogen groups appear to be the reactive groups and their action is intensified by substitution of methyl for amino hydrogen.—W. G.

Timber and plants; Laboratory tests on the value of preservatives for —, and a new solution for spraying plants. R. Falek. Z. angew. Botanik, 1919, [11 and 12]. Chem.-Zeit., 1919, 43, Rep., 313.

PURE cultures of wood-destroying fungi were cultivated on a broth-wart-agar medium, and the rate of growth was measured accurately for different concentrations; portions of the cultures were then added to wood impregnated with various preservatives to determine the efficiency of each as a means for protecting the wood against the fungi. Only strong preservatives in high concentration were found to be sufficiently reactive; sodium fluoride, 2,4-dinitrophenol, dinitro-*o*-cresol, and mixtures of 85–95 parts of sodium fluoride with 15–5 parts of the dinitrophenol or nitro-*o*-cresol, were found to be among the most efficient. These preservatives however, particularly sodium fluoride, when used in dilute solutions, have very little effect upon the spores of saprophytes and parasitic fungi. Copper hydroxide, also, which is much used for spraying plants, is of no use for the preservation of wood. The author recommends a 10% “resinol”-lime or “resinol”-magnesia spray liquor. Either of the liquors can be applied readily to any parts of plants. They are freshly prepared for use by adding either 300 c.c. of calcium chloride solution (sp. gr. 1.14) or 125 grms. of magnesium sulphate in 5 litres of water to 300 c.c. of a concentrated alkaline solution of “resinol” in 5 litres of water. The experimental work has all been carried out in the laboratory, and the results have yet to be tested in actual practice.—L. A. C.

Chloropicrin; Comparative action of — on the weevil and the tribolium. G. Bertrand, Brocq-Rousseau, and Dassonville. Comptes rend., 1919, 169, 1428—1430.

THE tribolium is much more resistant than the weevil to the action of chloropicrin and this affords a means of separating the two parasites. This applies both to the isolated parasites and when they are present in maize.—W. G.

PATENTS.

Water; Process for separating air and other gases from —. C. Hülsmeier, Düsseldorf-Grafenberg. Ger. Pat. 311,336, 3.7.14.

A FILTER-BED for removing air and other gases from water consists of a number of loosely packed layers of iron borings and wood or animal charcoal arranged alternately. Compared with the same quantity of materials arranged as a single layer of iron borings with a layer of charcoal on top, the

series of alternate layers is more efficient, does not exhaust so rapidly, and is more readily cleaned.

—L. A. C.

Sewage sludge; Method of concentrating —. J. Grossmann, Manchester. U.S. Pat. 1,325,787, 23.12.19. Appl., 6.10.14.

SEE Eng. Pat. 22,778 of 1913; this J., 1914, 981.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Nicotine; Quantitative biological method for the estimation of —. H. Fühner. Biochem.-Zeits., 1918, 92, 355—364.

By means of the contraction produced by the action of nicotine on the plain muscle of the leech, it is possible to estimate quantitatively 0.01 mgrm. of the alkaloid. The myograph of the nicotine solution of unknown strength is compared with that of a solution of known strength.—S. S. Z.

Physostigmine; Toxicological detection of —. H. Fühner. Biochem.-Zeits., 1918, 92, 347—355.

QUANTITIES of 0.005—0.01 mgrm. of physostigmine can be detected by means of the biological eye-reaction in the cat. The author finds that in the case of the plain muscle of the leech minute quantities of physostigmine in the presence of acetylcholine increase, the contraction of the muscle brought about only to a small extent by acetylcholine alone. By this joint action of the two drugs on the muscle of the leech 0.0001 mgrm. of physostigmine can be detected with certainty.—S. S. Z.

Digitalis; Pharmacology of compounds resembling — in their activity. M. Walter. Biochem.-Zeits., 1918, 92, 267—282.

THE diffusibility, electrical charge, adsorption, and the reversibility of the action on the heart of compounds resembling digitalis in their activity were studied. No connection between these properties could be established.—S. S. Z.

Polygonaceae; Properties of the —. [Determination of hydroxyanthraquinone derivatives.] A. J. Steenhauer. Pharm. Weekblad, 1919, 56, 1084—1101.

THE botanical structure of the leaves of various plants of the Polygonum order is described. Their chemical components have been investigated to a limited extent only. The author gives methods for extraction of these. Anthraquinone derivatives are found in several species. For the estimation of hydroxyanthraquinone derivatives a colorimetric method is most suitable; 0.6 gm. of powdered material is boiled for 1 hour in an Erlenmeyer flask with a reflux condenser with 15 c.c. of dilute sulphuric acid. After cooling the mixture is transferred to a flask of 125 c.c. capacity, 90 c.c. of benzene is added, and the mixture is well agitated for 3 hours; 3 grms. of tragacanth powder is then added, and after again shaking, 75 c.c. is filtered off (=0.5 gm. material) and shaken with 5% potassium hydroxide. The liquid is made up to a suitable volume, filtered, and its strength determined by comparison with a solution of emodine in 5% potassium hydroxide (1:1,000,000) or a solution of nickel (1:250,000).—W. J. W.

Balsams; Examination of various —. L. van Itallie. Pharm. Weekblad, 1919, 56, 1185—1201.

THE authors have collected, and themselves contribute, many data in regard to numerous balsams, including *balsamum copaiva*, *balsamum toluatanum*,

balsamum peruvianum. Their reactions and properties are tabulated and discussed at some length.

—W. J. W.

Toxins and their antitoxins; Mutual precipitation of —. Application to the titration of antidiphtheric and antitetanic sera. M. Nicolle, E. Debains, and E. Césari. Comptes rend., 1919, 169, 1433—1434.

THE technique is as follows: The filtrates of the diphtheric and tetanic cultures are saturated with anhydrous sodium sulphate, the precipitates obtained being dried *in vacuo* over sulphuric acid and reduced to homogeneous powders. 0.8 gm. of the powder is dissolved in 10 c.c. of distilled water and mixed with an equal volume of a previously melted 10% solution of gelatin in "physiological water." The mixture is distributed in a number of test-tubes, 1 c.c. in each, and cooled in an ice-chest. To these tubes in series is added 1 c.c. of the antitoxic serum at varying dilutions, 1/20, 1/50, 1/100, etc. After two hours at the ordinary temperature the tubes are examined, a positive result being indicated by the appearance of a white disc at the junction of the gelatin and the serum. It is important that the toxic solution and the serum should both be absolutely limpid. Antidiphtheric sera containing 300 units per c.c. and antitetanic sera containing 4000 units per c.c. both give positive results at a dilution of 1/50.—W. G.

Halogenation XIX. Replacement of sulphonic groups by chlorine and the preparation of organic chloro-derivatives. R. L. Datta and H. K. Mitter. J. Amer. Chem. Soc., 1919, 41, 2028—2038.

WHEN chlorine is passed into an aqueous solution of a sulphonic acid the sulphonic group is readily replaced by chlorine with very good yields. In some cases additional chlorination occurs. Aromatic compounds, which in addition contain a hydroxy group exhibit a special facility for this change, and the same has also been found true of amino compounds, such as the nitroanilines. (See further J. Chem. Soc., 1920, i., 157.)—W. G.

Sulphonic groups; Replacement of — by nitro groups by means of nitrous gases. R. L. Datta and P. S. Varma. J. Amer. Chem. Soc., 1919, 41, 2039—2048.

THE sulphonic groups in aromatic compounds may be very easily replaced by nitro groups by the action of nitrous gases. In the course of the action more nitro groups frequently enter with the formation of highly nitrated compounds. The action is generally effected in aqueous solution, the nitrous gases being passed in to saturation, and it may immediately follow the sulphonation without the preliminary isolation of the sulphonic acid. Many nitrations which cannot be brought about at all by nitric acid can be readily accomplished by this method. In general, aromatic compounds which are already substituted by hydroxy groups or by halogens readily undergo this replacement of the sulphonyl group by a nitro group, in some cases more nitro groups entering the ring in suitable positions. In the case of the hydroxycarboxylic acids both the carboxyl and the sulphonyl groups are replaced by nitro groups. If, however, the compound does not contain either substituted halogens or hydroxyl groups, replacement of the sulphonyl group does not take place at all. Thus, benzenesulphonic acid is not affected by treatment with nitrous gases.—W. G.

Nitrous esters and nitro compounds; Direct conversion of — into nitriles by catalysis. A. Mailhe and M. L. Bellegarde. Bull. Soc. Chim., 1919, 25, 588—593.

By passage of their vapours with hydrogen over

reduced nickel at 300° C., or a slightly higher temperature the nitrites (isoamyl, propyl, and heptyl) and nitro-compounds (nitromethane and nitropentane) examined were converted into the corresponding nitriles. The change is not complete, as a certain amount of amine, which escapes dehydrogenation, passes over, and in the case of the nitrates a portion of the ester undergoes hydrolysis by the water formed, giving the corresponding alcohol.

—W. G.

Amines; Iodination of aromatic — by means of iodine and persulphate. K. Elbs and H. Volk. *J. prakt. Chem.*, 1919, 99, 269—275.

VERY limited success has attended the application of this method (this J., 1913, 766) to aromatic amines and their acyl derivatives. The yields of 2-iodo-*p*-toluidine, 2-iodo-*p*-nitraniline, and 2,4-di-iodo-*o*-nitraniline were good; of 2-iodo-*p*-toluidine and 2-iodo-sulphanilic acid moderate; of 2,4-di-iodoaniline (accompanied by *p*-iodoaniline), *p*-iodo-acetanilide, and 3-iodo-*p*-bromoaniline poor. Indefinite products were obtained in other cases. Reference is made to unpublished experiments in which excellent results were obtained with anisole and phenetole. (See further *J. Chem. Soc.*, 1920, i., 154.)—J. K.

Veronal, luminal, and propional; Microchemical reactions of —. L. van Itallie and A. L. W. E. van der Veen. *Pharm. Weekblad*, 1919, 56, 1112—1117.

If small traces of veronal (diethylbarbituric acid), luminal (phenylmethylmalonylurea), and propional (dipropylbarbituric acid) are present in urine they may be isolated by treatment with lead acetate and hydrogen sulphide and obtained in a form suitable for identification by sublimation. Monoclinic crystals of veronal are obtained when its sodium hydroxide solution is treated with an acid, potassium bichromate, or ammonium phosphate. Lead acetate gives an amorphous mass which becomes crystalline on boiling. An irregular crystalline mass of compound crystals is obtained by addition of ammoniacal silver nitrate, and similarly, veronal thallium crystals are produced by treatment of the solution with thallium nitrate. Luminal and propional have been separated in the free state only. Ammonium phosphate causes their separation as drops, which very slowly acquire a crystalline state. Luminal and propional crystals probably belong to the rhombic class.—W. J. W.

Urea and hypobromite. L. Lescœur. *J. Pharm. Chim.*, 1919, 20, 305—314, 343—351, 374—381.

A CRITICAL examination of the reaction between urea and alkaline hypobromite solution showed that there is always a deficit in the quantity of urea found, whether it be calculated from the volume of nitrogen liberated, the carbon dioxide formed, or the hypobromite used in the reaction. The larger the proportion of free alkali to urea the smaller is the deficit, but the theoretical yield is never obtained. The loss appears to be due to the conversion of a small portion of the urea into sodium cyanate.—W. P. S.

Guaiaicol; Specific gravity of —. J. Colman-Nicoresti. *Pharm. J.*, 1920, 101, 26.

VARIOUS samples of guaiaicol had sp. gr. ranging from 1.111 to 1.130 at 15° C., whereas the B.P. standard is 1.16—1.20. The value given by the U.S. Pharmacopœia is 1.110—1.114 at 25° C., whilst other authorities give values ranging from 1.116—1.143. The most correct value is probably 1.143 (Béhal and Choay, this J., 1893, 618), which was obtained with synthetically prepared guaiaicol. Since the commercial product is largely prepared

from cresol, and will probably contain traces of phenol, which are difficult to remove, a somewhat lower sp. gr. than 1.143 should be permissible.

—C. A. M.

Acrolein; Stabilisation of —. V. *Stabilising action of substances with a phenolic group.* C. Mourou, C. Durraisse, P. Robin, and J. Pougnet. *Comptes rend.*, 1920, 170, 26—31. (See this J., 1919, 923 A; 1920, 42 A, 82 A.)

Or the monohydric phenols examined a naphthol showed the strongest stabilising action. The presence of additional hydroxy groups in the nucleus caused a notable increase in the stabilising power, except when two hydroxy groups were in the meta position to one another. The methyl ethers and the acetyl derivatives of the phenols did not show any stabilising action, and this is also true of mono- or polyhydric alcohols. The property is thus peculiar to the phenolic group as such. The stabilising power of the hydroxybenzoic acids was intermediate between that of benzoic acid and the corresponding phenols. Not only do the phenols and particularly the polyphenols inhibit the formation of insoluble resin (disacryl), but they also exert a strong inhibiting effect against the formation of soluble resin, even when the phenols are only present in the acrolein to the extent of 1 in 4000.—W. G.

Citric acid; Detection of free and combined —. T. C. N. Brooksmit. *Pharm. Weekblad*, 1919, 56, 1047—1052.

Citric acid is determined in acetic acid solution by oxidation with permanganate and formation of iodoform. Potassium acetate added to an alcoholic solution gives a clear solution with citric acid, but a precipitate with tartaric acid. Barium acetate precipitates citric acid from alcoholic solution. Details are given for the detection of citric acid in fruit juices and milk, and in its compounds—e.g., in citrates, *citras caffeini*, *citras ferricus et citras chinini*, *pyrophosphas ferricus cum citrate ammonico*, *citrophene*, *migrainum*.—W. J. W.

Carvomenthols; Isomeric — and resolution of inactive carvomenthol into the optical antipodes. I. and H. V. Paolini. *Atti R. Accad. Lincei*, 1919, [v.], 28, ii., 82—85, 131—137.

From the inactive carvomenthol obtained by the reduction of carvenone, the levo-isomeride may be isolated by crystallisation of the less soluble of the two carvomenthol strychnine phthalates and conversion of this into *l*-carvomenthol hydrogen phthalate and the latter into *l*-carvomenthol. The carvomenthol hydrogen phthalate prepared from the mother liquors of the above strychnine salt is syrupy, but when treated with the calculated proportion of ammonia and with silver nitrate, it yields insoluble silver *d*-carvomenthol phthalate and from this, by way of sodium *d*-carvomenthol phthalate and *d*-carvomenthol hydrogen phthalate, *d*-carvomenthol may be isolated. The properties of the various products are described. (See also *J. Chem. Soc.*, 1920, i., 170.)—T. H. P.

Hydrastis, Cola, and Strychnos; Analysis of preparations of —. L. M. van den Berg. *Pharm. Weekblad*, 1919, 56, 1124—1134.

Alcohol or ether from coal distillation gases. De Loisy. See IIa.

Electrolytic decomposition of organic acids. Heinrich and Schenk. See XI.

Iodometric estimation of acids. Kolthoff. See XXIII.

PATENTS.

Calcium citrate; Purification of — and manufacture of citric acid. H. D. Golding, J. Raschen, and United Alkali Co., Liverpool. Eng. Pat. 136,979, 20.3.19. (Appl. 6928/19.)

CALCIUM citrate is purified by treatment with sulphurous acid whereby a solution of citric acid and calcium bisulphite is produced; after the removal of insoluble organic matter, the solution is boiled with resulting loss of sulphur dioxide and re-formation of calcium citrate which separates as a precipitate. From this the citric acid is obtained by decomposition with dilute sulphuric acid. In obtaining citric acid from lime-juice or lemon-juice the solution is treated as above but with calcium bisulphite in place of sulphurous acid in the first operation.—D. F. T.

Cyanamide; Process of converting — into urea and ammonium compounds. S. Giertsen, Odda, Norway. U.S. Pat. 1,326,045, 23.12.19. Appl., 22.8.19.

CYANAMIDE is converted into urea by heating under pressure with aqueous sulphurous acid.—D. F. T.

Urea; Production of — from cyanamide. Farbwerk, Meister, Lucius, und Brüning, Höchst. Ger. Pats. (A) 311,018, 14.3.16, and (B) 311,019, 17.3.16.

(A) THE cyanamide is treated in acid solution with finely divided hydrated manganese dioxide, which is added in quantities smaller than the weight of nitrogen in the dissolved cyanamide. (B) Limited quantities of other hydrated oxides, produced *in situ* if necessary, may be used in place of manganese dioxide; e.g., stannic oxide, introduced in the form of pink salt (ammonium stannichloride), lead dioxide, chromium hydroxide, and ferric hydroxide.—D. F. T.

Camphor; Process for the production of synthetic —. R. L. Andreau, Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,326,248, 30.12.19. Appl., 27.2.17.

ISOBORNEOL is oxidised to camphor by heating to 75°–86° C. with nitric acid free from substances capable of inducing vigorous oxidation of the isoborneol by nitric acid at 40° C.; when the reaction has well started the temperature is reduced materially below 75° C.—L. A. C.

Glanders and oedema; Process for the production of preventive and curative agents for —. M. Piorkowski, Berlin. Ger. Pat. 302,405, 17.2.15. Addition to Ger. Pat. 302,404.

CULTURES of the bacteria of oedema or glanders prepared as described in the chief patent (this J., 1919, 963 A) after being maintained for several days at about 42° C., are heated by stages to 110° C. and dried.—L. A. C.

Oralic acid; Preparation of — from sugar and other carbohydrates. Kinzberger und Co., Prague. Ger. Pat. 310,923, 9.2.16. Int. Conv., 6.7.15.

PREVIOUS to oxidation by one of the customary processes employing nitric acid or oxides of nitrogen and a catalyst, the carbohydrate mass (wood excepted) is submitted to a preliminary treatment with nitrous fumes below 60° C. In this way the rate of the subsequent oxidation is moderated and any sudden development of heat avoided.—D. F. T.

Hydroxytrimethyleneglycine; Preparation of —. H. Krause, Dresden. Ger. Pat. 311,071, 27.9.17.

HYDROXYTRIMETHYLENEGLYCINE of the formula $\text{CH}(\text{OH})(\text{CH}_2\text{NH}\cdot\text{CH}_2\cdot\text{CO}_2\text{H})_2$, an amorphous yellow powder of m. pt. 73° C. (with decomposition) is ob-

tained by the interaction of glycine and formaldehyde in aqueous methyl alcoholic solution at 40° C.; if the glycine is replaced by its salts, e.g., of calcium or barium, the corresponding salts of hydroxytrimethyleneglycine are formed. The product arising from the reaction $2\text{NH}_2\cdot\text{CH}_2\cdot\text{CO}_2\text{H} + 4\text{CH}_2\text{O} = \text{C}_6\text{H}_{12}\text{O}_6\text{N}_2 + \text{H}\cdot\text{CO}_2\text{H} + \text{H}_2\text{O}$, is isolated by evaporation of the solution under reduced pressure, or, in case of the salts, by precipitation by the addition of a suitable organic liquid. Hydroxytrimethyleneglycine and its salts possess powerful germicidal properties, but are innocuous to higher organisms.—D. F. T.

Sulphite-cellulose waste liquors; Extraction of a neutral saponin from —. Chem. Fabr. L. Meyer, Mainz. Ger. Pat. 311,139, 7.8.17.

THE saponin is separated from the waste liquor by the usual methods for saponin precipitation or by acidifying with hydrochloric acid or other mineral acid and adding a concentrated saline solution, e.g. of common salt; the saponin is removed by filtration, washed with a solution of common salt, and dried.—D. F. T.

Sweetening agents. Ger. Pat. 311,213. See III.

Chlorination products of propylene; Process of producing —. E. C. R. Marks, London. From Union Carbide Co., New York. Eng. Pat. 137,247, 2.7.19. (Appl. 16,489/19.)

SEE U.S. Pat. 1,315,547 of 1919; this J., 1919, 848 A.

Acid anhydrides and chlorides; Manufacture of organic —. T. H. Durrans, Oxford, Assignor to A. Boake, Roberts and Co., Ltd., London. U.S. Pat. 1,326,040, 23.12.19. Appl., 10.7.18.

SEE Eng. Pat. 130,399 of 1918; this J., 1919, 739 A.

XXII.—EXPLOSIVES; MATCHES.

Nitrogen; Estimation of — in celluloid and explosives by means of the nitrometer. P. Nicolardot and H. Vourloud. Chim. et Ind., 1919, 2, 1317–1320.

ESTIMATIONS of nitrogen in celluloid and explosives by the nitrometer usually give too low results. It is found that the camphor present reacts with the nitrocellulose in presence of sulphuric acid, and it is advisable therefore to carry out the estimation after precipitating the nitrocellulose from an acetone solution of celluloid by means of benzene, which retains the camphor in solution. Diphenylamine, present as a stabiliser, also causes erroneous results, the nitrogen figure being 0.7% too low.—W. J. W.

Nitro-derivatives of diphenylamine. Van Duin and Van Lennep. See III.

Replacing sulphonic groups by nitro groups. Datta and Varma. See XX.

PATENTS.

Nitro-derivatives of tetrahydronaphthalene and its homologues; Manufacture of —. Tetralin Ges.m.b.H., Berlin. Ger. Pat. 299,014, 17.3.16.

By nitrating tetrahydronaphthalene or its homologues with mixed acid or other nitrating mixtures containing not more than 25% of water, and preventing the temperature of the mixture from rising above 50° C. during the earlier stages of nitration, oxidation and formation of resinous products are almost completely avoided. A mixture of α - and β -nitrotetrahydronaphthalene is obtained by nitrating tetrahydronaphthalene with mixed acid or by the action of nitric acid of sp. gr. 1.47 on the

tetrahydro-compound dissolved in acetic anhydride, a mixture of acetic anhydride and acetic acid, or other indifferent solvent. It is a yellow oil, b. pt. 121° – 128° C. at 0.17 mm. By nitrating with a mixture of 160 parts of nitric acid of sp. gr. 1.17 and 200 parts of sulphuric acid monohydrate, 1,3-dinitrotetrahydronaphthalene is obtained. It forms yellow crystals, m. pt. 71° – 72° C., slightly soluble in alcohol and ether, and may be used in admixture with potassium chlorate, ammonium nitrate, or the like for the preparation of explosives. By further nitration of the dinitro-compound with a mixture of nitric acid and oleum a trinitro-derivative, yellowish-white crystals, m. pt. 80° – 81° C., is obtained.

Incendiary mass. A. Chanard, Rueil, France. U.S. Pat. 1,325,618, 23.12.19. Appl. 15.7.18.

SEE Eng. Pat. 131,641 of 1918; this J., 1919, 796 A.

XXIII.—ANALYSIS.

Lead; Use of — as a substitute for platinum. C. Hütter. Z. angew. Chem., 1919, 32, 380.

BASINS made of soft lead may be used instead of platinum basins for certain quantitative analyses. For example, they may be used for the determination of silicon, aluminium, sodium, and potassium in chrome ironstone, the mineral being decomposed on the water-bath by means of sulphuric acid, any change in the nature of the oxidation products present being prevented by introducing a current of carbon dioxide by means of a porcelain tube inserted into a hole in the cover of the basin. The lead sulphate produced may be separated without interfering with the further course of the analysis. —C. A. M.

Melting point methods at high temperatures. I. L. I. Dana and P. D. Foote. Chem. and Met. Eng., 1920, 22, 23–24.

THE authors deal with the determination of melting points by means of electrical pyrometers, and the primary standardisation of thermo-couples. It is impossible to obtain satisfactory freezing point curves with certain materials, e.g., silicates, on account of extremely slow crystallisation, and the melting point curve, although often poorly defined, must be employed. The differential method of measurement shows much greater sensitiveness to small thermal changes than the simple time-temperature curve. —C. A. K.

Rectification column; Glass — for the rectification of liquid mixtures and the recovery of volatile solvents. W. Elsner. Chem.-Zeit., 1920, 44, 11–12.

THE apparatus, which is constructed entirely of glass, consists of a vertical column, 50 mm. in diameter, and filled with ground or etched glass balls; the lower layers are formed of large balls, above these are layers of medium-sized balls, whilst the upper layers consist of small balls. A dephlegmator is fitted at the top of the column and is provided with a water jacket supplied from a constant-level reservoir. A still-head fits into the top of the dephlegmator and carries a thermometer and a side tube connected to a condenser. Ground-in joints connect the different parts of the apparatus. —W. P. S.

Indicator papers. D. J. de Jong. Pharm. Weekblad, 1919, 56, 465–467.

A DROP of acid placed on ordinary filter paper and treated with methyl orange shows only a weak reaction in the immediate neighbourhood of the drop, and beyond this is a neutral area or "water-ring." By preliminary treatment of the paper with

hydrochloric acid, followed by washing and drying, it becomes saturated with hydrogen ions and absorption of the indicator is prevented. The reaction thereby becomes more sensitive and the whole of the moistened area becomes coloured. Addition of a few drops of hydrochloric acid to methylene blue increases its sensitiveness for the same reason. —W. J. W.

Congorubin; Colour change of — under the influence of reactions and neutral salt action. H. Lüers. Kolloid-Zeits., 1920, 26, 15–20.

IN the presence of hydrogen ions at a concentration $5.5 \times 10^{-2} N$ congorubin changes from red to blue instantaneously, but with decreasing hydrogen ion concentration the rate of change decreases, and at $6.8 \times 10^{-4} N$ practically does not take place. In the presence of neutral salts (KCl) the rate of change is increased about 70 times, so that the presence of neutral salts increases the sensitiveness of the indicator to hydrogen ions by about this amount. In the presence of hydroxyl ions neutral salts decrease the sensitiveness of the indicator until with a concentration of $10^{-2} N$ hydroxyl ions the change does not take place. —J. F. S.

Electrometric titration. J. Pinkhof. Pharm. Weekblad, 1919, 56, 1218–1234.

A COMPREHENSIVE description of the potential method of titration. The apparatus and mode of procedure are described and the theoretical considerations involved are discussed. Details are given of special applications of the method to the estimation of halogens in presence of each other and of heavy metals by means of sodium sulphide; titration of bases using the mercury electrode; determination of the hydrogen ion concentration of liquids by means of the hydrogen electrode; titrations by means of the oxidation potential. A difference in potential of 1 millivolt is easily detected by a capillary electrometer. —W. J. W.

Electrometric titrations. J. Pinkhof. Chem. Weekblad, 1919, 16, 1163–1167.

THE suitability of various constant electrodes for specific cases of electrometric titration is discussed. These include titrations of halogens, of heavy metals with sodium sulphide, and of bases.

—W. J. W.

Hydrogen-ion concentration; Determination of —. J. Pinkhof. Chem. Weekblad, 1919, 16, 1168–1172.

THE author outlines a method for determination of the hydrogen-ion concentration and describes a suitable apparatus for the purpose. The best electrode was found to be a cadmium-amalgam electrode in solutions of cadmium of various concentrations. —W. J. W.

Acids; Iodometric estimation of —. I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 53–68.

THE iodometric method gives excellent results with strong acids, especially if these are diluted and the reagents allowed to stand for ten minutes before titrating. Accurate results are obtained with organic hydroxy-acids after addition of a salt of calcium, magnesium, or zinc. The method is unsuitable for phosphoric acid or weak organic acids not containing a hydroxy group, except formic acid. —W. J. W.

Carbon dioxide; Apparatus for the determination of small quantities of — by displacing it from liquids by a rapid current of air at ordinary temperature and pressure. A. Constantino. Atti R. Accad. Lincei, 1919, [v.], 28, ii., 118–121.

IN the apparatus described purified air is circulated repeatedly through a closed system by means of a

rotary mineral oil pump. The air passes first through the liquid, acidified if necessary, containing the carbon dioxide and then through a definite volume of standard baryta solution, which is subsequently titrated with acid in presence of phenolphthalein. The whole estimation occupies about 45 minutes. Test experiments with sodium carbonate solutions containing 6.4–21 mgrms. of carbon dioxide show that the method yields accurate results.—T. H. P.

Carbon dioxide and water; Micro-analytical determination of — in minerals. G. K. Almström. J. prakt. Chem., 1919, 99, 312–316.

THE author has successfully adapted Pregl's micro-analytical method for the elementary analysis of organic compounds to the determination of carbon dioxide in minerals, using vanadium pentoxide (dried at 300° C.) in place of potassium bichromate; the latter gives low results due to incomplete decomposition of the mineral. Owing to the difficulty of completely removing moisture from the vanadium pentoxide and the relatively large proportion employed (0.2–0.3 gm. per 20–30 mgrms. of mineral), high results are obtained for water. Penfield's method (Amer. J. Sci., 1894, [3], 48, 31) was, however, found suitable for micro-analysis, and gave satisfactory results.—J. K.

Fuel-value of volatile liquids. Barsky. See IIa.

Aromatic hydrocarbons. Tausz. See IIa.

Oil fractionation. Washburn. See II.

"Free carbon" in tar. Waterman and Barkhuysen. See III.

Thiophene in benzol. Meyer. See III.

Water resistance of fabrics. Veitch and Jarrell. See V.

Sulphurous acid. Kolthoff. See VII.

Chlorates. Kolthoff. See VII.

Iodides. Kolthoff. See VII.

Combustible matter in rocks. Fieldner and others. See VII.

Ferrotungsten. Löwy. See X.

Monel metal. Covitz. See X.

Aluminium dross. Bezzenberger. See X.

Arsenic in tin etc. Vallery. See X.

Sulphonated oils. Barber. See XII.

Lactose in altered milk. Hildt. See XIXa.

Butter and fats of coconut group. Gilmour. See XIXa.

Moisture in cereals. Nelson and Hulett. See XIXa.

Insoluble solids in jams etc. Clemens. See XIXa.

Pentosans. Manual and Dowell. See XIXa.

B. coli in water. De Waal. See XIXb.

Nicotine. Fühner. See XX.

Physostigmine. Fühner. See XX.

Hydroxyanthraquinone derivatives. Steenhauer. See XX.

Veronal, luminal, and proponal. Van Itallie and Van der Veen. See XX.

Antidiphtheric and antitetanic sera. Nicolle and others. See XX.

Urea and hypobromite. Lescœur. See XX.

Citric acid. Broeksmit. See XX.

Celluloid and explosives. Nicolardot and Vourloud. See XXII.

PATENT.

Gas-analysing apparatus. N. H. Wener, Assignor to Svenska Aktiebolaget Mono, Stockholm. U.S. Pat. 1,326,814, 30.12.19. Appl., 30.7.18.

See Eng. Pat. 118,114 of 1918; this J., 1919, 744A.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Alexander (United Filters Corporation). Continuous vacuum filters. 2581. Jan. 27.
Barron. Raising liquids. 3321. Feb. 3.
Barron. Sand filters. 3496. Feb. 4.
Bloch. Crushing machines. 3083. Feb. 2.
Bonillon and Worms. Filters for viscid liquids. 2645. Jan. 28.
Bonillon and Worms. Joint for connecting glass tubes or pipes to metal pipes etc. 2996. Jan. 31.
Doherty. Mixing machines. 3662. Feb. 6.
Elmore. Centrifugal driers. 3644. Feb. 6.
Hatfield, and Imperial Trust. Means of separating substances. 3809. Feb. 7.
Inray (Mauss). Centrifugal separators etc. 3474. Feb. 4.
Koppers Co. 2972. See XIX.
Linden. 2695. See XIX.
Mumford. Apparatus for separating solids from liquids. 2637. Jan. 28.
Parr. Separating vapours of volatile liquids from air etc. 2583. Jan. 27.
Philip. Stills. 2836. Jan. 29.
Poore. Apparatus for treating finely divided substances etc. 3212. Feb. 2.
Reavell. Leaching etc. 3134. Feb. 2.
Travers. Furnaces. 2837. Jan. 29.
Wilson. Drying-pans. 3238. Feb. 3.

COMPLETE SPECIFICATIONS ACCEPTED.

10,187 (1919). Kilburn (Sulzer Frères). Refrigerating apparatus. (137,975.) Feb. 4.
10,307 (1919). Rennison. Reversible gas-heated furnaces. (137,977.) Feb. 4.
21,830 (1919). Grey. Mixing apparatus. (138,286.) Feb. 11.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Avellana. Wood gas producer. 2718. Jan. 28. (Spain, 28.1.19.)
Coles. Leading-in wires and filaments for electric lamps. 3248. Feb. 3.
De Bruyn, Ltd., and Revis. Preparation of decolorising charcoals. 3698. Feb. 6.
Deuts. Gasglühlicht A.-G. Producing tungsten incandescent lamps. 3375. Feb. 4. (Ger., 17.2.19.)
Deuts. Gasglühlicht A.-G. Evacuating bulbs of electric incandescent lamps etc. 3491. Feb. 4. (Ger., 18.5.18.)

Ellis (Foundation Oven Corporation). Coke ovens. 2826, 2828, 2829, 2945, 2946, 2947. Jan. 29 and 30.

Ellis (Foundation Oven Corporation). Distillation ovens. 2927. Jan. 29.

Igranic Electric Co. 3503. *See* XXIII.

Muhlfield. Fuel-burning furnaces and methods.

Rendell. Artificial fuel. 3736. Feb. 6.

Tully. Gas-producers. 3499. Feb. 4.

Tully. Manufacture of gas for heating and lighting. 3500. Feb. 4.

Walker. Burning pulverulent fuels. 2704. Jan. 28. (U.S., 15.2.19.)

Williams. Utilisation of combustible gases for heating furnaces etc. 3589. Feb. 5.

COMPLETE SPECIFICATION ACCEPTED.

13,123 (1919). Beswick and Rambush. Working of gas-producers. (138,003.) Feb. 4.

III.—TAR AND TAR PRODUCTS.

APPLICATION.

Burt, Boulton, and Haywood, and Miles. Manufacture of phthalic acid. 3691. Feb. 6.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Bouillon and Worms. 2645 and 2996. *See* I.
Bouillon and Worms. Treatment of cellulose filaments, threads, or films. 2995. Jan. 31.

Bouillon and Worms. Production of cellulose threads or filaments. 2997. Jan. 31.

Bouillon and Worms. Means for twisting cellulose threads or filaments. 2998. Jan. 31.

Gähler. Producing fibre for spinning from papyrus. 2999. Jan. 31. (Ger., 3.7.19.)

Great Northern Paper Co. Paper-making. 2721. Jan. 28. (U.S., 28.3.19.)

Newsprint Reclaiming Corporation. Removal of printers' ink from printed matter. 3342. Feb. 3. (U.S., 1.7.18.)

Stulemeyer. Treatment of viscose. 3116. Feb. 2.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Coles. Decorating textile fabrics. 3246. Feb. 3.
Dowdney and Spurr. Dual-dyed wool. 2994. Jan. 31.

Farrell. Impregnating fabrics with caustic etc. liquor. 2487. Jan. 27.

Norweb. Fireproof, metallising, and waterproofing lace etc. 2890. Jan. 30.

Poulson. Rendering textile fabrics waterproof and fireproof. 2623. Jan. 28.

COMPLETE SPECIFICATIONS ACCEPTED.

5429 (1919). Fish. Bleaching apparatus. (137,947.) Feb. 4.

5538 (1919). Wrigley and Henshilwood. Finishing fabrics. (137,948.) Feb. 4.

10,822 (1919). Ashworth. Machines for dyeing or similarly treating yarns in the form of hanks, skeins, or bundles. (137,980.) Feb. 4.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Chamney. Manufacture of ammonium polysulphide. 2766. Jan. 29.

Kereszey and Wolf. Production of solid basic magnesium hypochlorite. 3206. Feb. 2. (Hungary, 19.4.19.)

Sheard. Making neutral sulphate of ammonia. 2880. Jan. 30.

Wilton. Manufacture of neutral dry ammonium sulphate. 3308. Feb. 3.

COMPLETE SPECIFICATIONS ACCEPTED.

6999 (1919). Ekeley and Stoddard. Method of obtaining pure tungstates. (138,211.) Feb. 11.

13,108 (1919). Amsterdamer Superfosfaatfabriek. Manufacture of salts of ammonia. (138,002.) Feb. 4.

23,375 (1919). Datta. Manufacture of alkali chromates and iodine. (138,291.) Feb. 11.

VIII.—GLASS; CERAMICS.

APPLICATION.

Travers. 2837. *See* I.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Arent. Protective treatment of materials. 3537. Feb. 5. (U.S., 5.2.19.)

Bell and Haw. Method of burning cement clinker. 2533. Jan. 27.

Maguet. 2433. *See* X.

Ramsey. Manufacture of concrete bricks or blocks. 3725. Feb. 6.

COMPLETE SPECIFICATIONS ACCEPTED.

20,513 (1918). Boehm and Reihl. *See* XIII.

1653 (1919). Coderre and Kynoch. Preparation of timber for impregnation. (137,911.) Feb. 4.

15,534 (1919). Illemann. Composition for use as a dustproof and waterproof road or like covering material. (138,017.) Feb. 4.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Aktieselskabet Zink, and Raeder. Production of zinc. 3279. Feb. 3. (Norway, 15.2.19.)

Cobbett and Slec. Furnace converter for oxidation of zinc, tin, etc. 3135. Feb. 2.

Coles. Process for hardening steel and iron. 3094. Feb. 2.

Coles. Vapour galvanising or sherardising. 3095. Feb. 2.

Coles. Electro-deposition of iron. 3524. Feb. 5.
Eklund. Recovering tin from stanniferous waste products. 3181. Feb. 2. (Sweden, 1.2.19.)

Fletcher. Electro-deposition of metals on iron and alloys of iron. 2710. Jan. 28.

Heraeus Ges. Vacuum melting and refining metals and alloys. 3594. Feb. 5. (Ger., 11.1.18.)

Heraeus Ges. Purifying and refining iron. 3731. Feb. 6. (Ger., 11.4.18.)

Higginson. Flux for soldering. 2798. Jan. 29.

Kitto. Process for reducing tin ores. 3709. Feb. 6.

Lang. Manufacture of iron and steel. 3672. Feb. 6.

Lyell, Lyell, and Munro. Solder for aluminium and its alloys. 3632. Feb. 6.

McConway and Vigeon. Pre-leaching apparatus for treating calcined ore etc. 3692. Feb. 6.

Maguet. Reducing fused slag to granular form. 2433. Jan. 26.

Newton. Solder. 3372. Feb. 4.

Rare Metals Reduction Co. Alloys, and processes of producing same. 2580. Jan. 27. (U.S. 7.12.15.)

Thomas. Magnetic materials, and method of manufacturing the same. 2442. Jan. 26.

Walther. Method of smelting whereby pure metal is obtained from concentrated ore in one operation. 3317. Feb. 3.

COMPLETE SPECIFICATIONS ACCEPTED.

18,974 (1917). Basset. Treatment of ores in the blast furnace. (112,275.) Feb. 11.

3464 (1919). Grondal. Lixiviating and washing granular or pulverulent ore. (137,930.) Feb. 4.

9460 (1919). Mumford, Ltd., and Mumford. Alloy, and method of making and using the same. (138,228.) Feb. 11.

15,471 (1919). Comp. des Forges et Aciéries de la Marine et d'Homécourt. Heating air to be supplied to blast furnaces. (128,582.) Feb. 4.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Coles. 3524. *See* X.

Etchells and Greaves. Electrical resistance furnaces. 2372. Jan. 26.

Fletcher. 2710. *See* X.

Norske Aktieselskab for Elektrokemisk Industri. Suspension of electrodes in electric furnaces. 2657. Jan. 28. (Norway, 28.1.19.)

Tagliaferri. Electric furnaces. 3217. Feb. 2.

COMPLETE SPECIFICATION ACCEPTED.

19,383 (1919). Olsen. Material for the manufacture of electrodes of electric furnaces. (138,272.) Feb. 11.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Belton. Continuous hydrogenation of unsaturated oils, fats, etc. 2410. Jan. 26.

Bolton and Lush. Activating and maintaining activity of metallic catalyst during hydrogenation of oils, fats, etc. 2544. Jan. 27.

Clayton and Nodder. Manufacture of edible fats. 3814. Feb. 7.

De Bruyn, Ltd., and Revis. 3698. *See* II.

Gadal. Decomposition of fatty acid glycerides by sulpho-aromatic decomposing agents. 3694. Feb. 6. (Norway, 29.4.18.)

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATION.

Ioco Proofing Co., Vickers, Ltd., and Nuttall. Condensation of phenolic bodies with aldehydic compounds. 3191. Feb. 2.

COMPLETE SPECIFICATIONS ACCEPTED.

16,335 (1918). Morris. Reparation of antimony colours. (138,137.) Feb. 11.

20,513 (1918). Boehm, Ltd., and Reihl. Manufacture of paint oils suitable for cement and concrete. (137,878.) Feb. 4.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATION.

Wood. Method of puering or bating skins. 3406. Feb. 4.

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Edwards. 2838. *See* XX.

Sams. Manure, and method of making same. 3687. Feb. 6.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATION.

De Bruyn, Ltd., and Revis. 3698. *See* II.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Coressy. Diastimeters. 2676. Jan. 28.

Fawsitt. Process of finishing beer. 3101. Feb. 2.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Barron. 3496. *See* I.

Chamney. 2766. *See* VII.

Chamney. Fungicides. 2767. Jan. 29.

Clayton and Nodder. 3814. *See* XII.

Edwards. 2838. *See* XX.

Ges. f. Verwertung Chem. Produkte. Producing exchange materials for purifying air. 3645. Feb. 6. (Ger., 22.6.16.)

Jackson. Coffee substitute. 3792. Feb. 7.

Kingzett, and Sanitas Co. Manufacture of disinfectants. 3734. Feb. 6.

Koppers Co. Purification of liquids. 2972. Jan. 30. (U.S., 15.2.19.)

Law. Formaldehyde-producing apparatus. 3301. Feb. 3.

Linden. Apparatus for treating effluent etc. and for separating and recovering matters of different densities. 2695. Jan. 28.

Schwartz and Schwartz. Preparations for utilising dried fruits and concentrated fruit juices. 2980. Jan. 31.

COMPLETE SPECIFICATION ACCEPTED.

22,274 (1919). Brechot. Refuse destructor furnaces. (133,297.) Feb. 11.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Burt, Boulton, and Haywood, and Miles. 3691. *See* III.

Edwards. Manufacture of arsenical compounds and preparations for treating parasitic infections of animals and plants. 2838. Jan. 29.

COMPLETE SPECIFICATIONS ACCEPTED.

22,095 (1919). Hall Motor Fuel, Ltd. (Dunham). Production of polymerised compounds from unsaturated hydrocarbons. (138,046.) Feb. 4.

22,766 (1919). Bayer und Co. Manufacture of derivatives of bromoacylised urea compounds. (132,795.) Feb. 11.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Bloch, Renwick, Storr, and Ilford, Ltd. Radio-sensitive materials. 3675. Feb. 6.

Gleichmar. Three-colour photography. 2769. Jan. 29. (Ger., 25.7.19.)

XXII.—EXPLOSIVES; MATCHES.

APPLICATION.

Kostevitch. Means for testing condition of smokeless powder and explosives. 3283. Feb. 3.

XXIII.—ANALYSIS.

APPLICATIONS.

Igranic Electric Co. (Cutler-Hammer Manufacturing Co.). Determining calorific value of combustible fluids. 3503. Feb. 4.

Svenska Aktiebolaget Mono. Gas-analysing apparatus. 2680. Jan. 28. (Sweden, 29.1.19.)

COMPLETE SPECIFICATION ACCEPTED.

1716 (1919). Cossor. Apparatus for ascertaining the composition of furnace gases. (133,156.) Feb. 11.

I.—GENERAL; PLANT; MACHINERY.

Gases; Absorption of — in spray systems and towers. E. M. Baker. Chem. and Met. Eng., 1920, 22, 122—124.

CONSIDERING the general case of the absorption of a gas in a liquid which exerts an appreciable vapour pressure due to the dissolved gas, the theoretical absorption (K) under given conditions may be increased by applying any means which increases the partial pressure of gas in the mixture, or reduces the vapour pressure of the gas in the liquid. The rate of absorption depends on the difference (D) between the partial and vapour pressures, on the degree of contact (I) between the liquid and the gas mixture, and the time of contact (T). The actual absorption (P) for any gas and liquid may be represented: $P = CKDIT$, C being a constant. K and D will be fixed generally by plant conditions, and I and T by the design and operation of the plant as a whole. The formula is not capable of direct mathematical application, but may be useful in the comparison of various systems of absorption.—C. A. K.

PATENTS.

Shaft furnaces and gas producers. E. F. Chaudière, Paris. Eng. Pat. 137,168, 3.3.19. (Appl. 5220/19.)

THE furnace is of the type used in the production of lime and cement, for roasting and calcining ores, and for blast furnaces, gas producers, etc. The vertical furnace is provided with a central vertical core consisting of two concentric cast-iron columns connected by radial ribs, the lower part of the core being surrounded by another hollow column, and the upper part by refractory masonry containing recuperating flues. A fan situated at the bottom of the central core exhausts the combustion products and thus causes a flow of air upward through the central column, where it is preheated, and air is also drawn upwards through some of the surrounding passages. Part of the latter air current passes into the furnace through openings about the middle of its height, and the remainder continues upward to meet the central current at the top. The hot air then passes downward through other flues in the core, and is also discharged into the furnace about the middle of its height. A strongly heated zone is thus formed in the furnace, and the position of this zone is stabilised. Air is also admitted to the lower part of the furnace through additional flues in the central core, and preheating flues are also provided in the outer casing of the furnace.—W. F. F.

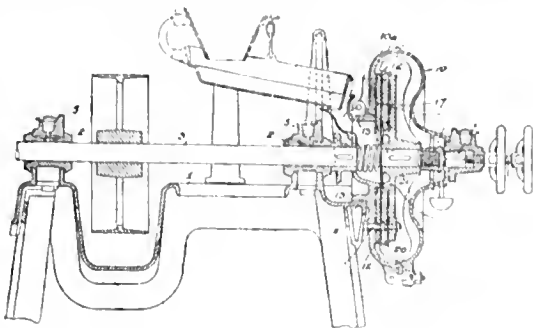
Drying apparatus. A. Nordstrom and C. A. Oberg, Buenos Aires, Argentina. U.S. Pat. 1,326,525, 30.12.19. Appl., 23.10.17.

A HORIZONTAL cylindrical casing rotating on its axis is provided with several inner concentric cylindrical partitions attached to the two end walls alternately so as to form a continuous passage. Longitudinal ribs may be provided on the inner walls of some of the partitions, and also helical ribs of opposite thread in every two successive drums. The material is fed into the innermost compartment which is exhausted, and is discharged at the outer periphery of the casing to an elevator formed integrally with the casing. The air between the various partitions is heated by heating coils.—W. F. F.

Grinding mills of the rotating disc type. F. Corbett, Rhyl, Wales. Eng. Pat. 137,231, 5.6.19. (Appl. 14,249/19.)

THE object is to secure the accurate setting of the

fixed disc in a grinding mill having a rotating disc coacting with a fixed disc. The driving shaft, 3, is carried in bearings, 2, supported by the bed-



plate, 1. The bedplate is also formed with a socket, 11, to receive the back plate, 10a, of the casing, 10, which supports the fixed disc, 12; the socket is bored out at the same time as the bearings, 5, to ensure concentricity. The face, 17, of the casing back, 10a, is machined true with the cylindrical part, 13, so that when the fixed disc, 12, is secured to it the grinding face will be accurately transverse to the shaft, 3. The rotating disc, 20, is fixed to a chuck, 21, which is keyed to the shaft, 3, the chuck, 21, being bored and faced at the same operation.—W. F. F.

Oil emulsions; Apparatus for the treatment of —. C. W. McKibben, Houston, Tex. U.S. Pat. 1,327,835, 13.1.20. Appl., 18.12.18.

THE separation of water from an emulsion with oil is effected by making the mixture follow a tortuous path between two vertical concentric pipes in a tank; the space between the pipes is occupied by a series of discs with openings near their axes and their peripheries alternately; the inner pipe may be heated.—D. F. T.

Gas and liquid contact apparatus. G. E. Darier, Chêne, Switzerland. U.S. Pat. 1,327,422, 6.1.20. Appl., 30.12.16.

SEE Eng. Pat. 101,163 of 1916; this J., 1917, 1170.

Caustic soda or the like; Machine or apparatus for breaking up or pulverising —. W. Blacker, Stalybridge. U.S. Pat. 1,327,901, 13.1.20. Appl., 4.1.19.

SEE Eng. Pat. 120,309 of 1918; this J., 1918, 766 A.

Filter. E. Zahm, Assignor to Zahm Manufacturing Co., Buffalo, N.Y. U.S. Pat. 1,328,221, 13.1.20. Appl., 25.5.17.

SEE Eng. Pat. 108,897 of 1916; this J., 1917, 1086.

Cooling-plate for continuous rectification apparatus. E. A. Barbet, Paris. U.S. Pat. 1,328,259, 20.1.20. Appl., 26.3.17.

SEE Eng. Pat. 105,549 of 1917; this J., 1918, 197 A.

Atomising materials in a melted state; Process and apparatus for —. E. Odum, Paris. U.S. Pat. 1,328,416, 20.1.20. Appl., 2.1.19.

SEE Eng. Pat. 122,103 of 1918; this J., 1920, 113 A.

Mixing apparatus for granular, powdered, or like material. W. S. H. Bevin and J. S. Rawsthorne, Liverpool. U.S. Pat. 1,328,486, 20.1.20. Appl., 26.6.17.

SEE Eng. Pat. 101,332 of 1916; this J., 1916, 1052.

Electrical separation. Eng. Pat. 116,104. See XI.

Electrical purification of gases. Ger. Pat. 314,030.
See XI.

II.A.—FUEL; GAS; MINERAL OILS AND WAXES.

Asphalt and coal; Origin of — J. Marcusson.
Chem.-Zeit., 1920, 44, 43–44.

NATURAL asphalts are derived from petroleum by evaporation of the lighter fractions, and by the partial polymerisation and condensation, or oxidation, or decomposition by sulphur and sulphur compounds, of the residual hydrocarbons. Polynaphthenic acids form a link between petroleum and asphalts, and are distinguished from naphthenic acids by the insolubility of the copper salt in hydrocarbons. They are brownish-black compounds of a consistence varying from that of thick oils to that of resins. They are soluble in alcohol, glacial acetic acid, and chloroform, but insoluble in benzene. Their specific gravity is above 1.0. The alkali salts are soluble in water; those of the alkaline-earths and heavy metals are insoluble. Their iodine value is approximately 20. They are probably formed by combination of two molecules of a high-boiling petroleum hydrocarbon. On heating they are converted into anhydrides; at higher temperatures neutral, unsaponifiable asphalts are produced. Humic acid is the corresponding link between coal and vegetation. Its constitution is doubtful, although it certainly contains a furfuran group. It may be regarded as a polymerised or condensation form of mucin. The processes by which petroleum and cellulose are respectively converted into asphalt and coal are analogous.

—W. J. W.

Coke; Principal innovations in the recovery of by-products in the manufacture of metallurgical — C. Berthelot. Bull. Soc. Ind. Nord, 1919, 46, 87–192.

THE author reviews the various systems of by-product recovery, and indicates those which seem to him to be the most practicable and the various modifications which he has introduced. Descriptions are given of hot condensation systems (Solvay, Bawag, Carrès, and Otto), cold systems (Klönne, St.-Claire Deville), condensation in horizontal tubes, and condensation in cylindrical condensers. Of all these systems, that of St.-Claire Deville alone satisfies the conditions necessary to obtain a suitable absorption of the naphthalene and ammonium salts, whilst giving the best possible yield of ammonia and benzol, because the gas and the condensation products are cooled simultaneously. In hot condensation systems the gas is not completely stripped of naphthalene, and 3–5% of ammonium salts is lost in the tar, from which it is difficult to recover them. The tar is washed with water in a column; if this is not done the ammonium chloride, which constitutes 60–80% of the salts, dissociates, and the hydrochloric acid thus produced corrodes the tar distilling plant. Xylene, cumene, and phenol are lost in the liquors, which condense when the gas cools after its passage through the saturator. The author compares the value of turbo-, Roots, and Beale exhausters, stating that turbo-exhausters are only useful for purified gas, whilst Roots exhausters are easily worn out by the impurities in the gas. Beale exhausters are recommended as being very robust, and capable of being employed for even 150 cm. of water pressure. Two regulators are recommended for controlling the suction of the gas, *viz.*, that made by the Compagnie pour la Fabrication des Compteurs et Matériel d'Usines à Gaz, and the Kress regulator. In the recovery of benzol the Zschokke system is largely used in France, but the author

prefers splash washers, which give a yield of 75% (compared with 70% with the Zschokke system). In the recovery of ammonia the losses in the plant per metric ton are given as:—In evaporation, 40 grms.; in washing, 9 grms.; in distillation, 12 grms.—total, 61 grms., equivalent to 240 grms. of ammonium sulphate. In the manufacture of the sulphate a special comparison of the Feld and Burkheiser processes is made as regards the simultaneous recovery of ammonia and sulphur, and an examination of the possibility of transforming cyanogen into ammonia by oxidation. The Feld system consumes less water but has the higher steam consumption. The recovery of fixed ammonium salts is complete in the Burkheiser system, but loss occurs in the Feld system. It is difficult to obtain a pure sulphate in either plant, and the cost of installation does not compare favourably with the older systems. Attention is drawn to results obtained at the experimental plant of the Association of German Gas Manufacturers at Karlsruhe. Five German coals were tested, and the mean result showed that 8.02% of the ammonia was lost in the tar. To remedy this a process has been devised (see Fr. Pat. 439,509; this J., 1912, 711) according to which the ammonium chloride is condensed in solid form before the gas is scrubbed. A comparison of the German and French (Mallet) systems for the recovery of benzol from the wash oils shows that the French system is more economical than the German one in the ratio of 1.0 to 1.2 in heat expended.—A. G.

[Gas] retort linings. P. M. Grempe. J. Gasbeleucht., 1919, 62, 734–735.

METHODS of lining gas retorts etc. with a coating of carborundum are described. Carborundum powder is used, and is made into a paste with water-glass or fireclay and water. The proportions used are 75 parts of carborundum and 25 parts of water-glass or 85 parts of carborundum and 15 parts of clay by weight. The whole must be well mixed and kept agitated during application. A deposit of $\frac{1}{2}$ mm. thickness is sufficient for most purposes, corresponding to about 1200 grms. of carborundum per sq. m. of surface. The deposit is allowed to stand for 24 hours and then slowly heated. These carborundum linings have worked successfully in many types of retorts and furnaces with coal, coke, or gas firing. The mixture may also be used for repairing flaws in the retorts.—W. P.

Lignite; Carbonisation of — R. Geipert. J. Gasbeleucht., 1919, 62, 742–744.

A DESCRIPTION is given of the carbonisation of lignite at the Mariendorf gasworks. The lignite was delivered in the form of rough lignite, small lignite, and briquettes, the respective calorific values being 2700 cal., 3500 cal., and 4900 cal. A large reduction in volume was observed on carbonisation, the height of coke in a fully charged vertical retort 5 m. high falling to 305 cm. in the case of the small lignite, and to 290 cm. with the briquettes. The resulting coke was very fine. In the former case the coke burnt rapidly on being discharged from the retort. Somewhat better cokes were obtained by carbonising mixtures of the briquettes with pit coal. A comparison of the yields and costs of the carbonisation of pit coal and lignite shows the latter to be uneconomical.—W. P.

Water-gas production in horizontal retorts and its economy. E. Goffin. J. Gasbeleucht., 1919, 62, 729–731. (See this J., 1919, 523 A.)

FURTHER particulars are given of the steaming of horizontal gas retorts at the Heddernheim gasworks, and the advantages of this system over that of the separate production of water-gas are discussed. The coke used for the production of 100 cub. m. of water-gas was 54.06 kilos. A greater

water supply was required in the coolers to cool the mixed gas on account of the undecomposed steam carried over. The heat units supplied in the gas from 3 settings in 1 hour was 2,463,900 cals. with steaming and 2,244,600 cals. without steaming. The necessity for clearing the ascension pipe, and cleaning the receivers and retorts was lessened, and the life of the retorts thereby improved. The costs of production are also examined.—W. P.

Water-gas sampling device. R. A. Carter. Amer. Gas Assoc. Gas J., 1920, 149, 196—197.

THE apparatus described is designed to determine automatically the quantity of gas made during a run, and to collect a representative sample of the gas, not at a uniform rate, but at a rate dependent upon the rate of production. A purified stream of gas is passed at a uniform pressure through an adjustable orifice, the opening of which is controlled by a diaphragm which, in its turn, is actuated by the drop in pressure across a fixed resistance. This resistance is the wash-box, and the drop in pressure across it is proportional to the rate of flow or production of the gas. The sampling device consists primarily of a pressure-regulating governor, a differential pressure governor (a diaphragm directly connected to an adjustable orifice), a condenser, a purifier, and a holder to collect the sample. The holder is calibrated to show the volume of gas made.—W. P.

Flame; Propagation of — in mixtures of methane and air. Part I. Horizontal propagation. W. Mason and R. V. Wheeler. Chem. Soc. Trans., 1920, 117, 36—47.

A DESCRIPTION is given of the phases, other than the uniform movement, during the horizontal propagation of flame in mixtures of methane and air. The duration of the uniform movement of flame, set up initially when a mixture is ignited at the open end of a horizontal glass tube closed at the other, is controlled by the factors which influence the establishment of resonance in the column of gases in the tube: speed of flame, and length, diameter, and uniformity of bore of the tube. With mixtures containing between 10 and 10.5% of methane, and with a brass tube 210 cm. long and 5 cm. in diameter, the speed of flame during the uniform movement was 90 cm. per second over a distance of 80 cm. Faint undulations in the flame front appeared after the flame had travelled 32 cm., but these did not affect the mean speed of the flame. The amplitude of the vibrations increased gradually at first and then more rapidly, the mean speed of the flame falling to 64 cm. per second. Eventually the "vibratory movement," which owes its origin to an undulation of abnormal amplitude, was established. During the forward motion of the flame during the vibratory movement a speed of 2430 cm. per sec. was attained, but the maximum mean speed of flame over the section of the tube photographed was 278 cm. per sec. When ignition takes place at the closed end of a horizontal tube, open at the other, no uniform movement takes place, but the speed of the flame increases rapidly as it travels towards the open end. The speed of flame in some of the mixtures reached 29 m. per sec. over the last half metre of the tube. In longer and wider tubes the flame was extinguished during violent vibrations after the flame had travelled 10 m. The extinction was caused by the contraction of the gases behind the flame dragging the flame back and mixing unburnt gas with the products of combustion. With the tube opened at both ends no such extinction was noted, the flame accelerating in speed as it passed along the tube. This is the condition which would lead to the most disastrous results in industry, for, although the initial speed of the flame is not then so great as when ignition is at a closed end, continued propagation is assured,

and there may be developed momentarily during the vibratory movement velocities and pressures as great as any produced throughout the life of a flame started at a closed end.—W. P.

Flame; Propagation of — in complex gaseous mixtures. Part IV. The uniform movement of flame in mixtures of methane, oxygen, and nitrogen. "Maximum-speed mixtures" of methane and hydrogen in air. W. Payman. Chem. Soc. Trans., 1920, 117, 48—58. (See also this J., 1920, 94 A, 95 A).

THE mixture of hydrogen and air for complete combustion, that is, the mixture having the greatest heat of combustion, contains 29.6% of hydrogen, but the mixture in which the speed of the uniform movement of flame is the greatest contains 38%. A similar "displacement" of the maximum-speed mixtures is observed with all inflammable gases when mixed with air, including gases, such as carbon monoxide, the thermal conductivities of which are less than that of air. The speed of propagation of flame during the uniform movement depends mainly upon the rate of conduction of heat from layer to layer of the mixture, which in turn varies according to the difference in temperature of the burning and the unburnt gases and to their thermal conductivities, and upon the rate of reaction between the combining gases, which for a given combustible gas will vary with the composition of the mixtures, according to the usual laws of mass action and with the temperature of the reaction. The rate of reaction appears to be the determining factor in the position of the maximum-speed mixture, the displacement being shown to be due to the influence of mass action upon the rate, this not having its maximum effect in the mixture with the greatest calorific value. The displacement is in the opposite direction, that is, with excess oxygen, when oxygen burns in an "atmosphere" or constant composition mixture of nitrogen with a combustible gas. Further, the maximum-speed is that of the mixture containing the gases in combining proportions when hydrogen or methane burns in pure oxygen, since in this mixture both the effect of temperature and of mass action have their greatest value. The speed of the uniform movement of flame has been determined in mixtures of methane with a number of different atmospheres of nitrogen and oxygen containing more oxygen than air and with pure oxygen. The maximum speeds of the uniform movement of flame were 55 m. per sec. for the mixture $\text{CH}_4 + 2\text{O}_2$ and 66 m. per sec. for the mixture $2\text{H}_2 + \text{O}_2$. The uniform movement gives place to the detonation-wave after quite a short distance of travel of the flame in the fastest mixtures, but with the detonation-wave the maximum-speeds are obtained with mixtures containing excess of methane or hydrogen.—W. P.

Oil shales; Retort plant for the distillation of —. L. Simpson. Chem. and Met. Eng., 1920, 22, 71—72. (See this J., 1920, 147 A.)

CERTAIN qualities of shale yield by-products, the recovery of which from the spent shale is profitable, varying either according to their quantity or local conditions, even after the maximum oil yield has been secured. The temperature distribution does not render the Scotch dual retort satisfactory in this respect. It is suggested that shale should be distilled in a "single" retort giving a satisfactory yield of oil, and that the spent material should then be heated directly in a horizontal rotary kiln. The fixed carbon in certain shales is sufficient to produce, on combustion, the lower limit of temperature (1150° C.) required for the maximum recovery of ammonia. Fine grinding of the spent shale would assist in the production of ammonia,

and the recovery of potash becomes feasible if the final temperature in the kiln is increased.—C. A. K.

Asphaltic base acid sludge obtained in refining petroleum and shale oils; Utilisation of —. C. Baskerville. J. Ind. Eng. Chem., 1920, 12, 30—31.

THE "acid sludge," obtained in refining petroleum and shale oils with sulphuric acid, is usually heated, by means of live steam, with strong sulphuric acid to separate oil capable of being refined and to carbonise the rest of the material. The mass, which after washing still contains 3—15% of free sulphuric acid, is then burned with coal. If the treatment with sulphuric acid is stopped prior to the carbonisation of the asphaltic material, the mass separates into three layers, viz., oil, asphaltic sludge, and excess of sulphuric acid. The sludge, containing 15—25% sulphuric acid, is washed once with five times its quantity of water, treated with a slight excess of freshly slaked lime, and kneaded in a mill. If sufficient heat has not been generated to render the mass fluid the mixture is heated to about 220° C. and then run into suitable containers. Finely divided limestone may be substituted for the lime. The asphaltic material is incorporated with 10—40% of calcium sulphate. It adheres well to masonry, wood, metal, etc., forming a waterproof coating, and can be mixed with rosin etc. If desired, the asphaltic substances may be extracted by the usual solvents.—C. A. M.

Coal pyrites. Yancey. See VII.

Gas producers. Atkinson. See VIII.

PATENTS.

Powdered fuel; Combustion of —. J. S. Atkinson, and The Powdered Fuel Plant Co., Ltd., London. Eng. Pat. 137,450, 17.4.19. (Appl. 9893/19.)

THE powdered fuel is fed into the combustion chamber in intimate mixture with air by means of a fan, the admission of air being governed by suitable means, such as a hit-and-miss device, in accordance with the load on the plant and the amount of fuel being supplied. A secondary supply of air direct to the combustion chamber may also be used and governed in the same manner.—W. P.

Inert gases; Apparatus for preparation of —. E. E. Molas, Paris. U.S. Pat. 1,327,769, 13.1.20. Appl., 2.7.18.

THE burnt gases from an internal combustion engine are withdrawn through a pipe with an adjustable intake valve. A check valve in the same pipe is actuated by the force of the explosion. The storage tank contains a liquid having a basic character.—W. P.

Petroleum and other hydrocarbons; Fractional distillation of crude —. A. F. G. C. P. J. von Groeling, Assignor to Atlas Process Co., Inc., New York. U.S. Pat. 1,327,184, 6.1.20. Appl., 22.4.16.

EACH of a number of stills is provided with a separate condenser and receiver, and each receiver is attached to an apparatus for creating a high vacuum, which is automatically maintained at a constant degree, in the stills, condensers, and receivers. The means for heating the stills can be controlled so as to vary the temperature of distillation in the different stills.—L. A. C.

Evaporator [for distilling mineral oils]. W. Meischke-Smith, San Francisco, Cal., Assignor to Shell Co. of California. U.S. Pat. 1,327,247, 6.1.20. Appl., 26.3.18.

OIL is delivered as a thin film on to the inner surface of the side walls of a still, shaped in the form

of an inverted frustum of a cone, by means of a conduit enclosing the upper edge of the side walls and having an open circular slit on the inside of the wall. Means are provided for supplying oil to the conduit, for removing vapour from the still, and for drawing off residual oil from the bottom of the still.—L. A. C.

Liquids, fluids, and oils; Process for the conversion of — [to lower boiling oils]. J. H. Adams, Brooklyn, N.Y., Assignor to The Texas Co., Houston, Tex. U.S. Pat. 1,327,263, 6.1.20. Appl., 30.3.11. Renewed 11.4.19.

OILS to be converted into lower boiling products are heated, in a liquid form, in a chamber to a cracking temperature, and the vapour pressure generated in the chamber is maintained on the oils. The vapours are led to a condenser through connections, all of which are in open communication to ensure a uniform pressure from the chamber through the condenser. The lower boiling oils are withdrawn from the condenser.—L. A. C.

Bituminous matter; Process of recovering — from shale. H. D. Ryan, Boulder, Colo., Assignor to National Oil Machinery Corporation, New York. U.S. Pat. 1,327,572, 6.1.20. Appl., 28.3.18.

SOLIDS (shale) containing bitumen are digested with a heavy oil at a temperature high enough to liquefy heavy hydrocarbons present in the shale, but insufficiently high to distil heavy fractions of oil.—L. A. C.

Lubricating oils; Production of — from asphaltic materials. Deutsche Erdöl-Akt.-Ges., Berlin, F. Seidenschmür, Charlottenburg, and C. Köttnitz, Berlin-Lichterfelde. Ger. Pat. 305,105, 4.3.16.

ASPHALTIC materials are distilled with steam in rarefied air or in a vacuum. The oils thus obtained are highly viscous and, after treatment with steam to remove the lighter fractions, have a high flash point. About 60% of high quality cylinder oil may be produced. The residual coke is free from oil and very little gas is evolved in the process.—W. J. W.

Lubricating oils; Process for preparation of highly viscous and combustible — from bituminous materials, especially lignite tar. Deutsche Erdöl-Akt.-Ges., Berlin, F. Seidenschmür, Charlottenburg, and C. Koettnitz, Berlin-Lichterfelde. Ger. Pat. 307,166, 7.11.16.

LIGNITE is gasified in producers, the tar recovered being distilled with superheated steam, with or without vacuum (see preceding abstract). Tar obtained from lignite briquettes yielded 20% of machine oil (viscosity: 6.2 at 50° C.; flash point: 188° C.), and 10% of spindle-oil (viscosity: 4 at 20° C.; flash point: 150° C.); the yield of paraffin was not diminished.—W. J. W.

Fire grates for combustion of small or powdered fuel. A. E. Davis, Birmingham. Eng. Pat. 137,375. (Appls. 1040, 15.1, and 1931, 27.1.19.)

Gas retorts; Apparatus for maintaining predetermined pressure conditions in —. A. A. Macintosh, St. Kilda, and W. H. Hunt, Melbourne, Victoria. U.S. Pat. 1,329,129, 27.1.20. Appl., 2.4.17.

SEE Eng. Pat. 105,563 of 1917; this J. 1918, 292 A.

Gas; Manufacture of —. M. C. Whitaker and W. F. Rittman, New York, Assignors to Synthetic Hydro-Carbon Co., Pittsburgh, Pa. U.S. Pat. 1,327,001, 6.1.20. Appl., 3.4.14.

SEE Eng. Pat. 17,822 of 1915; this J., 1917, 957 A.

Gas-producer for propelling vehicles. D. J. Smith, London. U.S. Pat. 1,327,495, 6.1.20. Appl., 29.11.18.

SEE Eng. Pat. 120,599 of 1917; this J., 1919, 5 A.

Gas-generator. T. R. Wollaston, Manchester. U.S. Pat. 1,327,893, 13.1.20. Appl., 19.1.18.

SEE U.S. Pat. 113,856 of 1917; this J., 1918, 233 A.

Gas-producers. Eng. Pat. 137,168. See I.

Oil emulsions. U.S. Pat. 1,327,835. See I.

Ammonium sulphate. Ger. Pat. 314,598. See VII.

Thionate solutions. Ger. Pat. 314,628. See VII.

Cooling blast-furnace gases. Eng. Pat. 137,378. See X.

Analysing gases. Eng. Pat. 136,992. See XXIII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Electric lamp; A new —. F. Schröter. Elektro-techn. Zeits., 1919, 186. Chem.-Zeit., 1919, 45. Rep., 303.

A LARGE cathode and smaller anode are arranged within a pear-shaped globe containing a mixture of neon and helium at a pressure of 8–10 mm. A discharge is produced between the electrodes employing 220 volts. Where alternating current is employed, the electrodes are of the same size and are arranged symmetrically. Iron, aluminium, or potassium can be used as cathode. Iron possesses certain advantages. The lamp is designed for an energy consumption of from 1 to 5 watts at 220 volts. The intrinsic brilliance of the lamp is about 0.1 Hefner candle per sq. cm. Its life is determined by the sputtering of the cathode. A number of directions in which the lamp may be employed is indicated.—J. S. G. T.

PATENTS.

Heating of vessels; Process for indirect — [using esters of phthalic acid as heat-transmitting medium]. Akt.-Ges. f. Anilin - Fabrikation, Berlin-Treptow. Ger. Pat. 302,581, 11.3.17.

NEUTRAL esters of phthalic acid, which are mobile liquids and can therefore be easily poured, may be used as a heat-transmitting medium. They are stable and non-inflammable, with a high boiling point (280°–290°C.) and a flash-point above 160°C. —W. J. W.

Electrode; Arc-lamp —. W. R. Mott, Lakewood, Ohio, Assignor to National Carbon Co., Inc. U.S. Pat. 1,326,605, 30.12.19. Appl., 23.1.19.

AN arc-lamp electrode contains a combination of potassium silicate and boric oxide, in a form less hygroscopic than either of the materials alone. —B. N.

Electric arc-lamps; Diminution in the energy expenditure per candle power of —. O. Lummer, Breslau. Ger. Pat. 303,262, 26.5.14.

THE carbons are impregnated with suitable substances, such as alkaline-earth fluorides, and are employed to afford a flaming arc which is operated intermittently. Under such conditions the area of the positive crater is reduced and the efficiency of the arc is increased.—J. S. G. T.

Direct current arcs; Arrangement of —. O. Lummer, Breslau, G. R. Mylo, Charlottenburg, and Rütgerswerke A.-G., Berlin. Ger. Pat. 304,221, 28.7.15.

A NUMBER of arcs are struck between a number of electrodes of like polarity surrounding a central electrode of opposite polarity. The central electrode is constituted of a number of parallel sections capable of being separately adjusted. The

arrangement permits of the attainment of a higher temperature than usual. An enhanced illuminating effect is thereby obtained from vapours introduced into the arcs. It is also possible to impregnate the negative electrode with salts which hitherto could either not be employed at all or at most only in very small quantities.—J. S. G. T.

Arc lamp having more than one arc. O. Lummer, Breslau, G. R. Mylo, Charlottenburg, and Rütgerswerke A.-G., Abt. Planawerke, Berlin. Ger. Pat. 303,355, 19.3.16.

THE inner electrode of a multiple arc (see Ger. Pat. 304,223 above) is composed of sections which may be impregnated with different salts. Such sections may be in intimate contact with one another or may be separated by a good thermal conductor. For example, the electrode may be constituted of a solid portion together with a second portion perforated by channels or wicks, these being impregnated with salts, which are entirely absent from or present only to a small extent in the solid portion of the electrode. The two halves may if desired be provided with channels having similar or different characteristics. The device enables the temperature of the negative electrode to equal that existing in the positive crater.—J. S. G. T.

Arc electrodes for the production of short wave-length radiation. Siemens-Schuckertwerke G. m. b. H., Siemensstadt. Ger. Pat. 315,217, 8.3.18.

CERTAIN metals or compounds thereof, the radiations of which show maxima corresponding approximately in wave length with the minima in the radiation of the iron arc, are incorporated along with iron or compounds thereof in the electrodes with a view to enriching the spectrum of the arc in the region of short wave lengths included between 400 μ and 250 μ . Titaniferous iron, zinc, and magnesium or their compounds may be used either singly or in conjunction for this purpose. Cryolite, zinc borate, and potassium magnesium fluoride may be incorporated along with titaniferous iron. The arc is struck in an atmosphere containing very little oxygen.—J. S. G. T.

Electrodes for high vacuum discharge-tubes. C. H. F. Müller, Hamburg. Ger. Pat. 305,521, 1.11.17.

THE electrodes are composed either wholly or in part of chemically pure iron, more particularly of electrolytic iron.—J. S. G. T.

Röntgen-ray tube. Siemens und Halske A.-G., Siemensstadt. Ger. Pat. 311,941, 6.1.18.

SUITABLE elements or their oxides are superposed upon the anticathode in a number of thin layers penetrable by the electrons, and preferably in the form of intersecting lines in close juxtaposition forming a kind of grating. Such coatings serve to increase the hardness of the X-rays. As an example, a platinum anticathode may be coated with one or more of the elements, polonium, thorium, and uranium.—J. S. G. T.

III.—TAR AND TAR PRODUCTS.

Coal tar; Resinous constituents of —. J. Marcussou. Z. angew. Chem., 1919, 32, 345–346.

FINELY-POWDERED coal yielded on extraction with pyridine a product which when sulphonated was completely insoluble in water, whereas tar resins form soluble sulpho acids. Hence these resins are not present as such in the original coal. Experiments indicated that they are produced by an oxidation process. The three tar resins, previously de-

scribed (this J., 1918, 403 A), were artificially prepared by blowing air into coal tar oil of high b. pt. at about 120° C. The product, when treated with acetone, yielded an insoluble deposit; the filtrate from this was freed from solvent, and the residue treated with glacial acetic acid, leaving a black, pitch-like mass which, when digested with acetone, yielded the solid resins. These resins behaved in the same way as those separated from coal tar. They may also be produced by heating the oil for a long time under a reflux condenser. The thickening of tar lubricating oil in technical operations depends mainly on the production of tar resins. According to Ruszig (this J., 1919, 217 A) phenylxanthenes are constituents of tar lubricating oils, but artificially-prepared phenylxanthene did not yield a resin when blown at 120° C., and is therefore not the original substance from which the resins are derived. It is more probable that the resins are derived from unsaturated hydrocarbons, and their formation is analogous to that of asphaltic substances from mineral oils of high b. pt. Their physical and chemical characters are also similar. The tar resins appear to differ essentially from asphaltenes only in aromatic character, and may therefore be regarded as aromatic asphalts.

—C. A. M.

Cresol (cresylic acid); Purified —. H. C. Hamilton. J. Ind. Eng. Chem., 1920, 12, 50—52.

CRUDE cresol is dissolved in a solution of sodium hydroxide of equivalent alkalinity, and sufficient water added to dilute the sodium cresylate to about a 25% solution. The liquid is then boiled or subjected to the action of live steam until all the volatile impurities (pyridine etc.) have been removed, the evaporated water being replaced in order to prevent the solution becoming so concentrated that the cresylate is decomposed and free cresol volatilised. After cooling, any oil upon the surface of the liquid is skimmed off, whilst any naphthalene remaining unvolatilised is removed by filtration or centrifuging. Finally the cresylate is decomposed by the addition of an amount of sulphuric acid equivalent to the alkali originally used, and the separated cresol is redistilled.—C. A. M.

By-product recovery in coke manufacture. Berthelot. See IIa.

PATENTS.

Oils; Process of extracting — from coal-tar. H. H. Comber and J. W. Stalker, Winnipeg, Canada. U.S. Pat. 1,327,271, 6.1.20. Appl., 17.6.18.

COAL-TAR is agitated in the cold with wood alcohol; the upper layer, containing the oils and alcohol, is drained into a vessel containing water, which dissolves the alcohol. The oils are then drawn off from the bottom of the vessel.—L. A. C.

Coal tar and the like; Process for purification of — and simultaneous recovery of high-molecular resinous bases. E. Wirth, Wiesbaden. Ger. Pat. 303,273, 24.2.15.

TAR obtained by dry distillation of bituminous substances is freed from phenols by treatment with an alkaline solution and then treated with dilute, followed by more concentrated mineral acid. As the salts of the resinous bases (constituents of the so-called "acid tar") are soluble in phenol, its initial removal enables the resinous bases to be recovered. The tar may be freed from its more volatile constituents before being subjected to the treatment.—W. J. W.

Tar oils; Process for purification of heavy — and recovery of bases of high boiling point. E. Wirth, Wiesbaden. Ger. Pat. 304,306, 13.2.16. Addition to Ger. Pat. 303,273.

THE process described in the preceding abstract

may be applied to heavy tar oils to recover the bases from them by treatment of the acid solution with alkalis. The acid concentration is kept sufficiently low to prevent separation of resinous substances. The bases are obtained in the form of a viscous oil distilling mainly between 320° and 390° C. The mixture contains acridine and also non-crystallisable bases of the formula $C_{2n+1}H_{n+3}N$, beginning with $C_{13}H_{19}N$. These bases may be utilised for the manufacture of dyes and pharmaceutical preparations.—W. J. W.

Fractional distillation of hydrocarbons. U.S. Pat. 1,327,184. See IIa.

Lubricating oils. Ger. Pat. 307,166. See IIa.

Phthalic esters as heat-transmitting medium. Ger. Pat. 302,581. See IIa.

IV.—COLOURING MATTERS AND DYES.

Diazotisation; The factors of —. E. Tassilly. Bull. Soc. Chim., 1920, 27, 19—33.

Using the spectrophotometric method previously described (this J., 1914, 247), the authors have studied the diazotisation of a number of amines. The reaction is shown to be bimolecular and, in the case of aniline, its velocity is not modified by doubling the acidity of the solution. The diazotisation of sulphanilic acid is much more rapid when the concentration of the reacting solutions is increased, and it is favoured by an excess of sodium nitrite. The stability of diazo compounds with respect to time and temperature may be studied by this method.—W. G.

Mordant dyestuffs; New bases for the preparation of —. H. Bamberger. Z. angew. Chem., 1920, 33, 8.

THE chlorides of *o*-nitrophenol-*p*-sulphonic acid, 6-nitro-*o*-cresol-*p*-sulphonic acid, and 6-nitrosalicyl-*p*-sulphonic acid, obtained by treatment of their potassium salts with chlorosulphonic acid, react quantitatively with aromatic amines, diamines, aminocarboxylic acids, or aminophenols, and with aminosulphonic acids or aminophenolsulphonic acids of the benzene or naphthalene series. By reducing the resulting sulphamino-compounds with sodium sulphide, valuable bases are produced for mono- and di-azo mordant dyestuffs, which give colours ranging from yellow to bluish-violet.—J. K.

PATENTS.

Coal-tar dyes; Production of —. E. D. Kendall, Elizabeth, N.J., Assignor to Haggin Estate, Inc. U.S. Pat. 1,326,665, 30.12.19. Appl., 5.4.18.

DYES are produced from nitrobenzene or its homologues by electrolysis of a mixture of one or more nitro-compounds, acetone, water, and sulphuric acid.—L. L. L.

Dyestuffs and process of making same. A. S. Wheeler, Chapel Hill, N.C. U.S. Pat. 1,327,260, 6.1.20. Appl., 9.12.18.

A SOLUTION of a hydroxynaphthoquinone having less than three hydroxyl groups is treated with a halogen in excess; the halogenated product is then separated and converted into a salt.—L. A. C.

Azo dyes and process of making same. R. Taggesell, Buffalo, N.Y., Assignor to National Aniline and Chemical Co., Inc., New York. U.S. Pat. 1,327,688, 13.1.20. Appl., 6.9.19.

ONE mol. of tetrazotised *m*-toluylenediaminesulphonic acid ($CH_3NH_2 \cdot SO_3H = 1:2:6:4$) is combined with 1 mol. of a *m*-toluylenediaminesulphonic acid, and the product is then combined with a suitable unsulphonated compound.—L. A. C.

Dye. U.S. Pat. 1,326,367. See VI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Degreasing (scouring) wool and textiles with volatile solvents. H. Hey. J. Soc. Dyers and Col., 1920, 36, 11—16. (See also this J., 1919, 406A.)

THE author describes the degreasing of wool by solvents, giving a list of solvents and the properties of some of them. With "burry" greasy wool, carbonising in the grease is recommended, the grease preserving the wool. The warm material from the drying oven, after carbonising, is degreased by solvents and then the burrs are crushed and shaken out. The acid is then removed by washing with water, followed by soda, and a final washing with soap leaves the wool in good condition for the further operations. The consumption of acid is greater than by the ordinary method of carbonising after scouring, but is compensated for by the better results obtained. Owing to the absence of previous alkali treatment, the wool is finished in a better condition than by the ordinary method. *Sterilising wool.* Very dilute hydrochloric or sulphuric acid solutions are capable of rendering anthrax spores sterile, especially at high temperature. It is proposed to steep the raw greasy wool in a dilute acid bath, to which 1% of formaldehyde is added; after thorough impregnation with the liquid the wool is drained, centrifuged, and dried at a high temperature to sterilise.—L. L. L.

Alder wood; Chemical composition of —. C. G. Schwalbe and E. Becker. Z. angew. Chem., 1920, 33, 14—16.

A DETAILED discussion of the results of analyses of samples from trees of various ages. With increase in age, the proportions of lignin and of pentosan-free cellulose increase, whilst that of acetic acid obtainable by hydrolysis diminishes. Contrary to the view of Pringsheim and Magnus (this J., 1919, 714A), therefore, there is no relation between the proportions of lignin and acetic acid. The heartwood contains more lignin, but less cellulose than the sap wood.—J. K.

PATENTS.

Wool-scouring liquors; Process for treating —. G. Kolsky, New York, Assignor to The Diamond Match Co., Chicago, Ill. U.S. Pat. 1,325,873, 23.12.19. Appl., 24.12.15.

THE grease contained in the scouring liquors is coagulated by means of a soluble derivative of sulphurous acid, the sulphur dioxide of which is in unstable combination, so that on heating with water the compound is decomposed into materials which will readily separate from the liquor, and may be again used for the regeneration of the sulphurous acid derivative. The coagulated grease is separated and refined.—L. L. L.

Drying and carbonising machine [for fibrous materials]. T. Allsop and W. W. Sibson, Assignors to The Philadelphia Drying Machinery Co., Philadelphia, Pa. U.S. Pat. 1,326,161, 30.12.19. Appl., 19.2.19.

A RECTANGULAR casing is divided by horizontal partitions into compartments which are subjected to different heat conditions as required, and each compartment is provided with a horizontal endless band conveyor. The material is fed on to the uppermost conveyor outside the casing, and after passing through the compartment is transferred to the conveyor in the next compartment by means outside the casing, the ends of the conveyors being extended outside the casing for this purpose.

—W. F. F.

Paper or cellulose fabrics; Process for improving —. Exportingenieur für Papier- und Zellstofftechnik G.m.b.H., Berlin-Wilmersdorf. Ger. Pat. 303,861, 30.9.15.

THE fabric, preferably after treatment with a swelling agent, such as caustic soda, ammoniacal copper solution, lactic acid, etc., is subjected to fulling, either dry or damp, in order to compress it. Albumin may be used both as a swelling and a finishing agent, added either after the fulling or after the drying; it is preferable to add to it small quantities of acids or alkalis to increase the swelling effect and the ease of penetration of the albumin into the fibre, and of a hygroscopic substance to reduce the hardness of the fabric. If the fabric is to be exposed to wet conditions a water-repellent substance, such as wool-fat, should be added.—B. V. S.

[Reeds;] Process for obtaining technically valuable products [from —]. Verwertung inländischer Produkte, G.m.b.H., Charlottenburg. Ger. Pat. 304,285, 21.12.16.

THE root stem of the plant (*Arundo phragmites*) is separated by mechanical means, assisted, for instance, by treatment with weak caustic soda solution at 100° C., into a fibrous part suitable for textile purposes, such as jute or paper-yarn substitute, and a part containing starch and sugar suitable for production of alcohol or cattle fodder by fermentation processes.—B. V. S.

Rushes; Preparation of valuable fibres from —. Deutsche Typha-Verwertungsges.m.b.H., Charlottenburg. Ger. Pat. 308,564, 20.1.18.

THE rushes are crushed, treated with weak alkali, and then with dilute bisulphite solution, preferably at boiling temperature. Long, white, pliant fibres, suitable for spinning and of considerable strength are obtained.—B. V. S.

[Pea-pods;] Fibrous material [from —]. R. Pöhler, Frankenthal. Ger. Pat. 307,626, 19.2.18.

By a fermentation process or by treatment with hot water or steam the outer layer of pea-pods is dissolved away, leaving the inner fibrous layer in a form suitable for spinning etc. The solution of the outer layer contains sugar and is suitable for use in the preparation of marmalade etc.—B. V. S.

Typha fibres; Process for the after treatment of — separated by a chemical process. Deutsche Typha-Verwertungsges.m.b.H., Charlottenburg. Ger. Pat. 308,563, 28.11.17.

THE fibres are washed and then boiled with sodium bisulphite solution either in open vessels or in closed vessels under pressure.—B. V. S.

Textile materials; Process for waterproofing and softening —. L. Meyer, Charlottenburg. Ger. Pat. 314,968, 13.12.18.

THE material is treated first with a solution of a metallic salt, then dried and treated with an alkali and finally with soap or similar substance. For instance, artificial silk may be treated with aluminium formate, dried at 40°—45° C., then treated with ammonia, and lastly with a soap solution at about 50° C.—B. V. S.

Waste from paper industries; Process for rendering — absorbent. Reis und Co., Friedrichsfeld. Ger. Pat. 307,611, 20.2.18. Addition to Ger. Pat. 303,302 (this J., 1920, 15A).

AFTER the chemical treatment and before drying the waste is left for a long time exposed to the action of the solution of the residue of softening reagent (alkali etc.) remaining in the material.

This gives a more permanent absorbent quality than if the softener is thoroughly washed out before drying.—B. V. S.

Fabric for aircraft and process of making same. N. A. T. N. Feary, Peterborough. U.S. Pat. 1,327,707, 13.1.30. Appl., 21.4.19.

See Eng. Pat. 123,690 of 1918; this J., 1919, 678 A.

Paper; Process for making lines and water-marks on — and apparatus therefor. O. Suursalmi, Assignor to Kangas Pappersbruks Aktiebolag, Jyväskylä, Finland. U.S. Pat. 1,329,100, 27.1.20. Appl., 13.8.19.

See Eng. Pat. 123,082 of 1919; this J., 1919, 896A.

Saccharification of wood etc. Ger. Pats. 305,180, 309,150, and 310,149—50. See XVIII.

Cellulose products. Ger. Pat. 305,019. See XXII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Ink stains; Removal of — [from fabrics]. Gerhardt. Z. angew. Chem., 1920, 33, 32.

AN ink stain on a cotton cloth resisted the action of oxalic acid, oxalic acid and sodium chloride, and oxalic acid and zinc dust, but was removed completely by treatment with dilute potassium permanganate solution followed by sodium thiosulphate and citric acid, and subsequent washing with water.—W. P. S.

PATENTS.

Silk; Increasing the strength and elasticity and preventing or decreasing the subsequent weakening of fibres of loaded —. J. Korselt, Zittau, Saxony. Eng. Pat. 136,578, 24.8.17. (Appl. 12,188/17.) Addition to 101,684/16 (this J., 1918, 53 A).

THE strength and elasticity are increased or the subsequent weakening of loaded silk fibres is prevented or decreased by treating the silk with berberine or with alkaloid derivatives or decomposition products which can be produced synthetically, such as tropine, or methyl-phenmorpholine, or pimelic acid, or compounds produced by the fermentative or chemical decomposition of protein or nuclein, such as amino-acids of the aliphatic or aromatic group; hydroxyamino or thioamino acids; heteronuclear compounds of the iminazole, pyrrole, and indole groups; polypeptides and peptones; proline, histidine, tryptophane, protamines, histopeptone, etc.; guanine, cytosine, thymine, uracil, guanylic acid, inosinic acid, etc. All these compounds are oxidised by the loading material or by the oxygen of the air more readily than the fibroin of the silk. These compounds modify the handle of the silk by rendering it softer and prevent the action of the loading agents on the fibre as well as preventing or diminishing rotting with age resulting from the oxidation of the fibroin. After treatment with these protective agents the loaded silk showed an increase of 50—60% in strength and 25—40% in elasticity.—L. L. L.

Dyeing, washing, scouring, sizing, bleaching, and mercerising yarn in hank form; Machines for —. J. S. and S. S. Lord, Manchester. Eng. Pat. 136,666, 31.12.18. (Appl. 21,864/18.) Addition to 121,075/18 (this J., 1919, 71 A).

THE machine described in the chief patent is modified to enable it to be used for dyeing or treating hanks with different dyes or reagents. Each of the rollers is provided with a trough which when the arm carrying the respective rollers approaches the

vertical position is automatically charged with dye or reagent, and after the hanks are treated automatically emptied. A device is attached to the hank rollers for feeding or stripping the machine, and a suitable form of reversible gear is provided.—L. L. L.

[Indigo] dyeing machines. F. and F. Davis, Halifax. Eng. Pat. 136,991, 14.5.19. (Appl. 9108/19.)

A MACHINE for the dyeing of indigo on woollen or worsted piece goods comprises a vat containing an upper and lower tier of rollers submerged in the dye liquor. The upper tier consists of rollers of two diameters, the larger rollers being arranged alternately to two or more of the other rollers. The larger rollers are driven by means of chains and chain wheels, and resting on the driven rollers are jockey or nip rollers. The goods are taken through the liquor under a uniform tension, causing little or no disturbance of the dye bath; consequently there is no risk of curled edges, creases, or premature oxidation with resulting streaky dyeing.—L. L. L.

Dye and process of dyeing. J. A. Nieuwland, Notre Dame, Ind. U.S. Pat. 1,326,367, 30.12.19. Appl., 3.12.17.

THE dye-bath is prepared by the action of a mercuric salt upon a solution of *p*-phenylenediamine above the normal temperature. Fabrics may be dyed by subjecting them to the successive action of solutions containing respectively a mercuric salt and *p*-phenylenediamine.—L. L. L.

Paper yarn and cloth; Process for dyeing —. Chem. Fabr. Coswig-Anhalt, G.m.b.H., Coswig. Ger. Pat. 315,311, 15.3.18.

OXIDISING agents, such as perborate, percarbonate, etc., are added to the dye-bath. Brighter colours are obtained and the substance is softer to the touch, is more easily impregnated by waterproofing solutions etc., and is stronger than when dyed in the absence of the oxidising agent.—B. V. S.

Printing fabrics or the like; Machines for —. A. Samuel, Lyons, France. Eng. Pat. 126,946, 20.3.19. (Appl. 6926/19.) Int. Conv., 16.5.18.

THE machine comprises two carriages arranged to move at right angles to each other, one of these carriages being displaceable along the length of the printing table on which the fabric is placed, whilst the other carriage, carrying the printing device, is displaceable transversely for the purpose of printing the design of an engraved roller across the entire width of the fabric. When a strip has been printed, the first carriage is displaced longitudinally for a distance equal to the width of the printed strip and the second carriage, brought back to the rear, prints a new strip fitting adjacent to and in exact alinement with the first. The machine is fitted with a device for turning the printing rollers and also for raising these rollers during their return to the rear in order to avoid contact with the fabric.—L. L. L.

Colour fastness in dyed fabrics; Apparatus for testing —. J. W. Driver and F. W. Sunderland, Bradford. Eng. Pat. 136,971, 8.3.19. (Appl. 5788/19.)

THE dyed material is exposed in a frame to the concentrated beam of an arc lamp from which the infra-red or heat rays have been substantially removed by a light filter. The arc light is condensed by a plano-convex lens and projected on to a double convex lens, the frame holding the sample of fabric to be tested being placed slightly beyond the focus of the last lens. If necessary, a colour screen is interposed to act as a filter for infra-red or heat rays.—L. L. L.

Mergerising, dyeing, and other operations; Means for supporting and stretching hose and like knitted fabrics upon wire forms during —. A. E. Hawley, Hinekey, P. F. Crosland, Halifax, and F. G. Dixon, Cheadle Hulme, Eng. Pat. 137,386, 22.1.19.

[*Aniline*] *Black; Production of* — upon vegetable textile fibres, silk fibres, or mixtures of the same. E. A. Fourniaux, Manchester. U.S. Pat. 1,329,117, 27.1.20. Appl., 27.5.19.

SEE Eng. Pat. 126,201 of 1918; this J., 1919, 460A.

Starch. Eng. Pats. 135,591 and 135,588. See XVII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Coal pyrites; Chemical data on —. H. F. Yancey. Chem. and Met. Eng., 1920, 22, 105–109.

A GEOLOGICAL survey indicated that about 1,500,000 short tons of pyrites concentrates could be recovered from coal mining operations in the Eastern and Central coalfields of the United States. The sulphur content of about 120 collected samples, freed from adherent coal, was found to be in most cases sufficiently high for economic treatment in the manufacture of sulphuric acid. The colour of the specimen was no index of the probable purity, as many of the samples containing more than 45% of sulphur were dirty gray and non-crystalline, while bright brass-yellow crystals were often found to be low in sulphur value. The carbon contained in picked or washed pyritic material is not injurious, as material containing up to 10% of bituminous matter has been used for making sulphuric acid without trouble. Pyrites from coal contains only very small quantities of arsenic (0.005–0.056%) and phosphorus (0.003–0.083%), and a considerable proportion of the burnt cinder could be used for the production of low-phosphorus iron.—C. A. K.

Sulphuric acid, nitric acid, and their mixtures; Specific heat of —. P. Pascal and Garnier. Bull Soc. Chim., 1920, 27, 8–18.

Using an electrical method of heating, the authors have determined the specific heat of sulphuric acid at concentrations varying from 10 to 100% of acid, of nitric acid at concentrations varying from 10 to 98%, of mixtures of nitric acid and sulphuric acid, and of mixtures of nitric acid, sulphuric acid, and water in varying proportions. The results are set out in detail.—W. G.

Ammonia; Advantages of the synthesis of — at very high pressures. G. Claude. Comptes rend., 1920, 170, 174–177.

NUMEROUS advantages are claimed for the use of pressures up to 1000 atmos. (see this J., 1919, 885 A; 1920, 61 A) instead of 200 atm. as in the German method, in the synthesis of ammonia on an industrial scale. The contact chamber can be very materially reduced in size for the same production, and it is only necessary to pass the reacting gases over the catalyst three times instead of the considerable number of times necessary at the lower pressure. Again, at the higher pressure much more heat is evolved and may be used to produce power, and auto-reaction can be realised in much less cumbersome apparatus. At the high pressure the percentage of ammonia in the issuing gases may be 25%, and its partial pressure such that simple cooling with water is sufficient to liquefy the greater proportion of the ammonia as it issues from the reaction chamber. The ammonia in this form can be readily liberated as gas and thus easily used in the ammonia soda process. Finally, there is very little loss of pressure in the high-pressure pro-

cess compared with the German method, as it is only necessary to circulate the gas three times over the catalyst and the ammonia can easily be removed from the gases issuing from the reaction chamber.

—W. G.

Ammonia; Catalyst for the oxidation of —. G. A. Perley. Chem. and Met. Eng., 1920, 22, 125–129.

THE DIVERGENCE in the type of platinum catalyst employed in different installations is emphasised. Since the reaction is essentially a contact process a gauze of 0.0025 in diam. wire and of 120–150 mesh is more logical from the standpoint of economy in platinum than the gauze of larger mesh generally used. A three-layer gauze arrangement of mantle form represents the maximum upper limit for the economical use of platinum, and the layers should be separated in order to permit the much-diluted ammonia-air mixture, which passes the first layer, to be heated to a higher temperature than the original, more concentrated ammonia mixture. A suitably preheated mixture of gases is important if a rapid velocity of reaction is to be attained. It is probable that nitric oxide (NO) is not the initial product of the ammonia oxidation reaction. The decomposition reactions are dependent on the gas velocity, and for the efficient use of spaced gauzes a correct adjustment of the space-time-temperature factor is necessary.—C. A. K.

Hydrogen; Formation of free — in the reversed ammonia flame. F. C. G. Müller. Z. angew. Chem., 1920, 33, 24.

THE author finds that the products of combustion of ammonia in oxygen contain hydrogen and nitrogen in the ratio 2:3 by volume. Since the result is unaltered by cooling the flame by cold water (Raschig, Z. phys. Chem. Unterr., 1918, 138), it cannot be due to dissociation of ammonia. On the other hand, Raschig's assumption that the equation $2\text{NH}_3 + \text{O}_2 = \text{N}_2\text{H}_4 + 2\text{H}_2\text{O}$ represents the first stage of the reaction, followed by instantaneous dissociation of N_2H_4 and partial combustion of the hydrogen, does not account for the constancy of the proportion of hydrogen produced.—J. K.

Nitrogen trioxide; Existence of — in the gaseous state. E. Wourzel. Comptes rend., 1920, 170, 109–111. (See also this J., 1920, 151 A.)

Measurements of the contraction occurring when known quantities of nitric oxide and oxygen are mixed, the nitric oxide being in excess, indicate that, under a total pressure of about 0.25 atm., an almost stoichiometric mixture of nitric oxide and nitrogen peroxide contains about 25% of nitrogen trioxide.—W. G.

Nitric oxide; Velocity of oxidation of —. E. Wourzel. Comptes rend., 1920, 170, 229–231.

THE oxidation of nitric oxide is a reaction of the third order and shows no noticeable modification when half the nitric oxide is oxidised. The formation of nitrites or nitrosyl sulphate, observed when the gaseous products formed at the commencement of the reaction are treated with alkali or sulphuric acid, must be due to particular conditions of absorption and not of oxidation. Nitrogen trioxide is an ultimate product of the action and not an intermediate product. The velocity of the oxidation diminishes with rise in temperature.—W. G.

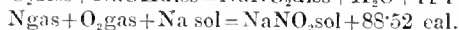
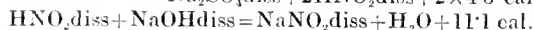
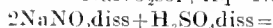
Sodium nitrite; Reversible oxidation of —. C. Matignon and E. Monnet. Comptes rend., 1920, 170, 180–182.

IT is well known that sodium nitrate is decomposed by heat, giving sodium nitrite and oxygen. It is now shown that if sodium nitrite is heated in oxygen at a pressure of 175 atm., the temperature being gradually raised during 9 hours from 395° C. to 530° C., it is almost entirely converted into

sodium nitrate. The heat of formation of the nitrate is given by $2\text{NaNO}_3\text{sol} + \text{O}_2 = 2\text{NaNO}_2\text{sol} + 45$ cals. The reaction is, however, too slow for practical application. Calcium nitrite is similarly converted into calcium nitrate.—W. G.

Sodium nitrite; Some properties of —. C. Matignon and G. Marchal. *Comptes rend.*, 1920, 170, 232–233.

Sodium nitrite has m. pt., 276.9°C . (corr.). The following heat equations have been found for it:—



Aqueous sodium nitrite is not oxidised at 100°C . by prolonged contact with oxygen under a pressure of 50–55 atm. even in the presence of a catalyst.

—W. G.

Sulphates; Volumetric determination of — by oxidation of benzidine sulphate with potassium permanganate. P. L. Hibbard. *Soil Sci.*, 1919, 8, 61–65.

A MODIFICATION of the method of Raiziss and Dubin (*J. Biol. Chem.*, 1914, 18, 297). The solution for the precipitation of benzidine sulphate should be free from iron, heavy metals, nitrates, phosphates, and organic matter, and should only contain between 0.5 and 1.0 mgrms. of SO_4 . The liquid (10–25 c.c.) is treated with one drop of phenolphthalein and then with dilute sodium hydroxide till just alkaline. It is then neutralised with $\text{N}/10$ hydrochloric acid and 0.1 c.c. added in excess for each 5 c.c. of solution. Keeping the liquid quite cold, 5 c.c. of 0.8% benzidine hydrochloride solution is added, and after 15–30 minutes the benzidine sulphate is collected in a Gooch crucible, washed with the minimum of cold water, transferred with the asbestos pad to the precipitation flask, and heated with 1 c.c. of 10% sodium hydroxide solution in a boiling water bath for a few minutes. Then 10 c.c. of dilute sulphuric acid (1 acid:1 water) is added and hot water to bring the volume to 100 c.c. $\text{N}/20$ potassium permanganate is run in until there is 5 c.c. in excess and the solution heated on the water bath for exactly ten minutes. Then 10 c.c. of $\text{N}/20$ oxalic acid is added and the titration completed with $\text{N}/20$ permanganate. The factor for the conversion of c.c. of permanganate solution into mgrms. of SO_4 is 0.12. It is advisable to make a blank determination.—W. G.

Calcium carbide manufacture; Notes on —. J. Baumann. *Chem.-Zeit.*, 1920, 44, 33–35.

Good contact between the electrode and its holder is essential; intermediate layers of copper gauze or a coating of graphite-dextrin paste tend to burn out the holder. The most suitable form of holder is the "copper pin" type. By the use of sprinklers for cooling the electrode holders water is economised and loss of current and risk of explosion in the furnace are avoided. Electrodes should be carefully prepared from good anthracite; they should have a fine-grained structure and should emit a ringing sound. If they are heated to too high a temperature they flake and disintegrate. The size of the raw materials is important. If the pieces are too large a deposit of carbide forms on the electrodes and lowers the potential. Dust in the "carbide smoke" may be removed by a cyclone; efforts to recover the lime formed by combustion of metallic calcium produced by dissociation of the carbide have so far been unsuccessful. By careful avoidance of local overheating through selection of the correct size of electrodes, the amount of "smoke" is reduced to a minimum. The life of the electrodes has an important bearing on econo-

mical running. The use of jackets, or coating the surface, to protect the electrodes from the effect of atmospheric oxygen is not effective. Means by which the raising and lowering of the electrodes are avoided would promise better results. The consumption of electrodes by carbide formation might be diminished by manufacturing carbide of lower strength than 80%. In continuing to use electrodes after they have burned down, the possible destruction of the more expensive electrode-holder must be borne in mind.—W. J. W.

Carbide furnace gases. P. Schl pfer. *Z. Elektrochem.*, 1919, 25, 409–414.

THE author has examined the gases and the dust from the electric furnaces used for the manufacture of calcium carbide. The carbon monoxide content of the gas was a maximum in the immediate vicinity of the electrodes; one such sample showed: CO_2 , 1.00%; O_2 , 1.33%; CO , 84.64%; H_2 , 5.82%; N_2 , 7.21%; whilst a sample taken 50 cm. from the electrode close to the surface of the charge gave: CO_2 , 15%; O_2 , 2.55%; CO , 16.53%; H_2 , 56.13%; N_2 , 9.79%. The combustion of the gases in the case of open furnaces, actually starts in the reacting mass and is completed so rapidly that carbon monoxide cannot be detected in the gases coming from the furnace. There is no noticeable damage done to plant life in the neighbourhood of carbide furnaces. For the manufacture of 1000 kilos. of carbide (containing 85% CaC_2) 1000 kilos. of lime and 600 kilos. of coke are used. There is a loss of 121 kilos. of lime, 60 kilos. of coke ash, and 23 kilos. of coke dust, all of which must be carried away by the gases in the form of dust. This dust is described as the plague of the carbide industry. A number of analyses of the dust, collected in different ways, are quoted. A sample collected in a coke scrubber contained: Sol. in HCl : MgCO_3 , 3.03%; CaCO_3 , 12.54%; CaO , 24.44%; CaSO_4 , 3.09%; Fe_2O_3 , 4.66%; SiO_2 , 3.77%; insoluble in HCl : C , 34.84%; ash, 9.58%; S and O_2 , 2.11%; H_2 , 0.49%. A sample from a settling tank, collected in the wet way, contained a much higher proportion of calcium carbonate and less lime, the latter having been further converted into carbonate by the CO_2 of the gases through the solvent action of the water. It is concluded that the dust from the furnaces originally contains much lime, which gradually becomes converted into carbonate. The amount of dust produced is proportionately much greater from larger furnaces than from smaller ones, and increases with increasing voltage. It also depends on the manner in which the furnace is worked and is lessened by the use of coarser grained lime. The author has calculated the heat balance-sheet of the process, and shows that the heat of the gases, if it could be utilised, is sufficient to burn more than half the lime used in the manufacture.—E. H. R.

Chromic acid; Continuous process for the regeneration of —. R. H. McKee and S. T. Leo. *J. Ind. Eng. Chem.*, 1920, 12, 16–26.

THE organic matter in waste chromium liquors may be destroyed by means of an electric current without any preliminary treatment. The best results are obtained at the ordinary temperature, and hard lead is the most suitable material for the anode. Preliminary filtration of the waste liquor from suspended organic matter (through a sand filter 2 in. deep) is important. In a continuous process of regeneration the fresh waste liquor was fed directly into the bottom of one end of the cathode chamber, and overflowed through an orifice at the other end into the anode chamber, in which the level of the electrolyte was kept somewhat lower. The finished solution was drawn off continuously at the opposite end of the anode chamber. The acid concentration in the cathode chamber was restored by the supply of fresh waste liquor, whilst

in the anode chamber it was reduced and balanced by the overflow liquid from the cathode chamber. The cell was rectangular, and the diaphragm was composed of "electro-filtros." In commercial practice a solution of 70 grms. CrO_3 equivalent per litre is commonly used as oxidising agent, so that the concentration of the anolyte should be maintained above that limit. Better results were obtained with a concentration of 96 grms. than with 80 grms. per litre. For a given current density the capacity of the cell is directly proportional to the anode surface. With a current density of 1.2 amp. per sq. dm. the equivalent of about $\frac{1}{3}$ lb. of chromic anhydride is produced in 24 hours for an anode surface of 8 sq. dm.; whilst with a current density of 2.4 amp. per sq. dm. about $\frac{1}{11}$ lb. is produced in 24 hours with same anode surface. Only about 3 k.w.h. of electric energy is required for each lb. of chromic acid regenerated, and the value of the sulphuric acid simultaneously regenerated is nearly as great as the cost of the electric energy.—C. A. M.

Adsorption and adsorption compounds. V. Adsorption compounds of cuprous oxide. L. Berzeller. *Biochem.-Zeits.*, 1919, 93, 230—237.

In precipitating cupric hydroxide from copper sulphate with sodium hydroxide some of the alkali is adsorbed by the precipitate. The black hydroxides adsorb more of it than the blue ones. Iodate ions are also adsorbed by cupric hydroxide, but in this case the blue hydroxides adsorb more of it than the black. The presence of sugar prevents the adsorption of sodium hydroxide by cupric hydroxide.

—S. S. Z.

Hydrocyanic acid; Identification of traces of —. Chelle. *Bull. Soc. Pharm. Bordeaux*, 1919, No. 2. *Ann. Chim. Analyt.*, 1920, 2, 21—21.

The phthalein test and the isopurpurate test may be used, but they are not specific reactions of hydrocyanic acid. The ammoniacal silver iodide test is specific, but conversion of the hydrocyanic acid into Prussian blue is the most trustworthy test; it may be used to identify the hydrocyanic acid in mercury cyanide or silver cyanide. (See also this J., 1911, 837; 1917, 195; 1919, 899 A.)—W. P. S.

Thiocyanogen; Free —. E. Söderbäck. *Annalen*, 1919, 419, 217—322.

Lead thiocyanate reacts completely with a dry ethereal solution of bromine cooled by ice-water to form lead bromide and a solution of thiocyanogen, CNS. Other organic solvents may be used and the lead thiocyanate replaced by silver, cadmium, mercury, zinc, thallium, or copper thiocyanate. A solution of chlorine in carbon tetrachloride reacts in the same way with silver or mercury thiocyanate, but not with lead thiocyanate. A solution of iodine in an organic solvent reacts with certain thiocyanates similarly, but in no case does the reaction attain completion. The solution of thiocyanogen converts iron powder into ferric thiocyanate and cuprous thiocyanate into cupric thiocyanate, and liberates iodine from solutions of iodides. By cooling an approximately $N/2$ solution of thiocyanogen in carbon bisulphide the substance is obtained in colourless crystals which melt at -3° to -2° C. and decompose at the ordinary temperature. (See also J. Chem. Soc., March, 1920.)—C. S.

Potassium anhydrotellurate; A reaction of —. P. Hulot. *Bull. Soc. Chim.*, 1920, 27, 33.

If potassium anhydrotellurate is suspended in hydrochloric acid (1 acid:2 water) and a few fragments of zinc are added the whole of the tellurium is precipitated as a powder.—W. G.

Welding of solid powders under pressure. T. von Hagen. *Z. Elektrochem.*, 1919, 25, 375—386.

An extensive series of experiments is described on

the behaviour of pure powdered inorganic substances when compressed into tablets, the object being to determine how the hardness, form, and strength of the tablet are influenced by the properties of the original material, such as its hardness, melting point, and crystalline form, by the size of grain of the powder, by the degree of pressure employed, and by the addition of other substances. Generally the substance examined was ground to pass a sieve of 25 meshes per sq. mm., and the tablets were formed under a pressure of 560 kilos. per sq. cm. The substances examined included oxides, sulphides, sulphates, halides, nitrates, and carbonates of many metals. The halogen compounds of the alkali metals all gave solid homogeneous tablets, the hardness of which was greater the higher the melting point of the salt. The anhydrous sulphates and the stable hydrated sulphates gave mechanically weak, "powdery" tablets or showed no cohesion, whilst those hydrated sulphates stable only at low temperatures, such as $\text{Al}_2(\text{SO}_4)_3$, $18\text{H}_2\text{O}$, gave "smooth" tablets (smooth surface with pulverulent interior) of high crushing strength. Oxides gave "powdery" tablets with the exception of PbO and PbO_2 , which gave "smooth" tablets. The sulphides and nitrates mostly gave "smooth" tablets, whilst those from most of the carbonates were "powdery." Calcium fluoride, silica, and carborundum show practically no cohesion. The influence of grain size is very marked in those substances which form "smooth" and "powdery" tablets, the hardness of the tablet being much greater with a fine-grained than with a coarse powder. When a substance having good binding properties was mixed in varying proportions with another which alone showed no cohesion, the effect was not proportional to the amount of the binding substance added; thus when potassium chloride was mixed with silica the improvement in the strength of the tablet was small up to 25% KCl and afterwards increased rapidly as the proportion of KCl increased. The curves obtained were much more regular when fine-grained particles were used than with coarse particles. Both hardness and density of the tablet, in the case of "powdery" and "smooth" bodies, increased with increasing pressure up to 9800 kilos. per sq. cm.; the density increased rapidly up to a certain point, beyond which it was practically constant. In the case of gypsum the final density under high pressure was equal to the density of the crystals, and in the cases of sodium and potassium chlorides the true density of the substance was very nearly attained. Those substances which form "plastic" crystals have the best cohesive properties under pressure, probably because the crystal particles, being deformable under pressure, are better able to expel the entrapped air particles and bind together than non-plastic crystals.—E. H. R.

By-product recovery in coke manufacture. Berthelot. See IIa.

Oil shales. Simpson. See IIa.

Iron and sulphuric acid. Fawsitt and Pain. See X.

Molybdenum and water. Chaudron. See X.

Melting point methods. Dana and Foote. See XXIII.

Tin salts. Mazuir. See XXIII.

PATENTS.

Acids; Manufacture of —. Sulphuric acid manufacture. E. D. Chaplin, New York. U.S. Pats. (A) 1,325,711 and (n) 1,325,712, 23.12.19. Appl., 27.1.16 and 10.3.16.

(A) CONCENTRATED sulphuric and hydrochloric acids are simultaneously formed by mixing sulphur di-

oxide, chlorine, and an inert gas with steam, the mixing being effected whilst reducing the pressure to which the steam is subjected. (b) Chlorine, employed in the manufacture of sulphuric acid, is again used by circulating hydrogen and chlorine continuously in a multi-stage circuit, the gases whilst combined as hydrochloric acid being mixed with oxygen and a neutral gas, each being supplied to the circuit at one stage thereof in a continuous stream. The mixture is heated to form water vapour and liberate the chlorine, whilst in a further stage sulphur dioxide and steam are supplied in continuous streams. The sulphur dioxide is converted into the trioxide, and the hydrogen liberated from the water vapour forms hydrochloric acid.

—B. N.

Nitric acid vapours; Condenser for —. A. Hough, New York. U.S. Pat. 1,326,267, 30.12.19. Appl., 30.1.19.

A VERTICAL, sectional casing contains a number of pipes arranged transversely, one above the other, and joined at adjacent ends—the joints being outside the casing—to form a continuous, zig-zag channel for the passage of the acid vapour. Inlet and outlet pipes for the cooling fluid are provided at the top and bottom of the casing, in which baffles are also disposed to compel the cooling fluid to traverse a tortuous path.—W. E. F. P.

Nitrous gases; Process for the sudden cooling of hot —. R. Mewes, Berlin. Ger. Pat. 298,846, 15.3.14.

THE hot gases are quickly and intimately mixed with 1–6 times their volume of previously cooled compressed air at the same or higher pressure, and the gas mixture is allowed to expand in an expansion engine so that the greater part of the heat and mechanical energy can be utilised to perform useful work, e.g., to compress the oxygen necessary for the reaction. The nitrous gases are then used for the manufacture of nitric acid or alkali nitrates.—A. R. P.

Ammonia; Catalyst for the oxidation of — by air or oxygen. W. Manchot and J. Haas, Munich. Ger. Pat. 300,651, 1.8.16.

SILVER oxide is intimately mixed with pumice and reduced by hydrogen at a little below 100° C., or with carbon monoxide at the ordinary temperature. Owing to the heat produced in the reaction the latter gas is, preferably, mixed with a considerable amount of air or nitrogen and passed over the mass at a temperature not exceeding 40° C. The silver oxide should be pure and dry; even very small quantities of impurities greatly impede the reduction. Ammonia, mixed with air or oxygen, is passed over the catalyst, which is heated until the reaction commences, after which sufficient heat is generated to continue the combustion. The temperature of the mass may be lowered to a barely visible red-heat or lower by altering the rate of flow of the gases. Oxides of nitrogen are the chief products at the higher temperatures and nitric acid at the lower.—A. R. P.

Nitric acid; Process for the production of high-grade —. Wülfing, Dahl, and Co. A.-G., Barmen. Ger. Pat. 300,712, 28.6.14.

ENOUGH strong nitric acid is mixed with sulphuric acid to make the H_2SO_4 content of the mixture equal to that of 60° B. (1.71 sp. gr.) acid. Saltpetre is then added and the mixture distilled, keeping the temperature as long as possible between 100° and 135° C. In this way all the nitric acid added, together with 60–70% of that in the nitrate, is recovered as acid of 48° B. (1.495 sp. gr.), and no decomposition of the nitric acid takes place.

—A. R. P.

Tungstates; Method of and means for the separation of fusible — from the gangue resulting from the decomposition of the ore. C. J. Head, London. Eng. Pat. 136,577, 27.6.17. (Appl. 9215/17.)

A MIXTURE of the finely-divided gangue material with alkali is heated on a plain hearth to produce alkali tungstate, and the charge is transferred to a perforated hearth which forms the cover of a filter-bed through which the molten alkali tungstate drains to the furnace well beneath. The two hearths, consisting of iron plates, are arranged side by side in the furnace. The filter consists of a layer of scrap iron turnings or/and other material, preferably containing wolfram, enclosed between the perforated hearth and a lower perforated plate. The function of the wolfram in the filtering medium is to enrich the alkali tungstate at the expense of any free alkali present.

—W. E. F. P.

Cyanides [for the preparation of ammonia]; Production of —. C. T. Thorssell and H. L. R. Lundén, Gothenburg, Sweden. Eng. Pat. 136,772, 20.6.19. (Appl. 15,560/19.)

IN the production of ammonia by the action of water on cyanides prepared by heating mixtures of coal with alkali- or alkaline-earth metal compounds in a current of nitrogen, the activity of the reaction mass becomes impaired by the formation of alkali- or alkaline-earth silicates and aluminates. According to the present invention the latter compounds are decomposed, and the silica etc. precipitated, after the ammonia-producing stage, by dissolving (or suspending) the mass in water and treating the solution with carbon dioxide or an acid stronger than silicic acid, but containing no element other than oxygen, hydrogen, carbon, and nitrogen (e.g., formic or acetic acid); or by making the solution caustic by the addition of lime.—W. E. F. P.

Chromium; Process of isolating salts of —. G. N. Vis, Paris. U.S. Pat. 1,324,328, 9.12.19. Appl., 22.1.18.

IN the recovery of chromium salts from chrome ores treated with alkali hydroxides the latter are dissolved out by means of organic solvents, and the alkali carbonates, aluminates, and silicates are separated from the chromates by means of carbon dioxide.—J. H. L.

Alkaline-metal monochromates; Process for transforming — into bichromates. G. N. Vis, Paris. U.S. Pat. 1,326,123, 23.12.19. Appl., 18.12.18.

A SOLUTION of alkali monochromate is treated with carbon dioxide and ammonia in presence of excess of ammonium bicarbonate, whereby sodium bicarbonate is precipitated and a solution of ammonium chromate obtained. The latter is treated with lime to produce calcium chromate, which is then converted into alkali bichromate by the addition of alkali bisulphate.—W. E. F. P.

Potassium carbonate; Manufacture of —. E. D. Chaplin, Montclair, N.J. U.S. Pat. 1,325,713, 23.12.19. Appl., 29.12.14.

A MICA mineral is heated with an alkali hydrate (hydroxide) to the point of fusion of the mica, and the product obtained is subjected, whilst the mica is still molten and under conditions to form carbon dioxide, to the action of a "carbon-supplying material" in the presence of oxygen.—B. N.

Barium oxide; Manufacture of —. H. Fleck, Golden, Colo. U.S. Pat. 1,326,332, 30.12.19. Appl., 5.11.17.

A MIXTURE of barium carbonate with petroleum coke is heated to the reduction temperature in a non-silicious container.—W. E. F. P.

Alumina from clay; Process for the extraction of —. H. G. Wildman, Montreal, Canada. U.S. Pat. 1,326,384, 30.12.19. Appl., 6.2.18.

THE alumina of the clay is first rendered soluble and then dissolved in the presence of sulphur dioxide.—W. E. F. P.

Aluminium hydride; Process for extraction of — from refractory bauxites. Recovery of aluminium hydride from strong aluminate liquors. I. Szirmay and A. Tet tleni, Budapest. Ger. Pats. (a) 299,652 and (b) 299,653, 25.1.16.

(A) BAUXITE is heated at 170° C. with caustic soda solution of 45° B. (sp. gr. 1.454) and sufficient lime to combine with the silica (1.7 mols. Na₂O and 1—2 mols. CaO to 1 mol. SiO₂) until the density of the solution rises to 50° B. (sp. gr. 1.53). The iron and titanium are precipitated as oxides and the silica as calcium silicate, and 96—98% of the alumina is obtained as a pure concentrated solution of sodium aluminate. (B) Cold water is added in several portions to the hot alkaline solution; after each addition the mixture is stirred and the precipitate filtered off. The number of steps necessary and the quantity of water added each time depend on whether it is desired to precipitate the greater part of the alumina or to recover the caustic soda in a more highly concentrated solution, and thus reduce the costs of re-concentrating it for use again in the process. The precipitate is washed with a small quantity of water, then digested hot with a solution of an ammonium salt, especially the chloride or sulphate; the resulting sodium salt is easily removed by further washing.—A. R. P.

Method of digesting finely-divided materials. [Extraction of potash from minerals.] T. C. Meadows, New York, and F. L. Sample, Teaneck, N.J., Assignors to American Potash Corporation, New York. U.S. Pat. 1,326,412, 30.12.19. Appl., 6.12.18.

IN a method of digesting a mixture of lime, water, and finely-divided potassium-bearing mineral under pressure the mixture is forced continuously through a heated pipe coil in which the desired pressure is maintained, and the potassium-bearing solution is separated.—W. E. F. P.

[Calcium] cyanamide-oven. G. E. Cox, Niagara Falls, N.Y., Assignor to American Cyanamid Co., New York. U.S. Pat. 1,326,412, 30.12.19. Appl., 21.7.19.

IN a cyanamide oven having a refractory lining and a resistor the latter is surrounded by a combustible tube, and a number of supporting, non-combustible surfaces, separated by interstices, are provided in close proximity to the refractory lining. A smooth sheet of combustible material rests against these supporting surfaces, and means are provided for introducing nitrogen into the oven and for conveying the gas thence to the tube.

—W. E. F. P.

Sulphur; Preparation of — from sodium bisulphate. A.-G. Dynamit Nobel, Vienna. Ger. Pat. 300,762, 18.4.16.

SODIUM bisulphate is heated with carbon and the issuing gases are passed into a solution of the sodium sulphide produced in a previous operation. The resulting solution of thiosulphate and sulphide is acidified with sulphuric acid or, preferably, bisulphate. The sodium sulphide may also be treated with the condensed gases produced during the reduction of the sulphate in such a manner that sulphur dioxide is always present in excess to prevent formation of, or decompose, hydrogen sulphide. The black bisulphate produced as described in Ger. Pat. 263,120 (this J., 1913, 909) may be calcined, giving the normal sulphate and sulphur dioxide;

the former is reduced to sulphide, which is treated with the gases produced during the calcining operation.—A. R. P.

Iron oxide slimes produced in the removal of iron from industrial water; Process for the conversion of — by aeration into a chemically active form. Praetorius und Co., Breslau. Ger. Pat. 305,083, 20.1.17.

THE material is dried and heated until incandescence spreads throughout the mass, which is then cooled out of contact with the air. The product, which consists of ferric and ferrous oxides, with possibly a small amount of metallic iron, dissolves slowly but almost completely in lemon-juice at 80° C. It is converted by ignition in the air into ferric oxide, which may also be prepared by continuing the heating of the original material in the air for some time after incandescence has ceased. The ferric oxide may be used as a pigment, for the preparation of thermit mixtures, or for the preservation of food.—A. R. P.

Ferrosoferric oxide; Process for the production of a — suitable for aluminothermic use. G. Bredig, P. Askenasy, and E. Schlumberger, Karlsruhe. Ger. Pat. 314,697, 19.2.16.

FERRIC oxide, e.g., burnt pyrites substantially free from sulphur, is charged continuously into a shaft furnace, through which an ascending or descending current of combustible gas is passed, the operation being so controlled that the reaction takes place in a manner similar to surface combustion; the molten material is discharged at the bottom. This product, which contains more ferrous oxide than corresponds to the formula Fe₃O₄, is atomised according to known methods, e.g., by forcing steam through the molten mass.—A. R. P.

Ammonium sulphate; Production of — from coal gas or producer gas. C. Francke, Berlin. Ger. Pat. 314,598, 30.12.17.

THE gas is mixed with sulphur dioxide at a temperature between 60° and 80° C. Dust is removed from the mixture, for example, by electrical precipitation, and the cleaned gas is subjected to the action of ozone. The ozone should be of high concentration and used in such quantity that the mixture does not contain more than 4% of oxygen. Sulphur is deposited along with liquid products of the reaction, and after separating it by filtration the filtrate is evaporated. A residue of ammonium sulphate is thus obtained. The sulphur dioxide driven off is used to treat a further quantity of gas.

—J. S. G. T.

Thionate solutions containing sulphite and bisulphite, such as are obtained by washing gases containing ammonia and hydrogen sulphide with polythionate solutions; Conversion of — into pure thionate solution. W. Feld, Linz. Ger. Pat. 314,628, 27.8.18.

SULPHUR is introduced into the solution at a temperature above the melting point of sulphur. The sulphur may be previously heated or melted, and the thionate solution, prior to, during, and subsequently to the introduction of the sulphur, is heated under pressure. The molten particles of sulphur come into intimate contact with the hot liquor, and thiosulphate formation is soon initiated. Loss of ammonia is diminished by working under pressure.

—J. S. G. T.

Monazite sand; Process for decomposing —. Chem. Fabr. Heilgensee Dr. V. Helholt und Dr. Remso, G.m.b.H., Heilgensee. Ger. Pat. 315,371, 20.3.18.

MONAZITE sand is boiled with caustic soda solution, whereby the rare earths are obtained as hydroxides,

and the phosphoric acid is recovered as sodium phosphate.—A. R. P.

Carbide; Process of producing —. J. H. Reid, Newark, N.J., Assignor to International Nitrogen Co. U.S. Pat. 1,327,736, 13.1.20. Appl., 15.3.13.

SEE Eng. Pat. 133,098 of 1918; this J., 1919, 900 A.

Potash-bearing silicate minerals; Process of extracting potassium [compounds] from —. *Process of obtaining combined potassium from minerals.* (A) S. R. Scholes, Beaver, and R. F. Brenner, Rochester, and (B) S. R. Scholes, Assignors to H. C. Fry Glass Co., Rochester, Pa. U.S. Pats. 1,327,781—2, 13.1.20. Appl., 10.5 and 20.8.17.

SEE Eng. Pat. 117,755 of 1917; this J., 1918, 579 A.

Cyanides and nitrides; Method for synthetic production of —. A. R. Lindblad, Stockholm. U.S. Pat. 1,328,575, 20.1.20. Appl., 18.10.18.

SEE Eng. Pat. 119,243 of 1918; this J., 1919, 681 A.

VIII.—GLASS; CERAMICS.

Glassworks furnaces and gas producers; Recent improvements in the designs of —. J. S. Atkinson. J. Soc. Glass Tech., 1919, 3, 148—166.

THE author recommends a Siemens type of open producer with a stepped grate. Water flows along a channel fitted to each grate bar and through slots in the channel, forming a curtain of vapour, some of which enters the producer with the air induced by natural draught. The gas outlet is situated about half-way down the back wall of the producer, and the level of the fuel (coke) is maintained above the top of this outlet. It is claimed that there is a great saving in labour, and that unskilled labour can be used. A pressure gas-producer with automatic feed and a Chapman stirrer is also described. The Stein recuperative glass furnace resembles the Hermansen furnace (this J., 1917, 336) in appearance, but in the former the recuperators are placed on each side, whilst in the latter they are under the furnace, and the air passages in the recuperators of the Stein furnace are vertical instead of horizontal, and there is a constant rise from the point where the air enters to that at which it leaves; this allows a positive flame pressure to be maintained on the hearth of the furnace. In a furnace with 12 covered pots each of 450 kilos. capacity, producing glass for lamp chimneys, the fuel consumption in the Stein furnace was 2000—2500 kilos. per 24 hours or 37—46% of the weight of glass melted, as compared with 65—110% in the Hermansen furnace.

—A. B. S.

[Glass] tank furnaces; Preliminary firing of —. E. E. Fisher. J. Soc. Glass Tech., 1919, 3, 147—148.

THE empty tank should be heated to the highest possible temperature, say 2700° F. (1480° C.), before charging, and maintained at that temperature in order to facilitate the formation of sillimanite and produce resistant tank blocks.

—A. B. S.

Chemical glassware; Investigations on —. C. M. M. Muirhead and W. E. S. Turner. J. Soc. Glass Tech., 1919, 3, 129—131.

AN American soda-zinc borosilicate glass containing SiO₂ 71.03, ZnO 5.30, Na₂O 10.74, and B₂O₃ 7.44%, with small amounts of alumina, lime, iron oxide, magnesia, potash, antimony, and arsenic was tested as previously described (this J., 1918, 692 A). This glass easily falls within the "good" resistance glasses.—A. B. S.

Chemical glassware; Proposals for the standardisation of —. H. Thiene. Z. angew. Chem., 1920, 33, 18—20.

THE author has been commissioned to work out proposals for the standardisation of chemical glassware in Germany, and now puts forward his detailed suggestions for criticism. He proposes that flasks of the same capacity of all types shall have the same width of neck, and would abolish tall conical beakers, round-bottom (not shallow) evaporating dishes, round-bottom flasks with short necks, and all types of Kjeldahl flask except the long-necked, round-bottom variety.—J. K.

Melting point methods. Dana and Foote. See XXIII.

PATENTS.

Glass. C. R. Peregrine, Charleroi, Pa., Assignor to Macbeth-Evans Glass Co. U.S. Pat. 1,327,569, 6.1.20. Appl., 24.9.17.

A GLASS is formed by fusing a mixture of 1500 parts of clear glass batch with compounds containing respectively 90—150 parts of aluminium and 90—140 parts of fluorine.—A. B. S.

Retorts and furnaces; Glazing cement for use in fireclay —. S. A. McMinn, London. Eng. Pat. 137,419, 28.2.19. (Appl. 5075/19.)

A GLAZING cement for lining retorts, furnaces, etc., is composed of a mixture of 33—50% of ground glass with fireclay or with a mixture of equal parts of a "clay" containing over 90% of silica and china clay.—A. B. S.

Diaspore; Process of treating — [for use as an abrasive]. O. Hutchins, Assignor to The Carborundum Co., Niagara Falls, N.Y. U.S. Pat. 1,326,595, 30.12.19. Appl., 28.6.19.

THE alumina content of diaspore is increased, and the material rendered more suitable for use as an abrasive, by expelling most of the combined water from the crushed mineral, mixing the product with a reducing agent, fusing the mixture in an electric furnace, and separating the reduced impurities.

—W. E. F. P.

Refractory plastic [material]; Process for making a —. E. R. Stewell, Oden, Mich. U.S. Pat. 1,327,448, 6.1.20. Appl., 16.4.19.

SILICON carbide containing free silicon is treated with a caustic alkali solution to generate hydrogen and, after the evolution of gas has ceased, is stirred so as to reduce it to its original volume.—A. B. S.

Abrasive material and process of making same. N. C. Harrison, Boston, Mass., U.S.A. Eng. Pat. 137,490, 14.7.19. (Appl. 17,560/19.)

SEE U.S. Pat. 1,314,061 of 1919; this J., 1919, 769 A.

IX.—BUILDING MATERIALS.

Slag bricks. R. Malfait, Rev. Mat. Construction et Trav. Publ., 1919, 123, 335—336.

GRANULATED slag contains a variable, but excessive, proportion of water, some very porous slags containing 60%. After storing under cover for at least a month the water content seldom exceeds 6—12%, and the slag can then be ground readily; the water still remaining effects the hydration of the calcium silicate, and thus forms the chief binding agent in the bricks. Any surplus of water dissolves part of the lime, and tends to disintegrate the bricks. In slag bricks subjected to the action of sea-water or other saline solutions, the most important factor is porosity, the least porous bricks being the most resistant. Greater durability is

secured by addition of colloidal gels such as glue, tannin, etc. The use of pitch as a binder gives still better results. Bricks made of ground slag mixed with finely ground chalk, when immersed for 8 days, gave up 25 grms. to distilled water, 20 grms. to a saturated (0.2%) solution of calcium sulphate, and 15 grms. to a 1% solution of a mixture of sodium, magnesium, and calcium sulphates. 100 grms. of powdered slag brick lost 3.235 grms. in distilled water and 2.560 grms. in calcium sulphate solution, and gained 2.195 grms. in a solution of mixed sulphates. Slag bricks immersed in a 3% solution of common salt for a month lost six to eight times as much as in distilled water, but on a longer immersion no further loss was observed. Although slag bricks are much less porous than those made of clay, they should not usually be employed in situations where they are in contact with sea water.—A. B. S.

Wood; Inaccuracy of treatment tests of impregnated — E. Bateman. *Chem. and Met. Eng.*, 1920, 22, 57—59.

ANALYSIS of wood which has been treated with an inorganic salt preservative seldom shows the same value for the salt absorbed as that calculated from the increase in weight of the treated wood. Blocks of wood in the green, air dried, and oven dried condition respectively were impregnated in a solution of calcium chloride. Concordant results by the two methods were obtained only when the wood was perfectly dry before treatment. Moisture present in wood before treatment acts as a diluent of the preserving solution, and any method of measuring absorption which is based on a measurement of the weight or volume of the solution before and after treatment is liable to considerable error. There is no reason to suppose that any chemical combination results from the treatment of wood with solutions of the usual inorganic salts.—C. A. K.

PATENTS.

Cementing material obtained from greensand. H. W. Charlton, Jones Point, N.Y., Assignor to American Potash Corporation. U.S. Pat. 1,327,145, 6.1.20. Appl., 22.5.17.

A GREENISH- or brownish-white cementing material is made by digesting a mixture of greensand and lime. When freshly made the product is plastic and self-hardening, and it can be still further hardened by treatment with steam.—A. B. S.

Paring and other purposes; Composition for — S. R. Murray, Indianapolis, Ind. U.S. Pat. 1,327,726, 13.1.20. Appl., 1.4.18.

EARTHY materials are ground, heated, mixed with sufficient bituminous cement to lightly coat the particles, cooled, re-ground, heated, and mixed with a further quantity of bituminous cement

—A. B. S.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Blast-furnace reactions; Equilibrium in — E. Terres and A. Pongracz. *Z. Elektrochem.*, 1919, 25, 386—407.

THE equilibrium conditions in the reaction $\text{Fe} + \text{CO} \rightleftharpoons \text{FeO} + \text{CO}$ have been investigated by a number of different observers with highly discordant results. The authors have modified the usual procedure for studying such a reaction. Starting with pure iron and carbon dioxide, the reaction was allowed to proceed for seven hours at a definite temperature and the gases were then analysed. The experiment was repeated at a number of temperatures between 550° and 950° C. and

the per cent. CO was plotted against the temperature. A similar set of experiments was made allowing the reaction to proceed for sixteen hours, and in the same way the seven and sixteen hour experiments were repeated, starting from ferrous oxide (prepared by the oxidation of pure iron with carbon dioxide) and carbon monoxide. In this way four graphs were obtained, all of them straight lines and all converging to a point at about 950° C. with about 75% CO in the gas, indicating that in seven hours equilibrium is already attained at this temperature. The two sixteen-hour graphs fell between the two seven-hour graphs and much closer together; they converge at about 810° C., showing that at this temperature complete equilibrium is reached in sixteen hours. The true equilibrium graph evidently lies midway between the two sixteen-hour graphs. It shows no minimum, but is a straight line, confirming the results of Schenck (this J., 1903, 630; 1905, 803; 1907, 692) and Levin (this J., 1912, 926) and contradicting those of Baur and Glaessner (this J., 1903, 699). A number of very careful experiments showed that the composition of the gas phase at equilibrium depends on the quantity of iron used. This can be explained on the assumption that the oxide phase is a mixture or solid solution of two components. Analyses of the solid phase showed that this always contained both ferrous and ferric oxides. The proportion of ferrous to ferric oxide never approached the composition required for Fe_3O_4 . The observations are therefore quite in accordance with the requirements of the phase rule. The fact that the proportion of carbon monoxide in the gas phase increases with the proportion of metallic iron present in the solid phase is of importance in the equilibrium of the blast furnace. It was shown experimentally that the fall in pressure always observed when carbon dioxide and iron are first heated together is due, not to a chemical reaction, but to absorption of the gas by the metal.

For carrying out the analysis of the solid phase a special process was devised. The metallic iron was dissolved out by means of mercuric chloride, which converts the metal into soluble ferrous chloride without attacking the oxides. The ferrous chloride was estimated by titrating with permanganate. The oxides were then dissolved in hydrochloric acid, and in separate portions the ferrous iron was determined by titration with permanganate and the ferric with titanous chloride. As a check, the whole of the iron in the sample already titrated with permanganate was again determined by titration with titanous chloride.—E. H. R.

Iron; Behaviour of — in contact with sulphuric acid. C. E. Fawsitt and A. A. Pain. *J. Proc. Roy. Soc. N.S.W.*, 1918, 52, 396—405. (See also this J., 1914, 231.)

DIFFERENCES in electro-chemical behaviour show that no real similarity exists between iron which is slowly dissolving in concentrated sulphuric acid and iron rendered "passive" by dipping into nitric acid. The action of the acid produces a form of ferrous sulphate monohydrate on the surface of the iron, which causes the slowing down or cessation of the reaction. In general, increasing dilution of the acid with water results in greater reactivity, but certain anomalies were observed in this respect, notably that 89.2% acid had considerably less action on the metal than 91%, 90.9%, 87.9%, or 85% acid. The solvent effect is in all cases noticeably increased by shaking the vessel containing the acid and the metal.—G. F. M.

Zinc ores; Electric resistance furnace for the treatment of — C. H. Fulton. *Amer. Inst. Min. and Met. Eng.*, Sep., 1919. *Chem. and Met. Eng.*, 1920, 22, 73—79.

Oxidised zinc ore, or roasted zinc concentrate, is

mixed with crushed coke and pitch, and formed into briquettes, which are hardened by baking at 400°–500° C. An electric current is then passed through a series of such briquettes, which act as resistors when arranged in columns, and the briquettes are heated to such a degree that the zinc is completely distilled. During the distillation process the briquettes lose 30–50% of their weight, but maintain their shape and volume. When made up with coke and pitch they become practical conductors at 450°–500° C., but if the coke is replaced by a proportion of coal the briquettes do not become conductors until a temperature of 620°–750° C. is reached, and a higher degree of preheating is necessary. The furnace consists of a firebrick base, the central part of which holds the condenser structure. The end portions contain electrode terminals and serve to support the briquette charge and a covering sheet steel retort. The two briquette charges are distilled alternately, and the retort cover is transferred over the one which is being heated. The retort bases and the condenser are heated independently to prevent the formation of "blue powder." A furnace taking 19 briquettes, weighing 600 lb. each, would have a capacity of about 8.5 tons of zinc concentrate per retort per 24 hours.

—C. A. K.

Zinc ores; Electric resistance furnace of large capacity for —. C. H. Fulton. Chem. and Met. Eng., 1920, 22, 130–135.

In operating a zinc-distilling furnace, using briquettes of zinc ore and a reducing material (see preceding abstract), no advantage was secured by replacing the bottom connexion between the retort chamber and the condenser by a similar connecting passage at the top. The condenser consisted of a steel shell lined with "sil-o-cel" brick, with an inner lining of firebrick. A longitudinal partition of firebrick caused a double passage of the gases. The base of the furnace was made of a movable pattern, so that the briquettes might be set in position after pressing, heated to about 500° C., and the whole quickly conveyed to the furnace for distillation, thus conserving the sensible heat of the charge after baking. A higher recovery of zinc is obtained by this method of distillation than by present-day retort processes, and estimated costs show a marked reduction over present costs.

—C. A. K.

Zinc; Recovery of — from low-grade and complex ores. D. A. Lyon and O. C. Ralston. U.S. Bureau of Mines, Bull. 168, 1919. 145 pages.

No technical difficulty exists in the successful flotation of zinc in the slimes from the Joplin district, though units of the size required are not capable of being developed at present. Roasting the mineral at 600°–900° C., in presence of salt, was unsuccessful, as in most cases zinc sulphide was not completely oxidised, and at no time did it seem possible to obtain more than 50% of the zinc in a form soluble in acid brine. For purposes of retort smelting it is important that no sulphur is left in the calcined material, and the chief objection to existing types of furnaces is that when the calcination of the blende is carried to the point of satisfactory elimination of sulphur, the costs of building and operation are very high. On the other hand, the conversion of zinc sulphide into zinc sulphate in the furnace occurs only partially, even at temperatures below that at which zinc sulphate decomposes (630° C.). It has been stated that 30–50% of the zinc is sulphated at Great Falls, Montana, in an Anaconda-Wedge roaster working at about 600° C. Marmatite ores (a double sulphide of iron and zinc) give better results in sulphate roasting than "free" ores, because of the catalytic effect of iron oxide, but a proportion of the zinc is rendered in-

soluble in sulphuric acid by the formation of zinc ferrites. Treatment of a suspension of zinc calcines in water by roaster gases containing sulphur dioxide gives as good an extraction of zinc (as bisulphite) as extraction with sulphuric acid, but it is claimed that the recovery of insoluble zinc sulphate by boiling the bisulphite solution is not complete owing to oxidation of the zinc to zinc sulphate. Chemical methods of precipitating zinc oxide from the solutions obtained by leaching the calcined ore are difficult.—C. A. K.

Chloride volatilisation experiments on ore from Tintic district, Utah, U.S.A. Chem. and Met. Eng., 1920, 22, 52–53.

ALTHOUGH laboratory investigations have almost invariably indicated that an economical recovery of metals which form volatile chlorides should be secured by roasting the mineral with sodium chloride, difficulties have always been encountered during trials on a semi-commercial scale. Laboratory work showed that a volatilisation of 90% of the gold, 91% of the silver, 87% of the lead, and 72% of the zinc could be obtained by muffle scorification of an ore from Eureka, Utah, which contained 62% insoluble matter, 5% CaO, 8% Pb, 2% Zn, and 15 oz. Ag and 0.1 oz. Au per ton. The composition and characteristic properties of this ore are such as to render it unsuitable for treatment by any developed process. Silver chloride, which is practically non-volatile at the temperature of the furnace (850° C.), becomes volatile when lead, sodium, or calcium chlorides are present. Sintering is not favourable to volatilisation, and may be prevented by the addition of lime. The lack of success in large scale trials is attributed to incomplete chloridising and to the difficulty of maintaining a proper temperature and atmosphere in the furnace. Zinc and copper, together with a portion of the lead, can be removed by leaching the fume with water, and gold, silver, and the remainder of the lead are recovered by fusing the residue with lime and coke. (See also preceding abstract.)

—C. A. K.

Copper; Production of — from secondary material. L. Addicks. Chem. and Met. Eng., 1920, 22, 119–122.

MISCELLANEOUS scrap copper-bearing material can always be introduced into the slag cupola, the primary function of which is to treat the anode and bar furnace slags, but little is effected except to concentrate the metals into more convenient shape. Scrap material is usually charged into the anode furnace, which ensures saving any contained silver and gold and getting the copper into electrolytic form. The delay in blowing and skimming caused by the addition of impure material is an objection, and the use of a separate furnace for this class of work might be an economy. By use of a reverberatory furnace having a magnesite bottom and chrome walls and arch, a material free from silver and gold (e.g., brass) can be converted directly into a casting copper. The metallic oxides oxidise in the order of their basicity, and a tin-copper slag, followed by a cobalt-copper slag, may be recovered. Zinc and iron are not of sufficient value to warrant special treatment. Nickel is usually separated as a soluble sulphate during electrolytic refining, and lead may be readily removed from molten copper in the form of litharge, which has been noticed at times to liquate through the furnace bottom.

—C. A. K.

Metals and alloys; Reports of the sub-committee for the standardisation of —. Doerinczel, Philippi, Bauer, and E. H. Schulz. Metall u. Erz, 1919, 16, 591–600.

Four reports are presented, dealing with the

nomenclature and the amount of permissible impurities in brasses, bronzes, and other copper alloys; and also a table showing the properties of the more important brasses and two tabulated schemes for the standardisation of brasses and bronzes showing the limiting amount of impurity allowable in many commercial alloys. The recommendations as to the naming of the alloys are substantially the same as those put forward by the Institute of Metals Committee (this J., 1914, 318).

—A. R. P.

Copper-antimony alloys; Expansion of —. P. Braesco. *Comptes rend.*, 1920, 170, 103—105.

THE mean coefficient of expansion between 100° and 300° C. of copper-antimony alloys increases with the percentage of antimony present up to 38.6% Sb, and then diminishes, the differences between the results with quenched and annealed alloys being only very slight. Certain alloys within the range 4 to 50% Sb show for a given temperature a considerable increase in volume, with increase in the antimony contents. Here, again, the maximum is reached at 38.6% Sb, and then rapidly drops to zero at 50% Sb. The maximum point in both cases corresponds to the definite compound Cu_3Sb .

—W. G.

Bismuth, tin, and wolfram ores; Treatment of —. W. E. Hitchcock and J. R. Pound. *Proc. Austral. Inst. Min. and Met.*, 1919, [35], 33—73.

A DETAILED account of the methods employed in mining, milling, and magnetic separation of tin, bismuth, and tungsten ores is given. After hand picking of the ore both before and after breaking the rock, the material is further reduced between rolls, and passes over jigs of the Hartz type. The overflow from the hydraulic classifiers passes to two Rittingen classifiers, and is fed on to Wilfley tables, from which the concentrates are treated in a buddle. All concentrates are dried and divided into four sizes by screening, each size being treated separately on a Wetherill magnetic separator. For economic handling the ore must be fed evenly on to the conveying belt and the full effect of the magnetic field utilised by bringing the magnets as close to the ore as possible. The speed of the cross belts should carry the particles clear of the magnetic field; a speed of about 300 ft. per min. is usually sufficient. This type of machine is not suitable for the treatment of slimes, and the best size of material is that "minus 10 and plus 20 mesh," averaging 0.04 in. diameter. The "seconds" concentrates yield after magnetic separation pyritic wolfram and pyritic tin-bismuth products, and these are roasted separately to convert the pyrites into magnetic iron sulphide or oxide. A further magnetic separation follows. The grade of the lower class concentrates is raised by separating the magnetic minerals (e.g., ilmenite, chromite, magnetite), and the tin is obtained along with the other non-magnetic minerals, such as quartz, zircon, and non-magnetic spinel.—C. A. K.

Wulfenite; its concentration and separation from barite. J. P. Bonardi. *J. Franklin Inst.*, 1920, 189, 47—61.

A RESUME is given of the occurrence in America of molybdenum ores, tests for molybdenum, methods for its quantitative estimation in ores, the properties of wulfenite, and the ordinary methods of concentration of the more commonly occurring wulfenite ores. This is followed by a detailed description of laboratory experiments on a "middling" material containing 52.2% barite and 5.50% molybdenum as wulfenite, along with quartz and some lead-containing minerals—mimetite, pyromorphite, vanadinite, etc. The original ore contained about 2% of molybdenum and was concentrated on a

Wilfley table, leaving the middling product experimented on. The ore was examined microscopically and subjected to screening, Wilfley table, and flotation tests, tabulated results being given. The flotation test, after a sulphiding treatment with sodium sulphide, showed little or no separation of the wulfenite from the barite owing to the presence of iron, and some of the wulfenite was lost by solution in the sulphiding liquor. The Wilfley table method gave a concentrate with 14% molybdenum and a recovery of from 60 to 70%.—B. V. S.

Molybdenum; Reversible reaction of water on —. G. Chaudron. *Comptes rend.*, 1920, 170, 182—185.

A STUDY of the system water vapour, molybdenum, hydrogen, molybdenum dioxide over the temperature range 700°—1100° C. The values of $K = p_{\text{H}_2\text{O}}/p_{\text{H}_2}$, obtained by the oxidation of the metal or the reduction of the oxide are in close agreement.—W. G.

By-product recovery in coke manufacture. Berthelot. See HA.

Cool pyrites. Yancey. See VII.

Melting point methods. Dana and Foote. See XXIII.

PATENTS.

Iron and steel; Process and ladle for the decaridation of — by means of calcium carbide. Röchlingsche Eisen- und Stahlwerke G.m.b.H., and W. Rodenhäuser, Völklingen. Ger. Pat. 298,847, 31.5.16.

MOLTEN calcium carbide is added to the metal bath from a ladle, the lining of which consists of carbon or a mixture of carbon and other refractory, the ladle being preheated electrically to prevent chilling of the carbide.—A. R. P.

Blast furnace gases; Apparatus relating to the cooling of —. J. E. Weyman, Newcastle-upon-Tyne. Eng. Pat. 137,378, 16.1.19. (Appl. 1150/19.)

INCREASED efficiency of cooling is obtained by using a forced circulation of the cooling water. The top box of the economiser is divided vertically in halves. Pipes extend from the top box vertically downwards, each pipe being divided by a partition which extends almost to the bottom of the pipe. The cooling liquid passes along one side of the top box and down half of each of the pipes attached to it, and up the other half to the other side of the top box. The pipes are circular in section to allow of the use of scraping gear and scrapers in the usual manner.—W. P.

Electric furnace; Arc-type — [for melting metals] and method of operating same. W. E. Moore, Assignor to Pittsburgh Engineering Works, Pittsburgh, Pa. U.S. Pat. 1,326,083, 23.12.19. Appl. 30.7.17.

METAL is melted in an electric furnace of the arc type, provided with a number of electrodes, by effecting a short-circuit or contact between the charge and one of the electrodes, whilst maintaining an arc between the charge and another of the electrodes; subsequently an arc is formed between the electrode, previously short-circuited, and the charge.—B. N.

Ores; Process and apparatus for extracting [roasting] —. R. Wallner, Bisbee, Ariz. U.S. Pat. 1,326,125, 23.12.19. Appl., 20.10.17.

ORE is roasted in a rotating retort which is heated externally, and through which fresh air is forced in opposite directions alternately.—W. E. F. P.

Fine-grained ores; Process for smelting — by introducing them into the blast furnace by means of the blast. O. Baumann, Amberg. Ger. Pat. 314,777, 3.2.18.

IN working the process described in Ger. Pat. 312,935 (this J., 1920, 70 A) the blast-furnace shaft is fed with nearly spherical lumps of limestone and coarse ore and fired with pulverised coal or combustible furnace gases blown into the tuyères together with fine-grained ore. Each tuyère is provided with a valve which regulates the amount of dust carried according to the volume of the blast. The shaft may be provided with an upper row of tuyères, through which air is introduced, whereby the gases are converted into carbon dioxide and the coarse ore is roasted in the shaft. The production of steel in the blast-furnace by this method is claimed without the intermediate production of pig-iron rich in carbon.—A. R. P.

Acid or like baths for treating wire. Oates and Green, Ltd., and C. H. Ashton, Halifax. Eng. Pat. 137,414, 25.2.19. (Appl. 4665/19.)

Titaniferous iron ores; Process for the direct production of refined iron and refined steel from —. J. J. and W. A. Loke, The Hague, Netherlands. U.S. Pat. 1,328,636, 20.1.20. Appl., 29.9.16.

SEE Eng. Pat. 5618 of 1915; this J., 1916, 52.

Steel; Process for making basic — in open-hearth steel furnaces. C. H. F. Bagley, Stockton-upon-Tees. U.S. Pat. 1,328,803, 27.1.20. Appl., 19.9.18.

SEE Eng. Pat. 116,663 of 1918; this J., 1918, 472 A.

Iron, steel, or cast-iron; Process of directly and completely transforming and reducing iron ores into —. G. Jakova-Merturi, Paris. U.S. Pat. 1,329,055, 27.1.20. Appl., 16.10.17.

SEE Eng. Pat. 129,351 of 1917; this J., 1919, 684 A.

Metallic objects; Method of developing defects in —. A. S. Macdonald, Great Neck Station, N.Y., and H. P. Macdonald, Montclair, N.J., Assignors to The Sneed and Co. Iron Works, Jersey City, N.J. U.S. Pat. 1,327,341, 6.1.20. Appl., 13.4.18.

SEE Eng. Pat. 134,791 of 1919; this J., 1920, 30 A.

Ferrosferrous oxide. Ger. Pat. 314,697. See VII.

XI.—ELECTRO-CHEMISTRY.

Regenerating chromic acid. McKee and Leo. See VII.

PATENTS.

Electrical separation of substances in suspension in gaseous media; Apparatus for the —. Soc. Gallet et Cie., Paris, and Soc. Poussin Rondeaux et Cie., Pantin, France. Eng. Pat. 116,104, 22.5.18. (Appl. 8529/18.) Int. Conv., 22.5.17.

AN apparatus for the electrical separation of materials in suspension in a gas comprises a perforated or non-perforated tubular electrode, with a wire along the longitudinal axis forming the second electrode and stretched and supported on insulators mounted in attached chambers. The gas inlet is arranged some distance above the insulator chamber, so as to create an electrical protecting zone consisting of a portion of the tubular electrode in which no dust is deposited. In an alternative form the gas inlet opens into the tubular electrode at about the middle of its length, the gas escaping from both ends, or in a further form an electrical protecting zone is formed by an auxiliary tubular electrode of reduced length carried by the insulator chamber.—B. N.

Electrical purification of hot, dust-laden gases; Collecting electrodes for the —. H. Püning, Münster. Ger. Pat. 314,030, 26.6.18.

THE electrodes are composed of refractory material containing alumina, magnesia, and silica, such as porcelain, firebrick, or like materials. With rise of temperature the conductivity of the electrodes increases. By the use of such electrodes gas may be freed from dust without the necessity of lowering its temperature.—J. S. G. T.

Electrodes for electro-osmotic processes. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. Eng. Pat. 135,816, 25.6.18. (Appl. 10,454/18.) Int. Conv., 20.10.17.

Two perforated sheets are connected together by means of distance pieces or by a corrugated sheet so as to form a grate-like carrier, on each side of which a wire fabric is fixed which is easily disconnected; or several wire fabrics of different degrees of fineness may be arranged one above the other on the two sides of the carrier.—B. N.

Electric furnaces. T. H. Watson and Co. (of Sheffield), Ltd., H. A. Greaves, H. Etchells, and W. Travis, Sheffield. Eng. Pat. 136,960, 21.2.19. (Appl. 4320/19.)

A HEARTH or lining, consisting of a layer of electrically conductive broken chromite, is employed in electric furnaces in contact with the metallic connections, and this is surmounted or adjoined by a ramming of silicious material mixed with tar or pitch, with or without finely-divided metal. The upper lining is of a depth sufficient to keep the bath of metal from contact with the chromite or other electrically conductive material employed.—B. N.

Induction-furnace. M. Unger, Pittsfield, Mass., Assignor to General Electric Co. U.S. Pat. 1,326,120, 23.12.19. Appl., 21.4.19.

THE furnace has a melting chamber the side walls of which are inclined outwards from each other at an angle of 60°—90°.—W. E. F. P.

Electric arc furnaces; Method of fixing electrodes in —. Ges. für Elektrostablanlagen m.b.H., Berlin, and W. Rodenhauer, Völklingen. Ger. Pat. 314,884, 27.10.17.

THE frame for making contact with the electrode surrounds the latter, and is angular or U-shaped in section. Below the frame and insulated therefrom is a ring gripping the electrode tightly and forming a support. There is considerable space between the frame and the electrode, and this space, together with that intervening between the ring and the frame, is tightly packed with compressed material containing much carbon.—J. S. G. T.

Battery-plate envelope. C. C. Carpenter, Assignor to U.S. Light and Heat Corporation, Niagara Falls, N.Y. U.S. Pat. 1,325,710, 23.12.19. Appl., 16.4.13. Renewed 8.5.19.

A BATTERY plate is protected by slotting it from one edge to a point adjacent to the opposite edge and enclosing it in alternate "punchings" of soft rubber having integral projections passing through the slots, and strips of metallic zinc arranged in planes at right angles to the surface of the plate. The open ends of the slots are closed and the soft rubber vulcanised to provide a hard rubber envelope, the plate being afterwards treated in an acid bath to dissolve the zinc.—B. N.

Electrode for electrolytic apparatus. R. D. Mershon, New York. U.S. Pat. 1,326,134, 23.12.19. Appl., 2.4.17.

AN electrode for electrolytic apparatus comprises a

crimped or folded plate of sheet metal having apertures in the bends of the crimps or folds.—B. N.

Electrolyte for electrolytic condensers. J. Slepian, Pittsburgh, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,326,297, 30.12.19. Appl., 16.1.19.

An electrolyte for condensers, lightning arresters, rectifiers, and the like comprises an aqueous solution of an alkaline-earth aluminate.—B. N.

Selenium cells; Process for the manufacture of —. O. Hannach, Berlin-Wilmersdorf, and H. Böcker und Co., Berlin. Ger. Pat. 304,261, 14.10.16.

Amorphous selenium is treated with a solution of quinoline, silver is added, and the mixture heated to about 200° C. The mixture is then cooled extremely slowly, say, over a period of from two to three days. Selenium cells constructed of the resulting product are extremely sensitive to light.

—J. S. G. T.

Electrolyte for high temperature combustion electric cells. Siemens und Halske A.-G., Siemensstadt. Ger. Pat. 308,585, 29.3.18.

In place of an electrolyte which is liquid at the working temperature of the cell an electrolyte of the nature of an enamel is employed, which is an electrolytic conductor, is impermeable to gas, and has a coefficient of expansion as nearly as possible the same as that of the metallic electrodes. The enamel is composed essentially of a fused mixture of an alkaline basis such as borax, alkali phosphate or silicate, alkalis or alkali carbonates, together with one or more metallic oxides, these latter being more or less conductors of the second class at ordinary temperatures, but conducting electrolytically at higher temperatures. The oxides of manganese, chromium, iron, vanadium, uranium, etc., may be employed for this purpose. Alternatively the enamel may be constituted of definite compounds of the alkalis and oxides, e.g., sodium ferate, sodium vanadate, or mixtures of such compounds. The enamel is fused, then powdered and employed as an electrolyte in the usual manner. As the enamel is impermeable to gas even in very thin layers, the combustion cell can be constructed so as to possess a very low internal resistance.

—J. S. G. T.

Depolarising agents employed in electric batteries. W. Keil, Berlin. Ger. Pat. 309,242, 16.7.15.

Soot is added to the customary depolarisers. The soot should be free from tarry and greasy matter, and if necessary these are removed by means of alkalis, acids or heat.—J. S. G. T.

Separator; Wooden — [for electric cells]. M. Löffler, Soest. Ger. Pat. 314,723, 27.7.17.

The separator is made of wood which has been immersed in, or treated with, a salt solution so that on subsequent drying crystals of salt are deposited in the pores of the wood. Shrinkage on drying is prevented, and wood so treated can be stored for any length of time. When the wood is brought into contact with acid in an electric cell the salt is removed either in whole or in part or converted into a harmless compound. The separator possesses an extremely high porosity, as the pores of the wood are neither constricted nor choked.—J. S. G. T.

Electric furnaces; Control of power absorbed in —. C. C. Gow and D. F. Campbell, London. U.S. Pat. 1,327,548, 6.1.20. Appl., 24.4.19.

See Eng. Pat. 125,522 of 1918; this J., 1919, 425 A.

Calcium cyanamide oven. U.S. Pat. 1,326,442. See VII.

Electric furnace. U.S. Pat. 1,326,083. See X.

XII.—FATS; OILS; WAXES.

Linseed oil; New hexabromide method for —. L. L. Steele and F. M. Washburn. J. Ind. Eng. Chem., 1920, 12, 52—59.

HEHNER and Mitchell's method of determining the amount of linolenic hexabromide yielded by the fatty acids of linseed oil has been modified by effecting the bromination in a solvent in which the hexabromide is soluble (e.g., chloroform), removing the excess of bromine by the addition of a reagent (amylene), separating the hexabromide and washing it thoroughly with ether previously saturated with hexabromide. The amount of hexabromide thus separated from the fatty acids of seven different samples of linseed oil ranged from 45.6 to 46.6%, duplicate determinations agreeing within about 0.5%. Apparently, therefore, the yield of hexabromide is a more constant value than the iodine value of raw linseed oil. Cottonseed and tung oil fatty acids gave no precipitate, whilst a sample of soya bean oil yielded 2.2%. Experiments with mixtures indicated that it may be possible to detect 5% of oils low in hexabromide yield in linseed oil by this method.—C. A. M.

Squalene: a highly unsaturated hydrocarbon in shark liver oil. M. Tsujimoto. J. Ind. Eng. Chem., 1920, 12, 63—72. (See also this J., 1916, 609, 1121.)

SQUALENE is of most frequent occurrence in the liver oils of the *Squalidae*, but is also found in considerable amount in the oils of several other species, including the frill shark and hasking shark (*Cetorhinidae*). For the approximate quantitative separation of hydrocarbons, including squalene, in shark liver oils 50 grms. of the oil is distilled under a reduced pressure of about 10 mm., and in a current of carbon dioxide or other indifferent gas. The acid value of the distillate is calculated into oleic acid and deducted from the weight of the distillate, the difference giving the approximate quantity (within about 1.6%) of hydrocarbons. Squalene obtained from aizamé shark oil had sp. gr. 0.8591 at 15°/4° C., and 0.8559 at 20°/4° C. In the Livache test it absorbed oxygen very slowly after the first three days, but the amount became nearly constant (24.3%) in 23 to 28 days. The oxygen absorption calculated from the formula $C_{30}H_{50}O_4$ was 23.66%. On debrominating the dodecubromide, $C_{30}H_{14}Br_{12}$, an oily liquid with refractive index 1.4950 at 20° C. was obtained. The formation of the characteristic hexahydrochloride is the most distinctive test for squalene (this J., 1918, 312 A). The hexabromide forms white lustrous crystals which turn brown at 115°—118° C. and melt to a brown liquid at 125°—126° C. Although the hydrocarbon "spinacene" isolated by Chapman from Portuguese shark liver oils (this J., 1917, 392, 602) differs slightly in some of its physical characters from squalene, it seems probable that the two hydrocarbons are identical. (See also J. Chem. Soc., March, 1920.)—C. A. M.

Squalene; Occurrence of — in the egg oil from a shark. M. Tsujimoto. J. Ind. Eng. Chem., 1920, 12, 73.

THE oil separated from the eggs of the Japanese shark *Lepidorkhinus kinbei* (Tanaka) was a light brown substance, which was mainly solid at the ordinary temperature. It had the following characters:—Sp. gr. at 15° C., 0.8997; acid value, 31.3; saponif. value, 107.0; iodine value (Wijs), 177.6; refr. index (20° C.), 1.4769; and unsaponifiable matter, 33.0%. When treated with dry hydrogen chloride in ethereal solution it yielded 20% of a white crystalline precipitate (m. pt. 123°—124° C.), which was identified as squalene hexahydrochloride.

Squalene has also been separated from the egg oil of the frill shark, *Chlamydoselachus anguineus*.

—C. A. M.

Mountain elder (Sambucus racemosa); Fatty oil from the berries of —. H. Thoms. Ber. dents. Pharm. Ges., 1919, 29, 598—627.

The seeds of the berries of the mountain elder (*Sambucus racemosa*, L.) yielded on hot expression 50% of a drying oil consisting essentially of the glycerides of linolic, linolenic, and oleic acids, and possibly of isolinolenic acid, together with some palmitic and stearic acids. Eight samples of the oil of different origin had the following characters: Sp. gr. 0.9242—0.9439 at 21° C.; n_D^{20} (1 sample) = 1.47967; acid value, 3.07—29.24; saponif. value, 186.9—198.1; and iodine value, 156.3—177.4. The fruit oil differed materially in character, a sample giving:—Sp. gr. 0.9214 at 21° C.; solidification pt., -4° C.; acid value, 5.59; saponif. value, 196.5; and iodine value, 98.6. The oil has toxic properties, which, however, can be removed by heating it to 200° C.—C. A. M.

Fat extraction apparatus. Pickel. See XXIII.

PATENTS.

Oxide of nickel catalysts employed in the hydrogenation of fatty bodies; Process for regenerating —. G. N. Vis, Paris. U.S. Pat. 1,326,122, 23.12.19. Appl., 21.11.17.

The spent catalyst is treated with a solvent to remove fatty bodies, calcined to destroy any remaining organic matter, washed with water to remove soluble inorganic impurities, and dried.

—W. E. F. P.

Fats and oils; Process for neutralisation of — by esterification of the free fatty acid. H. Schlinck und Co. A.-G., Hamburg. Ger. Pat. 315,222, 11.6.16.

The fat or oil is heated with glycol, for example: 100 kilos. of olive oil, containing 20% of free fatty acid, is heated for several hours under a reflux condenser with 2.2 kilos. of glycol. The water produced in the reaction is removed by passing a slow current of inert gas through the apparatus, or by the use of a vacuum, or by other suitable means.

—A. R. P.

Detergent; Preparation of a paste —. Chem. Werke München O. Bärlocker, G.m.b.H., Augsburg. Ger. Pat. 314,909, 16.1.18.

Sodium bicarbonate is added to a solution of water-glass to form a pentasilicate in which finely-divided silicic acid is suspended. The mixture is stabilised by the addition of magnesium hydroxide, which prevents hardening. The product dissolves in water to an opalescent liquid.—A. R. P.

Hydrogenating hard oils; Process for preparing catalytic agent for —. K. Kimura, Kobe, Japan. U.S. Pat. 1,327,396, 6.1.20. Appl., 1.8.17.

See Eng. Pat. 118,323 of 1917; this J., 1918, 594 A.

Oil; Extraction of — from vegetable fruits. N. A. Gavin, Kinshasha, Belgian Congo; M. M. Gavin, executrix, Assignor to Lever Bros., Ltd., Port Sunlight. U.S. Pat. 1,328,278, 20.1.20. Appl., 3.3.17.

See Eng. Pat. 111,676 of 1917; this J., 1918, 741 A.

Oil emulsions. U.S. Pat. 1,327,835. See I.

Fat for shortening. U.S. Pat. 1,326,276. See XIXA.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Colophony; Autorization of —. L. Paul. Koloid Zeits., 1919, 25, 241—246.

When a solution of colophony in alkali is treated with hydrochloric acid a voluminous precipitate of γ -pinic acid is obtained; this substance melts at 75°—76° C. when freshly prepared, but in eight months the melting point rises to 88° C. α -Pinic acid has a constant melting point, 81°—83° C., and shows a tendency to form liquid crystals. It is soluble in water and petroleum as also is γ -pinic acid. β -Pinic acid melts at 98°—100° C.—J. F. S.

Resins from Cochín China; Characteristics of some —. P. Nicolardot and C. Coffignier. Bull. Soc. Chim., 1920, 27, 71—74.

TEN specimens of resins from different sources have been examined. They closely resemble one another, are of the soft type and of no special interest for the manufacture of varnishes. Their characteristics, sp. gr., m. pt., and acid and saponification values are tabulated. None of these resins was completely soluble in N/2 alcoholic potassium hydroxide; and in every case the alcoholic solution gave either a milkiness or a precipitate on the addition of water.—W. G.

Abietic acid. D. Johansson. Arkiv Kem., Min., Geol., 1917, 6, No. 19.

ABIETIC acid is reduced by hydrogen in presence of platinum black to dihydroabietic acid, $C_{20}H_{32}O_2$, m.pt. 173°—178° C., but this compound gives approximately the same Hübl-Waller and Winkler iodine values as the parent acid. Further, the values increase with the duration of the reaction, and similar relationships are shown by *d*-pimaric acid and its dihydro derivative. It is, therefore, suggested that the unreduced acids contain at least two non-equivalent double bonds, or that substitution by iodine also occurs. No derivatives of this type could, however, be isolated. The author considers the presence of an aromatic or hydroaromatic nucleus in the molecule of abietic acid to be now definitely established (compare Easterfield and Bagley, this J., 1904, 989). (See further, J. Chem. Soc., March, 1920.)—J. K.

Asphaltic sludge. Baskerville. See IIA.

PATENTS.

Iron oxide slimes. Ger. Pat. 305,083. See VII.

Antiseptic material. Ger. Pat. 315,017. See XIXb.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; Synthetic —. H. P. Stevens. Bull. Rubber Growers' Assoc., 1919, 1, [3], 55—56.

TWO samples of German synthetic rubber, one consisting of a colourless transparent slab and the other of an opaque reddish-brown crêpe, were found to be free from fatty oils and "substitutes." The materials, which yielded respectively 473% and 437% on extraction with acetone and refused to dissolve in benzene although they swelled considerably, were difficult to work into smooth sheet and vulcanised much more slowly than natural rubber.

—D. F. T.

Rubber from matured coagulum (slab); Uniformity of —. H. P. Stevens. Bull. Rubber Growers' Assoc., 1920, 2, [1], 68—71.

THE statement of Eaton and Grantham as to the remarkable uniformity of "slab" rubber is not justified. (See also de Vries, this J., 1918, 664 A.)

—D. F. T.

[*Rubber*] *latex coagulant; Synthetic acetic acid as a —*. H. P. Stevens. Bull. Rubber Growers' Assoc., 1920, 2, [1], 71.

A SAMPLE of synthetic acetic acid prepared from acetylene, when applied as a coagulant, gave results identical with those observed with acetic acid produced from pyroligneous acid.—D. F. T.

[*Rubber*;] *Effect of varying proportions of acid in coagulation on the rate of cure [of —]*. H. P. Stevens. Bull. Rubber Growers' Assoc., 1919, 1, [1], 39—41.

THE observation of other investigators to the effect that an increase in the proportion of acetic acid causes a decrease in the rate of vulcanisation of the resulting rubber, is confirmed. The effect is a little more marked with smoked sheet than with air-dried crêpe and is still more marked for crêpe prepared from matured "slab" rubber. (See also de Vries, this J., 1917, 1140; Campbell, this J., 1917, 604.) —D. F. T.

[*Rubber*;] *Effect of exposure to air on maturation [of —]*. H. P. Stevens. Bull. Rubber Growers' Assoc., 1919, 1, [2], 42—44.

EXPOSURE to air during maturation does not appear to have any very marked effect on the rate of vulcanisation of the finished rubber, but open maturing generally produces a slightly faster curing rubber than maturing in closed vessels.—D. F. T.

Metrolac [for determining rubber content of latex]. H. P. Stevens. Bull. Rubber Growers' Assoc., 1919, 1, [2], 44—45.

ALTHOUGH the Metrolac cannot be relied on to give accurate results for the rubber content of an individual sample of latex, it is nevertheless useful for standardising bulked latex and for estimating the yields of rubber on an estate. In its application to the latter purpose the errors over a period tend to eliminate themselves by compensation. (See also de Vries, this J., 1919, 590 A.)—D. F. T.

[*Rubber*;] *Reactions of accelerators during the vulcanisation [of —]*. C. W. Bedford and W. Scott. J. Ind. Eng. Chem., 1920, 12, 31—33.

THE most active accelerators of the vulcanisation of rubber are products of the reaction of carbon bisulphide with strong organic bases, such as the piperidine salt of piperidyl-dithiocarbamic acid. Thiocarbamilide is produced by a similar reaction. The stable metallic dithiocarbamates lose hydrogen sulphide when heated to the temperature used in the vulcanisation of rubber, and form thiourea derivatives, so that they may act in the same way as thiocarbamilide in the process. The main reaction of accelerators containing methylene groups (methylene-aniline, methylene-diphenyldiamine) appears to be to substitute thiocarbonyl groups for the methylene groups, and the resulting compounds may be regarded as thiocarbamilide derivatives. The accelerating action of hexamethylenetetramine may be attributed to the formation of a dithiocarbamate. Apparently all accelerators of this type containing methylene groups react readily with sulphur to form thiourea derivatives, but this does not apply to the methylene groups in such compounds as piperidine, or pentamethylenediamine, which when heated loses ammonia and forms piperidine.—C. A. M.

Rubber compounds, Expansion of — during vulcanisation. C. W. Sanderson. J. Ind. Eng. Chem., 1920, 12, 37—40.

AN apparatus for testing the amount of expansion of rubber when exposed to heat, consists essentially of a hollow steel cylinder 2 in. high and 0.7978 in. in diameter, which may be filled with the rubber, and which is surrounded by a steam jacket. When the rubber expands on heating, its top surface acts

against a piston and spring which transmit the motion through a magnifying lever to a recording pencil. Above the piston is fixed a spring to maintain sufficient pressure upon the rubber to prevent it from "blowing." The rubber is heated at a constant rate, usually 20° C. per 5 mins., and the recording cylinder moved forward at each interval. The results, when plotted in curves, afford a means of distinguishing between different batches of the same stock, which may have contained different rubber or have been subjected to varying conditions of milling, etc. The summarised results indicated that the higher the rubber content the greater the expansion, and that the harder the crude rubber the less the expansion. The expansion increases with the amount of milling, whilst there is no break in the expansion at the point of vulcanisation. The increase in the sp. gr. is due to the pressure, and not to physical change or internal contraction of volume.—C. A. M.

Rubber; Increase in the volume of compounded — under strain. H. F. Schippel. J. Ind. Eng. Chem., 1920, 12, 33—37.

WHEN rubber containing a pigment is stretched it will separate from the pigment particles, causing spaces to be formed on each side of the latter, and thus producing a considerable increase in the total volume of rubber. The effect of stretching upon rubber containing various percentages (by vol.) of barytes, whiting, zinc oxide, china clay, red oxide, lampblack, and carbon black was determined by stretching test rings of the rubber upon a graduated series of steel bars and calculating the increase in vol. from the change in the sp. gr. of the material. It was found that the greater the mean diameter of the pigment particles the greater was the increase in volume under strain. In certain cases the increases under strain were very pronounced. For example, a sample containing 100 grms. of fine Para rubber, 30 grms. of litharge, 5 grms. of sulphur, and 337 grms. of whiting, which had been cured for 30 mins. at 40 lb. steam pressure, showed a volume increase of 52% at an elongation of 140%. —C. A. M.

PATENT.

Rubber; Testing device for determining the "viscosity" of —. R. B. Naylor, Springfield, Mass., Assignor to The Fisk Rubber Co., Chicopee Falls, Mass. U.S. Pat. 1,327,838, 13.1.20. Appl., 5.2.19.

THE test piece of rubber is held by two grips which can be rotated independently about an axis perpendicular to the general plane of the gripping surfaces; arrangement is made for the application of known pressure and torsional force between the grips and for the indication of the resulting relative rotation.—D. F. T.

XV.—LEATHER; BONE; HORN; GLUE.

PATENTS.

Iron-tanned leather; Preparation of tough and durable —. K. W. Mensing, Freiberg, Ger. Pat. 314,885, 2.2.16. Addn. to Ger. Pat. 314,187.

IN the tanning process by use of iron salts, described in the chief patent (this J., 1920, 121 A), the removal of the excess of iron salts as well as the treatment with alkalis may be carried out before drying or oiling the leather. The iron-tanning process may be followed by treatment with solutions of vegetable or similar tanning agents. It may also be followed by treatment with reducing agents, if necessary after treatment with dilute acid solution. In this latter case there is partial re-

removal of iron salts from the outer layers, resulting in bleaching and allowing milder "grain" to be obtained.—B. V. S.

Tanning extract; Process of the production of — from waste sulphite lye. H. B. Landmark, Drammen, Norway. U.S. Pat. 1,327,105, 6.1.20. Appl., 23.4.15.

SEE Fr. Pat. 474,336 of 1914; this J., 1915, 1105.

Cattle food [from tannery offal]. Ger. Pat. 314,957. See XIXA.

XVI.—SOILS; FERTILISERS.

Soil acidity. II. Its relation to the acidity of the plant juice. E. Truog and M. R. Meacham. Soil Sci., 1919, 7, 469—474.

THERE is considerable variation in the acidity of the juices of different species of plants, but for each particular species the acidity is usually limited to a rather narrow range, there being, in all probability, a certain acidity which is most favourable for the life processes of that species. In many cases soil acidity, by limiting the supply of lime available for the plants, affects the acidity of the juice or protoplasm of the plants. In addition to the soil conditions, weather conditions, lapse of time after cutting the plant, and the time of the day at which the plant is cut affect the acidity of the plant juice. There is a slight difference in acidity between the juice of the tops and of the roots of plants.—W. G.

Soil acids; Activity of —. R. E. Stephenson. Soil Sci., 1919, 8, 41—59.

USING a modification of Tacke's method (this J., 1918, 776A), the author has endeavoured to arrive at the degree of activity of the soil acids, the lime requirements being determined for the given soil by measurements after 3, 6, 9, and in some cases 23 hours' contact between the soil and the calcium carbonate. The results show that soil acidity may be divided into two parts, viz., that produced by the more reactive acids, which are capable of giving a toxic hydrogen-ion concentration; and that which may be described as potential rather than active, this acidity being capable, however, of slowly decomposing carbonates. Protein substances and amino-acids, so far as tested, previous to decomposition in the soil, do not react readily with carbonates. It is necessary to know something of the activity of the soil acids, as well as of the total potential acidity, commonly determined more or less inaccurately. It is not the capacity of a soil to decompose lime, but rather the intensity of decomposition which is most highly significant. (See further J. Chem. Soc., Mar., 1920.)—W. G.

Acid soils and the toxicity of manganese. M. J. Funchess. Soil Sci., 1919, 8, 69.

THE toxicity of certain plots on the Alabama Agricultural Experiment Station farm was ascribed to the presence of soluble manganese. From further studies of these soils the author now considers that that conclusion is untenable.—W. G.

Soya beans; Effect of inoculation and lime on the yield and on the amount of nitrogen in — on acid soil. E. B. Fred and E. J. Graul. Soil Sci., 1919, 7, 455—467.

INOCULATION of soya beans grown on an acid sand increased the yield of dry matter of the tops, the yield being further increased when inoculation was supplemented by an application of lime. An application of lime just sufficient to neutralise half the active soil acidity gave the maximum yield.

Similarly the nitrogen content of the crops was considerably increased by inoculation, due to greater fixation of atmospheric nitrogen.—W. G.

Vegetative growth in soils containing crude petroleum. R. H. Carr. Soil Sci., 1919, 8, 67—68.

THE growth of soya beans was apparently improved by the addition of small amounts of oil, up to 13,400 lb. per acre, but further additions beyond this point caused a diminution in the growth until with about 72,000 lb. of oil per acre the plant practically succumbed to the treatment. The damage seems to be due in part to the plant's inability to secure water rapidly enough to meet its needs.—W. G.

Plant growth; Effect of nitrogen-fixing organisms and nucleic acid derivatives on —. W. B. Bottomley. Proc. Roy. Soc., 1920, 91B, 83—95.

PREVIOUS work (this J., 1917, 728) having shown that a water extract of bacterised peat had remarkable stimulative properties on the growth of *Lemna minor* in water cultures, further experiments were made with the individual constituents of the water extract. A pure growth of *Azotobacter chroococcum* was sterilised in the autoclave in presence of water and added in small known amounts to *Lemna minor* plants in Detmer's nutritive solution. In six weeks there was recorded an increase in rate of growth and in dry weight of the crop over untreated plants. A similar result was obtained with *B. radiclecola*. An extract of the crude nucleic acid derivatives and of the adenine-uracil fraction from raw peat (see this J., 1917, 1143) stimulated growth also. When the extract of crude nucleic acid derivatives was added along with the growth of *Azotobacter chroococcum* to the same plants, the growth produced exceeded that of the total of the effects of the two constituents taken separately, showing that the stimulating constituents in the extract of peat differed from those in *Azotobacter chroococcum*. The ash of the nucleic acid derivatives and the ash of *Azotobacter chroococcum* had no stimulative effect.—J. H. J.

Nitrolim; Attempts to obtain — free from dust. N. Caro. Chem.-Zeit., 1920, 44, 53—56.

MUCH attention has been devoted towards obtaining nitrolim free from dust and without its caustic qualities, these drawbacks being due to the presence of unconverted carbide and free lime. Addition of water as a binding agent is unsatisfactory; although the carbide certainly undergoes decomposition, yet the lime, being in a "dead" condition, remains practically unchanged, and further the water itself causes decomposition of the cyanamide. Most of the remedies suggested, such as addition of colloids, or chloride of calcium or magnesium, are unsuitable inasmuch as they require water as a vehicle. If the amount of water added is much reduced, decomposition will not take place, but the binding qualities will not be permanent. Addition of the water in the form of crystalline salts led to no good results, nor did mechanical treatment of the mass by sieving or pressing after mixing with water prove effective. As a binding agent, paraffin oil gives most favourable results; it is essential that phenols should be absent, as these deleteriously affect the fertilising power of the nitrolim. To neutralise the caustic action of the product, sugar or sugar-containing solutions derived from molasses or cellulose manufacture may be added. The sugar combines with the lime to form saccharates, and fulfils a three-fold purpose inasmuch as it also acts as a binding agent for the dust, and aids the action of the fertiliser by development of bacteria. The water which is introduced with the sugar solution is to a large extent retained by the colloidal matter

present and its effect on the nitrolim is very slight. The admixture of nitrolim with other fertilisers is not industrially practicable as the mixtures are either too low in nitrogen content or are unstable.—W. J. W.

Seeds; Report on the proposed electrolytic treatment of — [Wolfryn process] before sowing. E. J. Russell. J. Ministry Agric., 1920, 26, 971—981.

For experiments were made with oat and barley seeds which had been treated by the Wolfryn electrolytic process (this J., 1914, 1067) and with similar untreated seeds. The weights of the crops produced varied very much, there being sometimes a gain and sometimes a loss resulting from the electrolytic treatment. In further similar trials, the majority of the results showed a loss or no improvement in the crops from the treated seeds. In field experiments also negative results were obtained, except in a few cases where seed was sown after roots had been fed off. It is concluded that the process is an uncertain one, which may or may not succeed in any given case.—J. H. J.

Plant juices. Haas. See XIX A.

Oxidation of vanillin by soil bacterin. Robbins and Lathrop. See XX.

PATENTS.

Fertiliser and method of producing the same. P. Radmann, Stockholm. Eng. Pat. 137,171, 5.3.19. (Appl. 5477/19.)

A MIXTURE of felspar, gypsum, and limestone is heated to 1000°—1250° C. (see Eng. Pat. 12,136 of 1914; this J., 1915, 138), and the product is disintegrated in a jaw crusher, ground in a ball mill, and used directly as a fertiliser. Up to 60% of calcium phosphate in the form of apatite or other mineral phosphate may be added to the mixture before heating.—J. H. J.

[Calcium] cyanamide containing free lime; *Process of granulating —.* V. Thrane, Christiania, Norway. U.S. Pat. 1,326,310, 30.12.19. Appl., 16.1.17.

CALCIUM cyanamide containing free lime is mixed with water and fed on to a moving surface, where it is pressed to accelerate the reaction between the lime and water. It is pressed again and dried, still on the moving surface, and is then broken up into granules.—J. H. J.

Dicalcium phosphate; Process of making — and fertiliser material produced therein. S. S. Sadtler, Springfield township, Pa. U.S. Pat. 1,326,533, 30.12.19. Appl., 10.3.19.

COMMUNUTED phosphate rock is subjected to the action of sulphur dioxide and steam at 100° C. The product contains dicalcium phosphate, monocalcium phosphate, and calcium sulphite, and is free from acid.—J. H. J.

Fertiliser; Process for the preparation of a stable, mixed — which can be easily distributed. F. Dahl, Hamborn-Bruckhausen. Ger. Pat. 314,404, 20.6.17.

AFTER neutralisation of the free lime in a mixture of an ammonium salt solution and ground basic slag, a sodium or potassium salt, or a mixture of both, is added immediately, with constant stirring. —A. R. P.

XVII.—SUGARS; STARCHES; GUMS.

Sugar; Balance of nitrogen during the manufacture of —. Precipitation of the proteid matter of the beetroot by sulphurous acid, bisulphites, and hydrosulphites. E. Saillard. Comptes rend., 1920, 170, 129—130.

IN sugar factories which distil their molasses and burn their vinasses the nitrogen of the beetroot is distributed in the following proportions: In the carbonatation cake, 15%; in the fodder pulp, 20%; in the press waters, 18%; liberated as ammonium during the processes, 17%; liberated during the incineration of the vinasses, 30%. Sulphurous acid and its compounds precipitate from sound beet-roots the same polarising substances as does basic lead acetate. They also precipitate as much proteid material from the juice as do copper hydroxide and aluminium sulphate when used together.—W. G.

Sucrose; Cryoscopic method for the determination of —. H. H. Dixon and T. G. Mason. Scient. Proc. Roy. Dublin Soc., 1920, 16, No. 1, 1—8.

A METHOD for the determination of sucrose in small volumes of liquid is based on the fact that after inversion the depression of freezing point of a sucrose solution is approximately doubled. Invertase is added to the sap or other liquid in the cold and the freezing point determined, after which the mixture is incubated at 30° C. for 48 hours and a second observation made. A differential thermoelectric device (Scient. Proc. Roy. Dublin Soc., 1911, 13, No. 4, 49) is used for ascertaining the freezing point, and the galvanometer is placed in a thermostat at 21° C. Advantages of the proposed method are that no treatment to eliminate proteins and other colloids is necessary, and that only a small volume of liquid, viz., about 2.5 c.c., is required. Readings of temperature may be obtained with an error of $\pm 0.003^\circ$, and 0.01° corresponds to 0.2% of sucrose.—J. P. O.

Lactose. J. Gillis. Rec. Trav. Chim. Pays-Bas, 1920, 39, 88—125.

A MORE detailed account of work already published. (See this J., 1918, 133 A.)—W. G.

Sugar in presence of protein degradation products. Last. See XIX A.

Inversion of sucrose in oranges. André. See XIX A.

Gluconic acid. Herzfeld and Lenart. See XX.

Fehling's solution. Bolin and Linder. See XXIII.

PATENTS.

Sugar-making apparatus; Process of cleaning —. E. H. Man, New Orleans, La. U.S. Pat. 1,326,280, 30.12.19. Appl., 23.11.17.

SUGAR-MAKING apparatus is subjected to the action of aqueous sulphurous acid to soften the scale, which is afterwards removed.—J. H. J.

Vegetable carbons [used in sugar refining]; Process of regenerating —. A. Adams and S. S. Peck, Honolulu, Hawaii. U.S. Pat. 1,326,159, 30.12.19. Appl., 16.7.17.

To regenerate vegetable decolorising carbon used for purifying saccharine solutions, the material is fermented, then heated with sulphuric acid and/or a salt to remove gummy material and subsequently washed.—W. F. F.

Lactose or milk sugar; Manufacture of —. G. Martin, Manchester. Eng. Pats. 135,614 (Appls. 19,692, 28.11 and 21,833, 30.12.18) and 135,969, 13.12.18 (Appl. 20,836/18).

(A) WHEY is concentrated *in vacuo* to about one-

fifth of its original volume and heated at 70°–100° C. to precipitate as much as possible of the proteins, and metaphosphoric acid is added to precipitate the remainder. After separation from the precipitate the whey is concentrated as usual. (b) One or more of the following protein precipitants may be used: Tannic acid, mercurous nitrate, phosphotungstic acid, magnesium sulphate.

—J. H. L.

Starch; Method of preparing — [for textile fibres or fabrics]. *Starch preparing apparatus*. L. W. Goold, Birmingham. From American Laundry Machinery Co., Cincinnati, Ohio, U.S.A. Eng. Pats. (a) 135,591 and (b) 135,588, 27.11.18. (Appls. 19,530 and 19,520/18.)

(A) A MIXTURE of starch and water, after being cooked and agitated in a suitable vessel by injection of steam, is "creamed" by circulating between the vessel and an external apparatus, being, for example, withdrawn from the bottom of the vessel and re-introduced into the upper part in the form of fine jets. This method of circulation may also be employed for cooling and maintaining the liquid at the right temperature for use. If necessary uncooked starch may be introduced into the circulating liquid when this is sufficiently cooled. (B) Apparatus for carrying out the above process comprise a vessel, the bottom of which is provided with steam injectors, and an external circuit comprising a pipe connecting the bottom of the vessel through a pump with the lower ends of a bundle of vertical tubes, the upper ends of which are connected with a return pipe terminating in a spraying device inside the top of the vessel. The tubular bundle is enclosed in a casing through which cooling water or, if necessary, steam may be passed.—J. H. L.

Adhesives [from starch]; Process for the production of — V. G. Bloede, Catonsville, Md. U.S. Pat. 1,324,332, 9.12.19. Appl., 19.6.18.

A MODIFIED starch, or starch base, is produced by heating raw starch between 300° and 400° F. (149° and 204° C.) for $\frac{1}{2}$ –1 hour, according to the purpose for which the product is required, the temperature and duration of heating being so regulated that the product remains practically insoluble in cold water.—J. H. L.

Sugar-cane; Process for macerating crushed — C. McNeil, Glasgow. U.S. Pat. 1,323,090, 13.1.20. Appl., 22.4.18.

SEE Eng. Pat. 116,521 of 1917; this J., 1918, 480 A.

Sugar solutions; Process for defecation of — E. C. R. Marks, London. From Industrial Apparatus Corporation, New York. Eng. Pat. 137,750, 23.6.19. (Appl. 15,766/19.)

SEE U.S. Pat. 1,317,607 of 1919; this J., 1920, 76 A.

Saccharification of wood etc. Ger. Pats. 305,180, 309,150, and 310,149–50. See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Malt analysis; Application of formol titration to — H. Langkammer and H. Leberle. Z. ges. Brauw., 1919, 42, 236–239, 247–252, 259–262, 271–273, 280–282.

SÖRENSEN'S method of determining amino-acid and polypeptide nitrogen by formol titration (see this J., 1908, 135) was applied to 31 malts, and the authors conclude that it may prove useful in comparing the brewing characters of different malts. The "total formol nitrogen," determined on extracts made by digesting the ground malt with water for 4–5 hours at 46°–48° C. and filtering

(cp. Lüers and Adler, this J., 1915, 1158), ranged in most cases from 170 to 270 mgrms. per 100 grms. of malt dry substance; very similar results were obtained in operating on laboratory worts prepared in the ordinary way. The "original formol nitrogen" in the malt was determined on extracts made after destroying the malt enzymes by means of alcohol (*loc. cit.*), and ranged in most cases from 90 to 150 mgrms. per 100 grms. of malt dry substance. Abnormally small amounts of "formol nitrogen" are found with very short-grown or highly cured malts, whilst high results are obtained with malts prepared by the Kropff and similar systems. Dark malts usually contain less "formol nitrogen" than pale ones owing to destruction of amino-acids by Maillard's reaction (cp. this J., 1912, 144), but if dark malts are very highly modified they may still contain a relatively high proportion of "formol nitrogen." In general, in a series of malts, the content of "formol nitrogen" increases with other characters depending on enzymic activity, viz., rate of saccharification, acidity (determined by titration), and yield of extract. Worts rich or poor in "formol nitrogen" yield beers possessing corresponding characters. The amount present in a wort does not influence the reproductive or fermentative activity of the yeast, provided it is not too low; but a very high proportion in wort may impair the stability of the beer by hindering the production of the most favourable hydron concentration in the course of fermentation. (See also Adler; this J., 1914, 367.)

—J. H. L.

Yeast; Enzymic activities of — T. Bokorny. Allg. Brau-u. Hopfenzeit., 1918, 58, 1093. J. Inst. Brew., 1919, 25, 314–315. (See also this J., 1916, 900; 1917, 608; 1919, 958 A.)

THE zymase of yeast is rendered inactive by 0.2% formaldehyde solutions, and within two days by 0.1%, but not by 0.05% solutions; the invertase, however, survives two days' contact with 1% solutions. Mercuric chloride of 0.1% solution likewise prevents fermentation without inhibiting invertase action. By treatment with phenylhydrazine a yeast can be obtained which will ferment dextrose but not maltose. Alcohol of 10–20% concentration almost or entirely prevents fermentation without permanently destroying the zymase. Absolute alcohol destroys zymase activity within 10 minutes, but does not render the invertase inactive within 20 days.—J. H. L.

Yeast; Sensitiveness of living — towards hydra-gen and hydroxyl ions. H. von Euler and F. Emberg. Z. für Biologie, 1919, 69, 349. Woch. Brau., 1919, 36, 166.

THE authors investigated the influence of various hydron concentrations on the activities of the zymase, invertase, and maltase of a bottom-fermentation beer yeast. The zymase showed more or less activity over the range $p_H=9-2$. With increase in the hydron concentration from $p_H=9$ the zymase activity increased very rapidly to a maximum value which remained unchanged over a considerable range, but diminished gradually to zero as the acid limit, $p_H=2$, was approached. The invertase in the living yeast was affected by different hydron concentrations in the same way as invertase in the free state. The maltase was rendered quite inactive by a very faint alkalinity, $p_H=8$; as this reaction still permits the zymase and invertase to act, it should be possible, by maintaining such a reaction, to ferment only the sucrose in mixtures of this sugar with maltose. The authors also studied the effect of prolonged contact of the yeast with liquids of various hydron concentrations between $p_H=3.5$ and 7.2, and found that such treatment influenced

the enzymic activities (except that of invertase) and the synthetic processes associated with growth and reproduction.—J. H. L.

Yeast; Adaptability of — to different temperatures. H. Zikes. *Allg. Z. Bierbrau. u. Malzfabr.*, 1918, 46, 359. *J. Inst. Brew.*, 1919, 25, 315.

Six varieties of bottom-fermentation yeast, after cultivation for 25 days at 8° C., were found to grow more rapidly at temperatures below 20° C., and more slowly at higher temperatures, than the same varieties after cultivation for 25 days at 25° C. Further, the yeasts cultivated at 8° C. showed a better adaptability to high temperatures than those cultivated at 25° C. did to low temperatures.

—J. H. L.

Fermentation; Loss of alcohol during —. P. Harder. *Woch. Brau.*, 1919, 36, 277–279. *J. Inst. Brew.*, 1920, 26, 55–56.

THE relation between original wort gravity, residual extract, and alcohol-content of beers, as given by Balling's original formula (see Schönfeld, this J., 1910, 292; 1911, 762) is not strictly accurate. With bottom-fermentation beer the author found, after fermentation, an alcohol-content 0.03–0.04% lower than that required by the formula, irrespective of whether the fermentation was carried out in a covered vat or in an open vat exposed to a continuous strong current of air. The discrepancy can therefore not be attributed to loss of alcohol by evaporation; and an actual determination of the alcohol entrained by the fermentation gases, in the case of a covered vat, showed a loss of only about 0.001% of alcohol by evaporation. Both found much greater losses, e.g., 1.5%, in the fermentation of distillery mash, doubtless owing to the higher temperatures employed.—J. H. L.

Beers; Use of the Zeiss immersion refractometer for thin —, and criticism of the determination of original gravity in general. Dietsche and Grave. *Woch. Brau.*, 1919, 36, 237–240. *J. Inst. Brew.*, 1920, 26, 54–55.

THE alcohol and extract-contents of thin beers, of 1–3% Balling, may be calculated either from Barth's formulae (see Ackermann and Toggenburg, this J., 1906, 286) or from simpler formulae proposed by Lehmann and Gerum. According to the latter authors the extract-content, in grms. per 100 c.c., is given by the expression $0.9(R_0 + L)/7$, and the alcohol-content, in grms. per 100 c.c., by $2(R_0 - L)/7$, where R_0 is the scale reading of the Zeiss immersion refractometer less 15 (the reading for water), and L is the sp. gr. at 15° C. referred to water = 1000 and diminished by 1000.—J. H. L.

Acetyl methylcarbinol; Specific reaction of 2,3-buteneglycol and of — products of butyleneglycollic fermentation. Lemoigne. *Comptes rend.*, 1920, 170, 131–132.

PROOF of the formation of acetyl methylcarbinol during the bacterial decomposition of sugars serves to differentiate certain groups of similar organisms. The carbinol may readily be detected amongst the products of fermentation by the following process, the reaction not being given by any of the other products. A little of the culture (10, 50, or 100 c.c.) is mixed with 5 c.c. of ferric chloride solution and distilled. The carbinol is thus oxidised to diacetyl which passes over. To the first 3–4 c.c. of the distillate, 15–20 drops of ammonia, 5 drops of a 20% solution of hydroxylamine hydrochloride, and 5 drops of a 10% solution of nickel chloride are added. After a time (the length depending on the amount of diacetyl present) a red crystalline precipitate of nickel

dimethylglyoxime is obtained. By this method acetyl methylcarbinol may be characterised at a dilution of 1 in 1,000,000.—W. G.

Urease and the theory of the action of enzymes by radiation. H. P. Barendrecht. *Rec. Trav. Chim. Pays-Bas*, 1920, 39, 2–87.

AFTER a criticism of previous work, the author deduces a formula showing the velocity of action of urease at constant temperature and hydrogen ion-concentration, based on the theory of enzyme action by radiation. An experimental verification is given of the general law from the action of urease. The conditions governing the action of urease are examined, including the influence of foreign substances. The reversibility of the hydrolytic action with high p_H is shown. The influence of the concentration of the urease on the direct synthesis of urea is examined. A simple apparatus is described suitable for the measurement of the concentration of H ions or OH ions at constant temperature, with a hydrogen electrode or an oxygen electrode respectively.—W. G.

Alcohol and water; Determination of the composition of mixtures of — by electrical conductivity measurements. I. M. Kolthoff. *Rec. Trav. Chim. Pays-Bas*, 1920, 39, 126–131.

THE following procedure is recommended for the determination of the alcohol content of beer or wine. The excess of carbon dioxide is removed from the liquid by bubbling air through it, and the liquid is then distilled with magnesia. Fifty c.c. of the distillate is mixed with 10 c.c. of approx. N/2 oxalic acid solution and the mixture is made up to 100 c.c. with water. Similarly 10 c.c. of the N/2 oxalic acid solution is diluted to 100 c.c. with water. The specific conductivities x_1 and x_2 of these two latter solutions are then determined. Then $f_1 = 100x_1/x_2$ and f_1 can be converted to f_{10} by means of a temperature coefficient. A table is given showing the relationship between f_{10} and the alcohol content of the distillate.—W. G.

PATENTS.

Beer; Process for preserving non-alcoholic — and other fermentable liquids. H. C. M. Franks, New York. Eng. Pat. 136,448, 25.4.19. (Appl. 10,315/19.)

THE liquid, maintained at about 35° C. in a pressure tank, is subjected to carbon dioxide under a pressure of about 60 lb. per sq. in., and agitated meanwhile, after which the pressure is reduced below 1 atm. These operations are repeated and finally the liquid is left for a time under a 60 lb. pressure of carbon dioxide. It may then be transferred to sterile receptacles and sealed. The process is stated to prevent or arrest all fermentative or bacterial action.—J. H. L.

Saccharification of wood and other cellulosic substances by treatment with sulphuric acid, and recovery of the latter. Zellstoff-fabrik Waldhof, and H. Clenm, Mannheim-Waldhof. Ger. Pat. 305,180, 12.5.17.

THE cellulosic material is made into a paste with sulphuric acid, which is then partly neutralised by ammonia, leaving only enough acid for the sugar formation. After saccharification the acid is completely neutralised and the liquor boiled down and cooled for separation of the ammonium sulphate. The ammonium sulphate left in the mother-liquor is of value as yeast food in the subsequent fermentation process. The spent wash from the fermentation process may be evaporated to obtain a fertiliser.—B. V. S.

Sugar and dextrin from wood and other cellulosic material; Process for obtaining — Zellstoff-fabrik Waldhof, and V. Hottenroth, Mannheim-Waldhof. Ger. Pats., (A) 309,150, 19.4.17; (B) 310,149, 7.9.17; (C) 310,150, 9.2.18.

(A) THE wood or other material is mixed into a paste with less than 3 parts of sulphuric acid not more concentrated than 85%, high pressures being avoided. This is allowed to stand for some time, then mixed with water, and boiled. The actual quantity of sulphuric acid depends on the quality of the material to be treated, but is generally from $\frac{1}{2}$ to 1 part of acid to 1 part of the material; the cellulose should not be dissolved. For example, 1 kilo. of sawdust requires 1 litre of 75% acid; after some hours' standing 14 litres of water is added. Lignin remains undissolved and the solution contains dextrin, which is converted into sugar by boiling. (B) The excess of sulphuric acid above that required for conversion of the dextrin into sugar, or the whole of the acid if the dextrin is required as such, is removed by dialysis. (C) By using a copper ferrocyanide dialysing membrane the acid remaining with the sugar, after conversion of the dextrin by boiling the solution, is removed, since sugar does not pass through such a membrane. —B. V. S.

Products from reeds. Ger. Pat. 304,285. See V.

XIXA.—FOODS.

Milk; Decomposition of hydrogen peroxide by micro-organisms extracted from pasteurised — M. Fouassier. Comptes rend., 1920, 170, 145—147.

IN addition to the lactic ferments certain organisms such as *B. subtilis*, *Tyrophix tenuis*, *Oidium lactis*, and a yeast have been isolated from pasteurised milk. All of these, except the lactic ferments, decompose hydrogen peroxide at a concentration of 1% in a lactose-peptone medium, but only the two first named can resist the action of hydrogen peroxide at a concentration of 4%. This decomposition of the hydrogen peroxide continues to be manifested after filtration of the culture medium through a porous candle, but is stopped if the medium is heated to 80° C.—W. G.

Milk; Detection of hydrogen peroxide in pasteurised — by means of guaiacum tincture. M. Fouassier. Ann. Chim. Analyt., 1920, 2, 9—11.

THE peroxydase necessary for the detection of hydrogen peroxide in pasteurised milk by the guaiacum test may be obtained from potatoes. A peeled potato is macerated with water, and the milk to be tested is treated with about 10% of its volume of the maceration.—W. P. S.

Gluten; Determination of dry — [in flour]. J. Bouyer. Bull. Soc. Pharm. Bordeaux, 1919, No. 2. Ann. Chim. Analyt., 1920, 2, 20—21.

THE gluten obtained in the usual way is pressed by the fingers into a cake, which is placed on a cover glass and dried at 105° C. for 30—45 mins.; the cake is then turned over, dried for a further 30—45 mins., and cut into strips by means of a pair of scissors. The strips are dried for 30 mins., then cut into small pieces and the drying continued to constant weight. This final drying requires about 6 hours.—W. P. S.

Sugar; Quantitative estimation of small quantities of — in the presence of the higher and lower products of protein degradation. E. Last. Biochem.-Zeits., 1919, 93, 66—82.

THE higher products of protein degradation like

albumoses and peptones, the presence of which interferes with the estimation of sugar, can be removed by means of mercuric chloride in neutral solution. The sugar can then be estimated by Bertrand's methods (this J., 1907, 60). In the presence of acids the precipitation of these products is incomplete. An excess of mercuric chloride must also be avoided in order to ensure good results; 2 grms. of mercuric chloride per gm. of peptone was found to be a suitable quantity. The presence of monoamino acids does not affect the accuracy of the sugar estimation by Bertrand's method, but ereptone does influence the results. This is due to the special atomic grouping in ereptone, which on boiling with alkali liberates ammonia, and this dissolves some of the cuprous oxide. Ereptone can also be removed from sugar solutions by precipitation with mercuric chloride in alkaline solution. The removal of both the higher and lower products of protein degradation with mercuric nitrate according to the Patein-Dufau method conduces to accurate results. —S. S. Z.

Plant juices; Electrometric titration of — A. R. C. Haas. Soil Sci., 1917, 7, 487—491.

A SIMPLIFIED and inexpensive type of the gas-chain apparatus, essentially of Hildebrand's form (Univ. Cal. Pub. Physiol., 1919, 5, 44), is described, and by means of it the p_H values of the juice of soya bean tops and rhubarb stalk were determined. The curves show the presence of greater quantities of buffer substances in rhubarb juice than in the juice of soya bean tops, the actual acidity of the rhubarb juice being much greater than that of the juice of soya bean tops.—W. G.

Sucrose; Inversion of — during the preservation of oranges. G. André. Comptes rend., 1920, 170, 126—128.

THE amount of sucrose inverted during four months when half of an orange is kept under aseptic conditions at the ordinary temperature bears no relationship to the acidity of the orange expressed as citric acid.—W. G.

PATENTS.

Casein and vegetable albumen, including gluten; Process for the manufacture of a colloidal solution, neutral to the taste, from — M. Monhaupt, Altona-Ottensen, Germany, Assignor to Naaml. Vennoots. A. Jurgens Vereenigde Fabrieken, Oss, Netherlands. U.S. Pat. 1,326,210, 30.12.19. Appl., 25.3.18.

CASEIN is treated with excess of magnesia, the excess not exceeding 1.5% by weight of the casein. —J. H. J.

Fat for shortening purposes; Comminuted edible — and method of making the same. H. A. Kohman, T. M. Godfrey, and L. H. Asche, Pittsburgh, Pa., Assignors to Ward Baking Co., New York, N.Y. U.S. Pat. 1,326,276, 30.12.19. Appl., 9.3.16.

FAT at a temperature above its melting point is sprayed into a cooling chamber, the spraying being regulated to cause the deposition of the fat in a sufficiently fine state of subdivision to permit of homogeneous incorporation with the material to be shortened.—J. H. J.

Cattle-food; Preparation of a — [from tannery offal]. E. Meyer und Co., Guben. Ger. Pat. 314,957, 9.11.15.

TANNERY offal is treated by known processes, e.g., mincing, cleaning, and cooking or treating with preservatives.—A. R. P.

Drying vegetables and the like; Apparatus for —. D. Brons, Assignor to N. V. Machinefabriek Brons, Zuidbroek, Netherlands. U.S. Pat. 1,326,909, 6.1.20. Appl., 28.5.19.

SEE Eng. Pat. 128,199 of 1919; this J., 1919, 875A.

Products from reeds. Ger. Pat. 304,285. See V.

Fibrous material etc. from pea-pods. Ger. Pat. 307,626. See V.

Lactose. Eng. Pats. 135,611 and 135,969. See XVII.

XIXB.—WATER PURIFICATION; SANITATION.

Industrial poisoning; Recent experiences on —. Koelsch. Z. angew. Chem., 1920, 33, 1—5.

A DISCUSSION of the general conditions resulting in poisoning is followed by an account of the symptoms and diagnosis of poisoning by alcohol, lead, mercury with its fulminate and chloride, arsenic and hydrogen arsenide, oxides of manganese, mineral acids, nitrous fumes, carbon tetrachloride, tetrachloroethane, trichloroethylene, amyl acetate, hydrocyanic acid, cyanamide, benzene and its nitro-derivatives, trinitrotoluene, tetranitromethane, trinitroanisole, and amino-derivatives of benzene.—J. K.

Humus; Chemical properties of — and their utilisation for the protection of combatants against asphyxiating gases. Griffon du Bellay and Houdard. Comptes rend., 1920, 170, 236—238.

SOIL absorbs gases such as benzyl bromide, chlorine, or carbonyl chloride, the retentive power varying with the type of soil, being almost nil for a very sandy soil and increasing with the amount of plant debris present. The phenomenon is chemical and exothermic. The fixation of the gas is facilitated by moisture. Humus dried at 100° C. has at the most only two-thirds of its original power of absorption. The results indicate that a layer of soil 60 cm. thick, well supplied with organic matter, could protect combatants for several hours against the war gases in use in February, 1916, provided the amount of gas passing through the soil did not exceed 1 litre per sq. decm.—W. G.

PATENTS.

Antiseptic material; Process for production of —. Verein für Chemische Industrie in Mainz, Frankfurt. Ger. Pat. 315,017, 21.4.16.

AN antiseptic material, particularly adapted to the formation of a stable, impermeable, sterile covering for various parts of the human body, such as the hand or arm, is obtained from such formaldehyde-phenol condensation products as are pliable and nearly liquid at body temperature. A solution of these in a suitable solvent such as alcohol is mixed with a solution of cellulose esters, to which may be added other antiseptics, colouring matters, or substances to increase the pliability.—B. V. S.

Iron oxide slimes. Ger. Pat. 305,083. See VII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Toxicity in sera and physical properties of colloidal gels. W. Kopaczewski and Z. Gruzewska. Comptes rend., 1920, 170, 133—135.

ANIMAL serum in contact with colloidal gels, such as gelose and pectin, acquires astonishing toxic properties, whereas other gels, of an apparently

identical consistency and structure, do not provoke this toxicity. It is now shown that the development of toxicity is dependent on the electrophoretic character of the gels. In the case of three silica gels prepared in different ways, electro-negative silica gel provoked marked toxicity in guinea-pig serum, whereas an electropositive gel or an amphoteric gel was without effect. From a study of the effect of starch gels, the importance of the micellar structure of the gels in the production of toxicity in sera by contact is shown.

—W. G.

Surface active substances; Adsorption of the so-called — by various adsorbents. L. Michaelis and P. Rona. Kolloid-Zeits., 1919, 25, 225—229.

CHARCOAL is by far the best adsorbent for non-electrolytes such as acetone, tributyrin, and heptyl alcohol. Of other adsorbents examined only in the case of talc was a measurable adsorption observed.

—J. F. S.

Aromatic diamines; Physiological experiments with —. R. Meissner. Biochem.-Zeits., 1919, 93, 149—163.

THE toxicity of some aromatic diamines, triamino-benzene, triaminotoluene, and triaminophenol has been tested on rabbits, cats, and frogs. (See J. Chem. Soc., Mar., 1920.)—S. S. Z.

Vanillin; Oxidation of — to vanillic acid by certain soil bacteria. W. J. Robbins and E. C. Lathrop. Soil Sci., 1919, 7, 475—485.

IN mineral nutrient solution containing vanillin as the only source of carbon, certain bacteria present in some Alabama soils (see Robbins, Alabama Agr. Exp. Stat., 1917, Bull., 195) oxidise the vanillin to vanillic acid, which at first accumulates in the culture solution but later is destroyed. Estes' acid mercuric nitrate reagent (this J., 1917, 352) gives satisfactory results for the estimation of vanillin in the presence of vanillic acid, but the phenol reagent of Folin and Denis (this J., 1912, 949) is unsatisfactory as vanillic acid, like vanillin, gives a blue colour with the reagent, the colour produced by the acid being much deeper than that produced by an equivalent amount of the aldehyde.

—W. G.

Gluconic acid; Preparation of — [from starch glucose]. A. Herzfeld and G. Lenart. Z. Ver. deut. Zuckerind., 1919, 122—128.

DURING the war a technical method for the preparation of gluconic acid to serve as a substitute for tartaric and citric acids was devised in Germany. Dextrose (starch glucose) was oxidised with chlorine or bromine, the excess of halogen was distilled off, and the liquid neutralised with sodium carbonate. Calcium carbonate was added, the temperature being raised during this operation to 90° C. to transform any lactone present, the liquid cooled, and the calcium gluconate obtained purified by recrystallisation. An almost quantitative yield is said to have been obtained. (See also J. Chem. Soc., Mar., 1920.)—J. P. O.

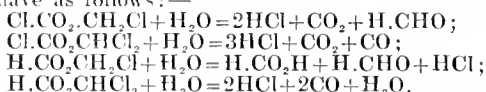
Urea; Behaviour of — towards hydrogen peroxide and a simple process for its purification and decolorisation. H. Kuntz-Krause. Kolloid-Zeits., 1919, 25, 240—241.

UREA is not affected by boiling with hydrogen peroxide but the colour (blue or green) which is often found in commercial urea and which consists of colloidal Prussian blue is decomposed, ferric hydroxide being precipitated.—J. F. S.

Chlorinated methyl formates; Industrial — and their analyses. M. Delépine. Bull. Soc. Chim., 1920, 27, 39—45.

WHEN methyl formate is chlorinated in sunlight

a number of chloromethyl formates and chloromethyl chloroformates may result. These esters when decomposed by dilute sodium hydroxide behave as follows:—



From the quantities of formaldehyde, formic acid, and carbon monoxide formed, together with a knowledge of the chlorine content of the original material, it is possible to form an idea of the constituents of the chlorinated material: 0.4 c.c. of the mixed chlorinated esters is added to 50 c.c. of *N*/1 sodium hydroxide solution and shaken. After half an hour the mixture is diluted to 125 c.c. Of this solution 25 c.c. is used for the estimation of formaldehyde by Romijn's method with *N*/10 iodine solution (this *J.*, 1897, 366), and another 25 c.c. is used for the estimation of formic acid by oxidation with potassium permanganate by St. Gilles' method (*Ann. Chim. Phys.*, 1859, 55, 374). For the estimation of carbon monoxide 0.3–0.5 grm. of the chlorinated material is introduced into a ureometer, treated with 10 c.c. of 4*N* sodium hydroxide, and the volume of carbon monoxide is read off as soon as the decomposition is complete.

—W. G.

Chlorinated methyl carbonates; Properties of the —. A. Kling, D. Florantin, and E. Jacob. *Comptes rend.*, 1920, 170, 231–236.

The physical properties of the nine possible chlorinated methyl carbonates are given as below:—

Formula	m.pt.	b.pt. at 760 mm.	b.pt. at 50 mm.	sp. gr. at 15°C.
$\text{CH}_3\text{Cl.O.CO}_2\text{CH}_3$	—	139–140°C.	67°	1.303
$\text{CO (O.CH}_2\text{Cl)}_2$	—	176°C.	99°	1.480
$\text{CHCl}_2\text{O.CO}_2\text{CH}_3$	—	147°C.	72°	1.421
$\text{CHCl}_2\text{O.CO}_2\text{CH}_2\text{Cl}$	—	178°C.	102°	1.562
$\text{CCl}_3\text{O.CO}_2\text{CH}_3$	–70°C (viscous)	165°C.	86°	1.535
$\text{CO (O.CHCl}_2)_2$	–40°C	182–183°C.	105–106°	1.618
$\text{CCl}_3\text{O.CO}_2\text{CH}_2\text{Cl}$	–70°C	150°C.	110°	1.631
$\text{CCl}_3\text{O.CO}_2\text{CHCl}_2$	–30°C (viscous)	196°C. (decomp.)	115°	1.686
$\text{CO (O.CCl}_3)_2$	78°C	203°C. (decomp.)	124°	—

All of these substances are lachrymators, those compounds containing the group O.CCl_3 being particularly toxic.—W. G.

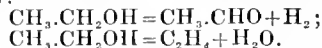
Acetone; Micro-method for the estimation of —. M. Richter-Quittner. *Biochem.-Zeits.*, 1919, 93, 163–172.

A MICRO-METHOD in which 1–2 c.c. of urine and 15–3 c.c. of alkali need only be used. The urine is distilled once with steam in the presence of acetic acid and a second time with dilute sulphuric acid. Blood or plasma need only be distilled once, and instead of the steam, air is passed through the heated flask. The titration of the distilled acetone is carried out with *N*/100 iodine and *N*/100 sodium thiosulphate. 0.1 mgrm. of acetone in 100 c.c. can be estimated with accuracy by this method. The quantity of urine and blood used must not contain less than 0.04 mgrm. of acetone.—S. S. Z.

Acetaldehyde, metalddehyde, and ethyl alcohol; Mode of pyrogenic decomposition, at high temperatures, of —. E. Peytral. *Bull. Soc. Chim.*, 1920, 27, 34–39.

WHEN acetaldehyde is submitted to a high temperature for a short time the main decomposition is expressed by $\text{CH}_3\text{CHO} = \text{CH}_4 + \text{CO}$, a second simultaneous change, which proceeds to a far less extent, being $2\text{CH}_3\text{CHO} = 2\text{CO} + \text{C}_2\text{H}_4 + 2\text{H}_2$. If the time of heating is prolonged secondary changes occur, with the formation of acetylene and benzene. Under similar conditions metalddehyde is first depolymerised to acetaldehyde, which then undergoes the

changes given above. In the case of ethyl alcohol there are two main changes, the first of which predominates:—



The acetaldehyde formed undergoes pyrogenic decomposition as described.—W. G.

Unsaturated substances; Action of iodine on —. *Pseudo-iodine value of essential oils.* R. Huerre. *J. Pharm. Chim.*, 1919, 20, 216–224, 250–257, 273–281.

IN the reaction between terpenes or terpenic essential oils and an alcoholic solution of iodine the absorption of iodine is independent of the concentration of the iodine solution, or of variations of temperature from 15° to 45° C., and is a function of the excess of iodine present. This absorption is not due, for the most part at all events, to addition of the iodine, and the name pseudo-iodine value is suggested for it. This value may therefore be defined as the maximum quantity of iodine absorbed in 2 hours by 100 grms. of hydrocarbon or essential oil from an alcoholic solution of iodine containing the optimum excess of iodine. In the case of pure cedrene the pseudo-iodine value (39.60) was much lower than that of pinene (238). The maximum absorption of iodine was obtained when a little more than 3 grms. of iodine was made to react with 0.25 grm. of cedrene. In the case of limonene the maximum absorption of iodine (309) was obtained when the amount of iodine present was 16 times that of the limonene.—C. A. M.

Terpene; Occurrence of the terpene — in the oil of *Eucalyptus megacarpa*. H. G. Smith. *J. Proc. Roy. Soc. N.S.W.*, 1918, 52, 529–533.

THE oil of *Eucalyptus megacarpa* contains pinene, limonene, and dipentene, about 30% of cineol, and possibly as much as 10% of terpinene. The latter was detected in the fraction boiling at 170°–190° C. by the formation of its nitrosite, m. pt. 155° C., and by its rapid oxidation in the cold with Beckmann's chromic acid mixture, as evidenced by the increased optical rotation of the residual oil (consisting mainly of limonene) after the removal of the inactive terpinene.—G. F. M.

Regenerating chromic acid. McKee and Leo. See VII.

Acetyl methylcarbinol. Lemoigne. See XVIII.

PATENTS.

Acetic acid; Process of recovering — and manufacturing acetates. H. W. Matheson, Shawinigan Falls, Canada. Eng. Pat. 137,388, 23.1.19. (Appl. 1730/19.)

THE hot vaporous mixtures emerging from the conversion tubes in the manufacture of acetone from acetic acid are passed into an alkaline solution at 85°–100° C., which is maintained in motion to promote the absorption of the acetic acid; concentration of the resulting solution occurs concurrently, and the resulting sodium acetate is finally separated from the water by crystallisation.—D. F. T.

Fatty acids; Process for the preparation of esters of —. Farbw. verm. Meister, Lucius, and Brüning, Höchst. Ger. Pat. 315,021, 19.9.15.

ACETYLENE is allowed to react, preferably at a high temperature, on an equimolecular mixture of a fatty acid and alcohol in the presence of the mercury salt of a mineral acid. For example, precipitated mercuric oxide is dissolved in acetic acid at 30°–40° C., and the solution treated, drop by drop, with strong sulphuric acid so as to precipitate mercuric sulphate in a finely divided state.

Ethyl alcohol is added to the mixture, which is heated to 70° C. in a shaking apparatus fitted with a reflux condenser, while a current of acetylene is passed through it. The acetaldehyde produced is separated from the ethyl acetate by fractional distillation. Formic acid and ethyl alcohol yield ethyl formate, and propionic acid and methyl ether give methyl propionate.—A. R. P.

Tobacco; Process of curing —. E. G. Reinhardt, Washington, D.C. U.S. Pat. 1,327,692, 13.1.20. Appl., 8.5.19.

The leaf, whether harvested separately or upon the stalk, is submitted to the action of air at about 105° F. (41° C.) and of about 60% relative humidity so as to reduce the moisture content and form a green overcast due to a film of dead chlorophyll cells in the leaf surface; the air is then changed to a temperature of 90° F. (32° C.) and a relative humidity of 95% for about 48 hours to bring the colour of the leaf to a mottled yellow, and the characteristic appearance and quality of well-cured tobacco are then developed by treatment with air at about 105° F. and of 72% relative humidity, 48—72 hours being generally sufficient for this final stage.—D. F. T.

Cacodyl and cacodyl oxide (Cadet's fuming liquid); Preparation of non-inflammable —. E. Merck, Darmstadt. Ger. Pat. 299,040, 16.3.15.

The inflammability of cacodyl and cacodyl oxide is greatly reduced, and even entirely overcome, by the addition of suitable amounts of sulphur.—W. J. W.

Odorous substances; Fixation of — in solid or liquid media. O. Bracmer, Hamburg. Ger. Pat. 314,829, 19.3.18.

TANNIC acid or gallic acid or one of their salts is added to the substance along with the characteristic essential oil, and in some cases with the further addition of sugar. The process is applicable to the manufacture of perfumes, soaps, scent-sachets, and, particularly, of artificial seasonings. For instance, in the preparation of artificial cinnamon, an intimate mixture of cinnamon oil, tannic acid, and sugar is incorporated with potato or turnip meal.—B. V. S.

Arsenical compounds; Aromatic —. Rockefeller Institute for Medical Research, Assignees of W. A. Jacobs, W. H. Brown, M. Heidelberger, and L. Pearce, New York. Eng. Pats. 120,381, 120,383, 120,384, and 120,385, 3.10.18. (Appls. 16,092, 16,094, 16,095, and 16,096/18.) Int. Conv., 3.10.17.

SEE U.S. Pats. 1,280,119, 1,280,121, 1,280,122, and 1,280,113 of 1918; this J., 1919, 268 A.

Citric acid; Manufacture of —. W. J. Mellersh-Jackson, London. From Citro Chemical Co. of America, Maywood, N.J., U.S.A. Eng. Pat. 137,396, 30.1.19. (Appl. 2376/19.)

SEE U.S. Pat. 1,288,293 of 1918; this J., 1919, 199 A.

Ether; Process for the continuous production of —. E. A. Barbet, Assignor to E. Barbet et Fils et Cie., Paris. U.S. Pat. 1,328,258, 20.1.20. Appl., 5.5.16.

SEE Eng. Pat. 100,406 of 1916; this J., 1917, 569.

Bases from tar oils. Ger. Pat. 301,306. See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Hypersensitising [photographic] panchromatic plates. S. M. Burka, J. Franklin Inst., 1920, 189, 25—46.

If panchromatic plates are treated with ammonia

before exposure, their speed is very considerably increased, but their keeping quality is considerably reduced. A similar treatment has an almost negligible effect on most ordinary or orthochromatic plates, and the effect is apparently due to interaction between the dyes used in the panchromatic plates and the ammonia. The speeding effect was greatest if a plain aqueous ammonia solution was used, but plates so treated would keep for only 2 or 3 days; the bath recommended for use contains 25% of ethyl alcohol and 3% of 20% aqueous ammonia solution. The plates are bathed for 4 minutes and dried rapidly. The increase of speed to white light is in most cases about 100%, and more to red light, being in some cases 400%. The sensitiveness in the red is also extended about 100 Angström units. The spectral sensitiveness curve is also somewhat smoother after treatment. A number of tables, diagrams, and illustrations are given, showing the effect of the treatment on various brands of panchromatic plate.—B. V. S.

[Photographic] sensitising with bichromate. R. Namias. II Progressio Fotog., 1915, 208. Bull. Soc. Franç. Phot., 1919, 6, 331—332.

CARBON-TISSUE paper sensitised with bichromate keeps for only a few days. If the normal salt or the normal salt with a small addition of alkali be used in place of the bichromate, the paper keeps almost indefinitely, but is too slightly sensitive for use. The combination of good keeping quality and high sensitiveness may be obtained by coating the paper with a mixture containing slightly alkaline chromate, and then treating it, just before use, for about half an hour in a closed box at ordinary temperature with the vapour of a volatile acid, such as acetic acid.—B. V. S.

PATENTS.

Wedge colour-filter for distant photography. O. Will, Munich. Ger. Pat. 309,167, 28.8.17.

A COMPOSITE colour-filter contains two or more wedge filters, cemented between glass; the different wedges may be of different colours and their slopes may be in the same or in opposite directions.

—B. V. S.

Selenium cells. Ger. Pat. 301,261. See XI.

XXII.—EXPLOSIVES; MATCHES.

Specific heat of mixtures of sulphuric and nitric acids. Pascal and Garnier. See VII.

PATENTS.

Explosives; Process for manufacture of —. Zentralstelle f. Wissensch.-tech. Untersuch., G.m.b.H., Neubabelsberg. Ger. Pat. 299,028, 15.6.16.

AN explosive consists of nitro-hexamethylenetetramine, $C_6H_{12}N_4O_6$ (Ger. Pat. 101,280), which may be mixed with other ingredients. The explosive is stable, sensitive to shock, and gives a good expansion in the Trauzl lead block.—W. J. W.

Nitrocellulose powder; Process for increasing the density or diminishing the porosity of — in the form of cords or threads. I. M. Voith, Heidenheim. Ger. Pat. 300,737, 21.4.17.

THE amount of collodion cotton added to the explosive for gelatinising purposes is raised from 20—25% to 27—30% in order to increase the density. To prevent the newly pressed powder from becoming porous when treated with water, the collodion cotton may be increased to 45%.

—W. J. W.

Explosives; Process for "drying" [removing solvent from] freshly manufactured —. I. M. Voith, Heidenheim. Ger. Pat. 302,560, 30.6.16.

THE removal of solvent from tubular or cord-shaped explosives may be effected by treatment with water or aqueous solutions of alcohol, acetone, salts, etc. The process is free from danger, and is said to be more rapid than drying by hot air or in a vacuum.—W. J. W.

Cellulose products; Process for displacement of liquids from — by other liquids. Elektro-Osmose Akt. Ges. (Graf Schwerin Ges.), Berlin. Ger. Pat. 305,019, 25.10.17.

THE displacing liquid is forced under pressure through a thin layer of the material, swelling of the material being prevented. The process may be carried out at raised temperatures. The density of the material may be increased by a preliminary pressing. The method may be utilised for the displacement of water from nitrocellulose by means of alcohol.—W. J. W.

Perchlorate explosives; Increasing the sensitiveness of — by addition of potassium permanganate as catalyst. C. Bunge, Bradegrube. Ger. Pat. 303,289, 8.4.17.

POTASSIUM perchlorate and potassium permanganate are crystallised together and the mixed crystals of suitable size are incorporated with other ingredients. By this means the full catalytic effect of the permanganate is made available, and its decomposition by other ingredients, such as oxalates, is avoided.—W. J. W.

Nitro-urea explosive. Badische Anilin- u. Soda-Fabrik, Ludwigshafen. Ger. Pat. 303,929, 17.7.15.

NITRO-UREA or its compounds or derivatives are mixed with inorganic oxygen-evolving compounds, or with other explosives. A suitable composition contains nitro-urea, 83%; potassium nitrate, 17%. If 10% of nitro-urea is added to 90% of ammonium nitrate it increases the "brisanee" of the latter. As nitro-urea is not very sensitive, a strong detonator is required.—W. J. W.

Ammonium perchlorate explosive. O. B. Carlson, Mansbo, Assignor to Aktiebolaget Carlit, Stockholm, Sweden. U.S. Pat. 1,327,985, 13.1.20. Appl., 25.9.17.

SEE Eng. Pat. 112,417 of 1917; this J., 1918, 637 A.

XXIII.—ANALYSIS.

Melting point methods at high temperatures. II. L. I. Dana and P. D. Foote. Chem and Met. Eng., 1920, 22, 63—70. (See also this J., 1920, 175 A.)

THE melting point of silicates is determined preferably in a tall form of metal crucible, using an unprotected thermo-couple, and leakage of current from the couple to the molten salt and from the furnace heating current to the couple must be considered in precision work. Platinum resistance thermometers give greater accuracy than thermo-couples below 800° C., but above this temperature the calibration is subject to changes on account of volatilisation of platinum, and of strains introduced by the warping of the mica frame. Above 1400° C. the melting point of metals is observed through a hole in the cover of a crucible containing the metal, by means of an optical pyrometer. The crucible should be set in an outer tube of non-porous material which is inserted into the heating chamber. A vacuum furnace, such as the Arseni graphite resistance furnace, is necessary if the metal is subject to oxidation. A modification of the

wire method may be used for metals melting above the range of platinum. The material in the form of wire or ribbon is heated electrically until it fuses and the highest intensity is measured by an optical pyrometer. This method involves a knowledge of the emissivity of the particular material. Micropyrometric observations are subject to error because of the unequal heating of the platinum strip and the sample resting on it by the traversing current. In general the specimen should be protected from reducing action in the furnace by means of a suitable surrounding tube. Sillimanite porcelain may be used for this purpose up to 1800° C., but no protection tubes have been found satisfactory for higher temperatures.—C. A. K.

Qualitative analysis in the presence of phosphoric acid; Procedure for —. H. Remy. Z. anal. Chem., 1919, 58, 385—392.

IN the scheme of analysis described, phosphoric acid is used as a group reagent. The solution, containing any of the common metals, alkaline-earth metals, and alkali metals, and phosphoric acid, is treated with hydrogen sulphide and the sulphides are filtered off; the filtrate is boiled, filtered, and oxidised with nitric acid. Ammonium carbonate is added until the solution is nearly neutral, the mixture is poured into an equal volume of ammonia and an excess of ammonium phosphate is added. Aluminium, chromium, iron, calcium, strontium, and barium are precipitated as phosphates and manganese and magnesium as ammonium-phosphates; zinc, nickel, cobalt, and alkali metals remain in solution (sol. 1). The precipitate is separated, dissolved in a small quantity of hot dilute hydrochloric acid and potassium sulphate solution is added; barium, strontium, and the greater part of the calcium are precipitated and may be separated from each other and identified in the usual way. The filtrate from the alkaline-earth sulphate precipitate is heated to boiling with the addition of solid sodium acetate and ammonium chloride; iron, chromium, aluminium, and manganese are precipitated (ppt. A). The filtrate from this precipitate is treated with ammonium oxalate to precipitate the remaining quantity of calcium, and is subsequently rendered ammoniacal to precipitate magnesium. The precipitate (A) is dissolved in hydrochloric acid, the solution heated and sodium carbonate is added as long as the precipitate formed redissolves; the solution is then poured, while warm, into a mixture of 20% sodium hydroxide solution and hydrogen peroxide. Ferric hydroxide and hydrated manganese dioxide are precipitated whilst chromium and aluminium remain in solution as chromate and aluminate, respectively. The iron is identified by the ferrocyanide reaction and manganese by conversion into permanganate. Aluminium is precipitated as hydroxide by boiling the aluminate solution with the addition of ammonium chloride, and chromium is identified by the usual tests. The solution (I) containing the zinc etc. is heated and treated with ammonium sulphide; on adding hydrochloric acid to this precipitate, the zinc sulphide dissolves leaving the nickel and cobalt sulphides insoluble. The two latter metals may be identified in the usual way. The filtrate from the total sulphide precipitate contains the potassium and sodium together with phosphoric acid, but the latter does not interfere with the perchloric acid test for potassium or with the pyroantimonate test for sodium.—W. P. S.

Calcium; Modified McCrudden gravimetric method for [the determination of] —. J. O. Halverson and J. A. Schulz. J. Ind. Eng. Chem., 1920, 12, 77—78. (See also this J., 1911, 1282.)

IN the modification proposed the calcium oxalate precipitate is collected on a specially prepared asbestos filter and then titrated with perman-

ganate. High-grade ignited asbestos is heated at 60° – 70° C. for 1 hour with dilute sulphuric acid (1:5), and then washed with water containing a trace of permanganate until free from acid. By shaking the washed asbestos with water it may be separated into three grades of fineness—fine, medium, and coarse. A layer of the coarse is placed in a Gooch crucible, and on this are placed layers of the medium and fine grades respectively. After the precipitate has been washed the filter and precipitate are transferred to a flask containing hot water, sulphuric acid (1:5) is added, and the oxalate titrated at 65° C. with permanganate.—W. P. S.

Arsenic; Process for the estimation of — with notes on the chemistry of the Marsh-Berzelius process. B. S. Evans. Analyst, 1920, 45, 8–17.

THE arsenic is evolved as hydrogen arsenide, which is passed over ignited copper and the arsenic retained by the copper is weighed. The apparatus used is essentially the same as in the Marsh-Berzelius method, but the tube attached to the drying tube has a drawn-out portion only about 2 in. long, the last inch of which is turned up. If this tube is made of silica it can be used repeatedly. A roll of thin sheet copper about 4 in. by 1 in., previously heated to redness and cooled, is introduced into the silica tube, which has a small fragment of ignited asbestos wool at the constriction, and the tube is supported horizontally over a Bunsen flame, whilst the portion containing the copper is covered by a small sheet iron muffle about $1\frac{1}{2}$ in. long, and a small asbestos sheet is used to protect the rubber connexion. After hydrogen has been passed through the tube for 2 or 3 mins. the burner is removed, the tube allowed to cool, the hydrogen replaced by air by means of a filter pump, and the tube weighed. It is then again attached to the hydrogen flask, again heated, and the hydrogen jet relighted. When the tube is red hot the solution containing the arsenic under examination is introduced into the flask, and the evolution of gas continued for 45 mins., the height of the hydrogen flame being maintained at about $\frac{1}{2}$ in. Finally the tube is cooled, and after removal of the hydrogen is weighed as before. The increase in weight divided by 0.986 gives the amount of arsenic. This correction factor was based upon results obtained with different amounts of pure arsenious oxide. Experiments indicated that the loss of arsenic was not due to its leaving the apparatus with the hydrogen. When pure zinc was used a scum of elementary arsenic formed on the surface of the acid and remained practically unchanged at the end of the test. This represented a considerable loss of arsenic, but by coating the zinc with cadmium, as suggested by Chapman, the floating arsenic is removed. To prevent separation of elementary arsenic it is advisable to keep the amount of arsenic added well below 0.5 gm. The addition of copper sulphate to the liquid in the flask prevents the formation of arsine by the formation of copper arsenide and by causing a large amount of elementary arsenic to separate. In the case of iron the amount of arsenic retained is approximately proportional to the amount of iron present. The arsenide residues invariably contained lead, derived from the cadmium, and apparently brought into combination with the arsenic through the influence of the iron. Sodium sulphate, disodium hydrogen phosphate, and zinc sulphate have no retaining effect. This method was used to test the amount of arsenic left in solution in the filtrate and washings in the magnesium arsenate method. It was found that the second filtrate containing no magnesium mixture dissolved about four times as much arsenic as the first, although its volume was not much greater. The amount of arsenic remaining in solution is by no means negligible, and it is

necessary to see that the volumes of both filtrates and washings are kept constant when solution factors are used to correct the results. (See also J. Chem. Soc., 1920, ii., 125.)—C. A. M.

Arsenic; Estimation of small quantities of —. W. van Rijn. Pharm. Weekblad, 1919, 56, 1072–1083.

SMALL quantities of arsenic, as little as 1 mgrm. As per litre, may be accurately estimated by the following methods:—Treatment with potassium chlorate and hydrochloric acid (Fresenius and Babo); absorption by ferric hydroxide (Paucke. Beitr. z. Nachw. v. Arsen, Inaug. Diss., Leipzig, 1908); acidification with sulphuric acid, addition of solution of bromine in potassium bromide, then ammonia and magnesia mixture; or treatment with ammonia and magnesia mixture and addition of sodium phosphate (Berntrop). The arsenic mirror is obtained by the use of Bloemendaal's apparatus and the final estimation by titration (Berntrop, Chem. Weekblad, 1906, 315). Comparison with a standard mirror is not sufficiently accurate. Wallpapers, paint and blind materials may be tested by the same methods.—W. J. W.

Fat extraction apparatus. J. M. Pickel. J. Ind. Eng. Chem., 1919, 11, 1053–1055.



THE extraction vessel consists of a small glass cylinder, A, in place of the usual flask; a crucible, B, containing the substance to be extracted is supported in the cylinder, indentations being provided in the side of the cylinder for this purpose. A cup, C, inside which at one side is a funnel (the bent stem of this funnel passes through the bottom of the cup), fits over the top of the crucible, and a condenser, A, is attached to the top of the cylinder. When the ether in the latter boils the vapours are condensed and fall from the condenser through the funnel into the crucible containing the substance. At the end of the extraction the condenser is rotated partially, so that the condensed ether falls into the cup and does not return to the cylinder.—W. P. S.

Uranium; Determination of — and its separation from other rare elements. C. A. Pierlé. J. Ind. Eng. Chem., 1920, 12, 60–63.

THE more common methods of determining uranium have been critically examined. Good results were obtained by adding ammonia solution to uranium nitrate solution, boiling the solution containing the precipitate for 15 mins., separating the precipitate, washing it with 2% ammonium nitrate solution, and drying and igniting it in a porcelain crucible. Precipitation of the uranium as uranyl ammonium phosphate by means of ammonium dihydrogen phosphate (Kern, this J., 1901, 1144) was found to be difficult, owing to the tendency of the precipitate to pass through the filter paper and to the readiness with which uranyl pyrophosphate absorbs moisture. Precipitation as uranium sulphide, and ignition of the precipitate after washing with hot 2% ammonium nitrate solution, invariably gave too high results owing to part of the sulphur being oxidised to sulphate during the ignition. The volumetric determination of uranium by means of potassium

permanganate, using Kern's modification (*loc. cit.*), also gave high results, owing to the reduction being carried below the quadrivalent condition of the uranium. A method for separating uranium from molybdenum, vanadium, and tungsten has been based on the fact observed by Peligot (Ann. Chim. Phys., 1842 [3], 5, 1) that uranyl nitrate is readily soluble in ether. Another method for separating uranium from vanadium depends on the fact that acetic acid of 95% or over when mixed with nitric acid (sp. gr. 1.42) in the proportion of 20 vols. to 1 vol. HNO_3 does not dissolve vanadium pentoxide, but readily dissolve vanadium nitrate.—C. A. M.

Sulphates. Hibbard. See VII.

Hydrocyanic acid. Chelle. See VII.

Potassium anhydrotellurate. Hulot. See VII.

Chemical glassware. Muirhead and Turner. See VIII.

Chemical glassware. Thieme. See VIII.

Linseed oil. Steele and Washburn. See XII.

Sucrose. Dixon and Mason. See XVII.

Malt analysis. Langkammerer and Leberle. See XVIII.

Thin beers. Dietsche and Grave. See XVIII.

Acetylmethylcarbinol. Lemoigne. See XVIII.

Alcohol-water mixtures. Kolthoff. See XVIII.

Pasteurised milk. Fouassier. See XIXa.

Dry gluten in flour. Bouyer. See XIXa.

Sugar in presence of protein degradation products. Last. See XIXa.

Plant juices. Haas. See XIXa.

Chlorinated methyl formates. Delépine. See XX.

Acetone. Richter-Quittner. See XX.

Pseudo-iodine value of essential oils. Huerre. See XX.

PATENTS.

Analysing gases [determining carbon dioxide in flue gases]; Absorption apparatus for —. P. R. Beulton, Birmingham. Eng. Pat. 136,992, 10.4.19. (Appl. 9136/19.)

A PORTABLE apparatus for analysing gases consists of a reciprocatory pump, desiccating, absorption and gauge chambers, an indicating gauge, and a controlling valve, so arranged that a sample of dry gas drawn in by the pump is discharged from one side of the piston, through the absorbent to the opposite side, which communicates with the gauge chamber. The displacement of the piston required to restore the initial pressure in the gauge chamber, or the pressure produced in the latter, is employed as a measure of the constituent to be determined.

—W. E. F. P.

Colour fastness. Eng. Pat. 136,971. See VI.

Viscosity of rubber. U.S. Pat. 1,327,838. See XIV.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Bailey and Hartley. Centrifugal separators. 5252. Feb. 21.

Beeston, Stevenson, and Beeston and Stevenson. Furnaces. 4528. Feb. 14.

Delaygue. Refrigerating. 4503. Feb. 13. (Fr., 1.5.14.)

Douglas. Condensers etc. 3851. Feb. 9.

Dupare and Urfer. 4729. See VII.

Elmore. Centrifugal driers. 3894. Feb. 9.

Engelke. Filtration of liquids. 4065. Feb. 10.

Fabry. Continuous stills for dissociation of solutions by heat. 4032. Feb. 10.

Feldenheimer and Plowman. Catalysts and catalytic reactions. 4800. Feb. 17.

Hilop. Drying-chambers. 4320. Feb. 12.

Hobbs. Drying and mixing machines. 4090. Feb. 10.

Kennedy. Crushing apparatus. 5223, 5224, 5227, 5228. Feb. 20. (U.S., 2.2.14, 6.3.14, 23.10.17, 14.1.18.)

Merz. Receptacles for crystallisation of solutions. 4267. Feb. 11.

Metallbank u. Metallurgische Ges. Evaporation of liquors. 3895 and 3896. Feb. 9. (Ger., 27.2.15 and 13.3.19.)

Millard. Gas-impregnating apparatus. 4869. Feb. 17.

Raper and Sutcliffe. Recovery of gases or vapours. 3988. Feb. 9.

COMPLETE SPECIFICATIONS ACCEPTED.

13,873 (1914). Wauthier. Grinding, crushing, or pulverising machine. Feb. 25.

7430 (1918). Linden. See XII.

17,256 (1918). Peck. Filter apparatus. (138,657.) Feb. 25.

18,691 (1918). Bateman. See II.

2213 (1919). Kirby and Lakin. Grinding and pulverising materials. (138,411.) Feb. 18.

6951 (1919). Western Electric Co. Producing vacua. (138,745.) Feb. 25.

16,774 (1919). Zack. Cooling or liquefying gases or gaseous mixtures. (129,292.) Feb. 25.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Austin Motor Co., and Walker. Apparatus for generating producer gas. 5002 and 5003. Feb. 19.

Beilby. Motor fuel. 4562. Feb. 14.

Blakeley, and Firth, Blakeley, Sons and Co. Gas purifiers. 3860. Feb. 9.

Bourdon. Gas-purifying apparatus. 4232. Feb. 11. (Fr., 6.3.19.)

Bull (Helgeby and Schieldrop). Carbonising coal. 4977. Feb. 18. (Norway, 19.2.19.)

Bury, Jones, and Minerals Separation, Ltd. Flotation treatment of carbonaceous matter. 4988. Feb. 18.

Davis and Levy. 3978. See XXIII.

Dowson and Mason Gas Plant Co., and Paton. Gas-fired furnaces. 4573. Feb. 14.

Drake, and Drakes, Ltd. Vertical retort settings. 3863. Feb. 9.

Engelke. Treating mineral oils. 4370. Feb. 12.

Fairweather. 4223. See XXIII.

Hacking. 4297. See IX.

Hallam. Petrol substitute. 5042. Feb. 19.

Harger. Lubricating oil. 4928. Feb. 18.

Kennedy. Retorts. 5220. Feb. 20. (U.S., 31.3.19.)

Southcombe and Wells. Lubricating oils. 4763. Feb. 17.

Terrell. 4108. See XXI.

Williams and Williams. Gas-producer. 5247. Feb. 21.

COMPLETE SPECIFICATIONS ACCEPTED.

18,691 (1918). Bateman. Deodorising, purifying, distilling, and vaporising oils and other liquids. (138,388.) Feb. 18.

- 2444 (1919). Hills. Manufacture of fuel briquettes. (138,414.) Feb. 18.
 3431 (1919). Fornas. Gas-producers. (123,323.) Feb. 18.
 3927 (1919). Sarnmark. Fillings for tanks etc. for storing acetylene or other gas. (123,534.) Feb. 25.
 10,175 (1919). Asserato. Combustion of solid pulverised fuel. (138,502.) Feb. 18.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

- Koppers. Purification of liquors containing phenoloid bodies. 4595. Feb. 14. (U.S., 15.2.19.)
 Melamid. 4591. *See* XI.
 Miersch. Recovering fluoric acid from sulphonating mixtures of organic sulpho acids and calcium fluoride. 4461. Feb. 13.

COMPLETE SPECIFICATIONS ACCEPTED.

- 13,356 (1919). Bloxam (Durand et Huguenin Soc. Anon.). Manufacture of condensation products from formaldehyde and aromatic derivatives. (138,796.) Feb. 25.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

- Anderson and Attack. Manufacture of vat dye-stuffs. 4166. Feb. 11.
 Ehrhardt and Ehrhardt. Manufacture of mordant-dyeing azo colour. 4642. Feb. 16.
 Hart and Stewart. Dyestuffs and mode of producing them. 5267. Feb. 21.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

- Braun. Manufacture of cellulose. 4693. Feb. 16. (Gr., 29.5.18.)
 Bronnert. Manufacture of viscose silk. 4877. Feb. 17.
 Courtaulds, Ltd., and Stokes. Washing etc. artificial filaments, fibres, etc. 3951. Feb. 9.
 Fearnley. Manufacture of paper. 3916. Feb. 9.
 Fearnley. Treatment of wool. 3874. Feb. 9.
 Soc. Anon. Etabl. A. Olier. Treatment of fibrous plants for conversion into cellulose for paper-making. 4213. Feb. 11. (Fr., 11.2.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 11,098 (1917). Korselt. Manufacture of silk. (108,489.) Feb. 18.
 11,895 (1917). Korselt. Manufacture of wool, hair, etc., and vegetable fibres. (109,051.) Feb. 18.
 13,569 (1918). Stevenson. *See* XXI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATION.

- King. Bleaching and bleaching-compounds. 5188. Feb. 20. (U.S., 28.6.19.)

COMPLETE SPECIFICATION ACCEPTED.

- 16,634 (1919). Färberei Weidmann A.-G. Apparatus for dyeing or otherwise treating fabrics. (129,975.) Feb. 18.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

- Bracq. Furnaces for roasting pyrites. 4262. Feb. 11.
 Brothers. Manufacture of plaster of Paris. 4890. Feb. 18.
 Duparc and Urfer. Synthetic production of ammonia. 4723. Feb. 16. (Switz., 13.3.19.)
 Duparc and Urfer. Production of catalysts. 4729. Feb. 16. (Switz., 13.3.19.)
 Gerber. Producing aluminium nitride. 4976. Feb. 18. (Switz., 18.2.19.)

- Maconochie and Ros. 4488. *See* X.

- Miersch. 4461. *See* III.

- New Jersey Zinc Co. 4369. *See* XIII.

- Norske Aktieselskab for Elektrokemisk Industri. Removal of iron from solutions containing aluminium. 5040. Feb. 19. (Norway, 22.2.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 6448 (1919). Soc. l'Air Liquide. *See* XVI.
 8152 (1919). Shroff. Preparation of barium chloride and other chlorides of alkaline-earth metals. (138,486.) Feb. 18.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

- Empire Machine Co. Means for obtaining glass from furnace. 3994. Feb. 9. (U.S., 15.11.13.)
 Imperial Trust, Institution of Gas Engineers, and Mellor. Clay etc. 4475. Feb. 13.
 Imperial Trust, Central School of Science and Technology, Mellor, and Moore. Moderants in use of clays, preparation of china, etc. 4476. Feb. 13.
 Soc. Boucher et Fils. Glass tank-furnaces. 4387. Feb. 12. (Fr., 27.9.18.)

IX.—BUILDING MATERIALS.

APPLICATIONS.

- Elborne. Manufacture of bricks, fictile articles, and cement. 5237. Feb. 21.
 Hacking. Composition of asphalt. 4297. Feb. 12.
 Lewis. Continuous kilns for burning bricks etc. 4398. Feb. 13.
 Mattison. Manufacture of fibrous cement products. 4337. Feb. 12. (U.S., 12.2.19.)

COMPLETE SPECIFICATION ACCEPTED.

- 14,182 (1919). Gronroos. Production of artificial stone or slabs of marble-like appearance. (138,799.) Feb. 25.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

- Allsebrook and Hetherington. Treatment of lead sulphido ores. 4886. Feb. 17.
 Coles. Preventing corrosion and pitting of steel. 3980. Feb. 9.
 Coles. Production of flexible zinc. 5000. Feb. 19.
 Coles. Sherardising drums. 5192. Feb. 20.
 Crosthwaite Engineering and Furnace Co., and Crosthwaite. Furnaces for heating crucibles for heating metals etc. 4146. Feb. 11.
 Diebl. Utilising and recovering sulphur in blast-furnace slag. 4694 and 4695. Feb. 16. (Ger., 16.1.17 and 24.9.17.)
 Gerber. Producing aluminium. 4975. Feb. 18. (Switz., 18.2.19.)
 Jackson (Forge Products Corporation). High-speed steel. 4109. Feb. 10.
 Jones. Heating metallurgical furnaces etc. 4623. Feb. 16.
 Levoz. Manufacture of indirect steel. 4864. Feb. 17. (Fr., 27.3.19.)
 McCaffery. Bessemerising iron. 4061. Feb. 10. (U.S., 13.11.18.)
 McConway and Vigeon. Pre-leaching apparatus for treating calcined ore etc. 5065. Feb. 19.
 Maconochie and Ros. Oxidation of zinc, tin, etc., and production of oxides of such metals. 4488. Feb. 13.
 Middlemiss. Apparatus for treating ores. 5129. Feb. 20.
 Treanor. Alloys for filling in defective castings. 4258. Feb. 11.
 Vautin. Treatment of ores. 4371. Feb. 12.

COMPLETE SPECIFICATIONS ACCEPTED.

- 4474 (1919). Huntington, Heberlein, and Co., and Bingham. Mechanical furnaces for roasting ores. (138,443.) Feb. 18.

6739 (1919). Soc. Anon. de Vedrin, and Marcotty. Roasting furnaces for sulphurous ores. (138,472.) Feb. 18.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Clark. Primary cells and batteries. 4958. Feb. 18.

Melamid. Manufacture of transformer oils from tar oils. 4591. Feb. 14. (Ger., 15.5.19.)

Pouchain. Negative plates for accumulators. 4117—4120. Feb. 10.

Swaay. Storage batteries. 4667. Feb. 16. (Holland, 15.2.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

1731 (1919). Matheson. Electrolytic cells. (138,406.) Feb. 18.

5706 (1919). Fery. Electric storage cells (121,211.) Feb. 25.

10,066 (1919). Ivey and Storey. Electric storage battery cells. (138,773.) Feb. 25.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Hey. Removing suspended matter from liquid oils and from solvents containing oils, greases, and waxes in solution. 5239. Feb. 21.

Pericarp Synd., and Trevor. Obtaining oil from fruit heads of palm trees. 5050. Feb. 19.

Townsend. 4853. *See* XIX.

Tuckfield. Purifying oils and fats. 5111. Feb. 20.

COMPLETE SPECIFICATION ACCEPTED.

7430 (1918). Linden. Separating oil, grease, etc., from water or other liquids, or separating two liquids of different densities. (138,655.) Feb. 25.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Edser and Reynard. Protection of surfaces and waterproofing articles. 4983. Feb. 18.

Guillochin and Guimet. Manufacture of ultramarine. 4576. Feb. 14.

Maconochie and Ros. 4488. *See* X.

New Jersey Zinc Co. Treating zinc oxide. 4369. Feb. 12. (U.S., 9.8.18.)

Reeve. Rust-preventing paint. 4741. Feb. 17.

COMPLETE SPECIFICATION ACCEPTED.

17,508 (1919). Bienenfeld. Paint. (138,810.) Feb. 25.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Helbronner. Manufacture of indiarubber products. 4264. Feb. 11. (Fr., 19.4.19.)

Hunter Dry Kiln Co. Treatment of rubber. 4240. Feb. 11. (U.S., 25.9.15.)

Peachey. Vulcanisation of caoutchouc. 4288. Feb. 12.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Beeber, Davis, and Krouse. Treating raw hides and skins. 4716. Feb. 16.

Knowles, and McArthur and Co. Manufacture of tar derivative tanning materials. 4155. Feb. 11.

Marks (Chicago Process Co.). Liming and de-hairing hides. 4080. Feb. 10.

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Norsk Hydro-Elektrisk Kvaelfstofaktieselskab. Nitrogenous fertilisers. 3990. Feb. 9. (Norway, 24.2.19.)

Sams. Chalk fertiliser. 4803. Feb. 17.

COMPLETE SPECIFICATION ACCEPTED.

6448 (1919). Soc. l'Air Liquide. Converting synthetic ammonia into a transportable product directly utilisable for agriculture in conjunction with the production of carbonate of soda. (131,870.) Feb. 18.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Loring and Stacey. Bleaching flour. 4458. Feb. 13.

Townsend. Improving medicinal fatty foods. 4858. Feb. 17.

COMPLETE SPECIFICATIONS ACCEPTED.

12,297 (1917). Bollmann. Manufacture of food-stuffs. (109,265.) Feb. 25.

7430 (1918). Linden. *See* XII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Chem. Fabr. vorm. Sandoz. Selective isolation of alkaloids. 4827. Feb. 17.

Selden Co. Phthalic anhydride. 4692. Feb. 16. (U.S., 14.10.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

10,374 (1917). Marks (Du Pont de Nemours and Co.). Production of aromatic amines. (138,372.) Feb. 18.

1729 (1919). Matheson. Manufacture of acetone. (138,679.) Feb. 25.

13,760 (1919). Soc. Chim. des Usines du Rhône. Manufacture of mono- and di- β -oxyethylamino-benzoic esters. (128,552.) Feb. 18.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Donisthorpe, and Dye Impression Photos, Ltd. Photographic printing. 5290. Feb. 21.

Schauermann. Photographic films etc. 5207. Feb. 20.

Terrell. Flashlight powders. 4108. Feb. 10.

Whitfield. Colour photography. 5120. Feb. 20.

COMPLETE SPECIFICATIONS ACCEPTED.

13,569 (1918). Stevenson. Manufacture of non-inflammable films or filaments for cinematographic and other purposes. (138,379.) Feb. 18.

420 (1919). Finnigan and Rodgers. Producing coloured photographs or cinematograph films. (138,396.) Feb. 18.

XXII.—EXPLOSIVES; MATCHES.

APPLICATION.

Eschbach. Manufacture of initial priming compositions. 4597. Feb. 14.

COMPLETE SPECIFICATIONS ACCEPTED.

10,328 (1917). Manuelli and Bernardini. Explosives. (138,371.) Feb. 18.

21,439 (1919). Atlas Powder Co. Explosive mixtures. (134,524.) Feb. 18.

XXIII.—ANALYSIS.

APPLICATIONS.

Davis and Levy. Apparatus for detecting small quantities of carbon monoxide. 3978. Feb. 9.

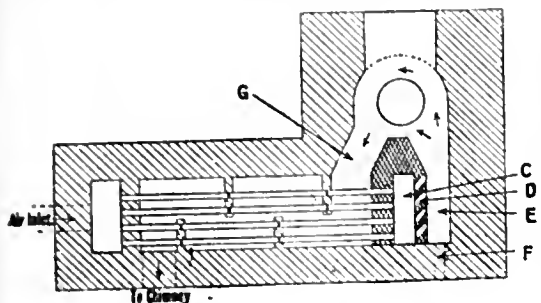
Fairweather. Gas calorimeters. 4228. Feb. 11.

Madsen. Recording differential thermometer and calorimeter. 5001. Feb. 19.

I.—GENERAL; PLANT; MACHINERY.

Recuperative furnace; A new type of —. W. Rosenhain and E. A. Coad-Pryor. Trans. Ceram. Soc., 1918-19, 18, 407-419.

AN experimental cylindrical recuperative gas-fired crucible or glass-pot furnace for temperatures above 1500° C., with natural draught, shown in horizontal



section in the fig., is 24 in. in diameter, 18 in. high internally, and contains 20 carborundum tubes each 4 ft. long and 1½ in. diameter. The air used for combustion passes through the carborundum tubes into a collecting chamber, C, thence, past baffles, D, into a mixing chamber, E, where it meets coal gas entering through three burners, F. The flame developed in E passes around the crucible, through the flue, G, into the recuperator and thence to the chimney. A highly-successful furnace on the same principle for testing refractory materials consists of a chamber 20 in. high with a floor, 2 ft. 9 in. wide and 1 ft. 6 in. deep, across which the flame passes horizontally and then downwards and through the recuperator which is at a lower level, the exit gases being compelled by three baffles to take a long zig-zag course around the carborundum tubes. The tubes are arranged in nine courses, each consisting of 13 tubes, each tube consisting of 4 shorter tubes 8 in. in length. The tubes and baffle walls are not tied in any way, so as to allow for expansion and contraction. A temperature of 1650° C. is readily attained in this furnace. The furnace consumes 600 cu. ft. per hour of town's gas of 500 B.Th.U. The air enters the furnace at 200°-300° C. below the furnace temperature. Magnesite bricks are quite unsuitable for lining the furnace; zirconia bricks are better but spall badly. Good firebricks washed over with a mixture of carborundum and china clay are satisfactory and easily renewed after 5 or 6 heats. The draught is created by a chimney stack less than 35 ft. high.—A. B. S.

Heating autoclaves by means of an oil burner. F. Muller. Chem. Weekblad, 1920, 17, 34-35.

THE heating arrangement consists of a laterally placed furnace of sheet iron lined with firebricks and having a small interior bridge of the same material, and a burner with two orifices of 2 mm. diam. which is supplied with oil and compressed air reduced to a pressure of 1½ atm. The autoclave is enclosed in an oil bath around which the burner gases circulate in two spiral paths. Cleaning doors are provided in the brick setting. A thin tar oil is used as fuel. The burner flame must be regulated so as to impinge on the furnace bridge, and must not extend beyond the furnace. To mitigate the effect of a possible explosion of oil vapour and air in the flues, produced by choking in the burner, the cleaning doors should not be clamped too tightly. The autoclave may be rapidly cooled by circulating the oil through a cooler.—W. J. W.

Cooling liquids in spray systems and cooling towers. E. M. Baker. Chem. and Met. Eng., 1920, 22, 173-176.

THE principle on which the various forms of spray systems and cooling towers are based is to bring the hot liquid into contact with cool air, cooling being effected by exchange of sensible heat and by evaporation of some of the water, thus removing some heat in the latent form. In general, most of the cooling is due to the latter effect, and in practice the amount of cooling is determined almost entirely by the extent of evaporation. The design of a cooling apparatus should aim to give complete saturation of the exit air and to discharge the air at as high a temperature as possible, as the partial pressure due to water vapour increases rapidly with increase in temperature. The cooling effect will always be increased by increasing the quantity of air, but the extent of the increase diminishes as the temperature of the exit air is reduced. The results of a series of tests on the "Ceco" spray system under varying conditions are described and illustrated by means of graphs. (See also this J., 1920, 179 A.)—C. A. K.

Refrigerating-machines. M. Ponchon. Chim. et Ind., 1920, 3, 24-27.

A REFRIGERATING machine consists essentially of a compressor, condenser-cooler, and evaporator-refrigerator, and is a device for transferring units of cold ("frigories") from the cooling water to the liquid to be cooled. Various tables and curves are given showing the effect of the temperature of the cooling water on the refrigerating efficiency and the power absorbed. By the addition of a pre-cooler through which the cooling water passes on its way to the condenser the temperature of the liquid to be cooled is brought nearer to that of the cooling water, and it is shown that an economy of power and greater efficiency may thus be attained. This, however, is only advantageous when the supply of cooling water is limited, but when this is the case the economy is considerable.—W. H. C.

PATENTS.

Drying apparatus. E. L. A. Savy, Paris. Eng. Pat. 123,984, 26.2.19. (Appl. 4856/19.) Int. Conv., 30.11.16.

THE apparatus is intended especially for substances such fruits, vegetables, and gelatinous or fine-grained pasty substances that are best dried in a current of warm, moist air in order to prevent the formation of a hard, dry shell which prevents escape of moisture from the interior. The material is placed on trays in a chamber through which air is passed after being heated (preferably by a steam coil). Most of the air is continuously circulated by means of a fan placed between the dryer and the heater, but a portion is allowed to exhaust to the atmosphere, and fresh air, which has been deprived of its moisture by cooling, is admitted to take its place. The regulation of these two quantities of air is effected by two dampers, one between the dryer and the suction of the fan which is regulated to produce a slight pressure in the dryer, and another in the inlet pipe for additional air.

—B. M. V.

Drying granular and other substances. J. J. Krüger, Copenhagen. Eng. Pat. 137,631, 5.2.19. (Appl. 2858/19.)

TRAYS which can be tilted are arranged one above the other within a tower, and heat is applied from heating elements arranged in the spaces above and below the trays. The material is fed in through a trap door at the top, and air is passed upwards through the tower. When the material in the

bottom tray is sufficiently dry the trays are tilted in succession from the bottom upwards, so that the contents of the lowest tray are discharged, and the contents of the other trays fall one step in succession, a fresh charge being admitted to the top tray. By operating the trays by mechanical means the process is made continuous.—B. M. V.

Drying machine [for clay]. De W. D. Irwin, Assignor to E. M. Knowles, East Liverpool, Ohio. U.S. Pat. 1,329,082, 27.1.20. Appl., 21.7.19.

THE material is carried by means of an endless conveyor through a horizontal drying chamber which is provided along the top and along at least one vertical side with openings which can be closed. A current of air is supplied through a chamber open at the bottom, which may be moved longitudinally along the top of the drying chamber so as to communicate with it through any desired openings. The material at any desired point may thus be subjected to the air current for any required length of time.—W. F. F.

Packing materials [resistant to chlorine] and processes for making the same. C. F. Wallace, New York, and M. F. Tiernan, Jamaica, N.Y., U.S.A. Eng. Pat. 131,871, 7.4.19. (Appl. 8797/19.) Int. Conv., 28.8.18.

A PACKING material, suitable for use in apparatus in which chlorine is employed, is made by dissolving beeswax in carbon tetrachloride and saturating asbestos string or rope with the solution. Graphite flake or powder is then incorporated with the asbestos and the solvent evaporated. The material may be wound around valve stems.—W. F. F.

Volatile solvents; Process for the collection and recovery of — evaporated into the atmosphere. L. A. Levy, London. Eng. Pat. 137,615, 27.1.19. (Appl. 1940/19.)

THE vapour-laden air is passed through one of two vessels containing active charcoal which absorbs the vapour; when the charcoal in one vessel has absorbed sufficient the air is passed through the other vessel, and the first one heated to recover the volatile solvent.—B. M. V.

Vapours [of volatile solvents]; Process for recovering — from air mixtures by sulphuric acid. A. Lehner, Berlin-Tempelhof. Ger. Pat. 303,396, 10.9.16.

THE process is applicable to the recovery of alcohol and ether from the air of explosives and artificial silk works. The air is passed through towers in series. The enriched acid from the lowest tower is withdrawn at intervals, e.g., by a siphon or pump, and the chamber filled with acid from a higher tower.—H. J. H.

Gas-fired [crucible] furnaces. J. W. Gaunt, and W. and J. Lawley, Ltd., West Bromwich. Eng. Pat. 137,628, 5.2.19. (Appl. 2778/19.)

A GAS-FIRED crucible furnace is built up in horizontal sections by two or more fire-brick annuli placed one on top of the other, so that the resulting chamber conforms to the shape of the crucible used. The bottom brick in addition has lateral ports for the entering gas flames, and the uppermost one an opening, preferably lateral, for the outlet of products of combustion. At points of local intense heat, blocks of a more refractory material, e.g., carborundum, may be inserted in the firebrick.

—B. M. V.

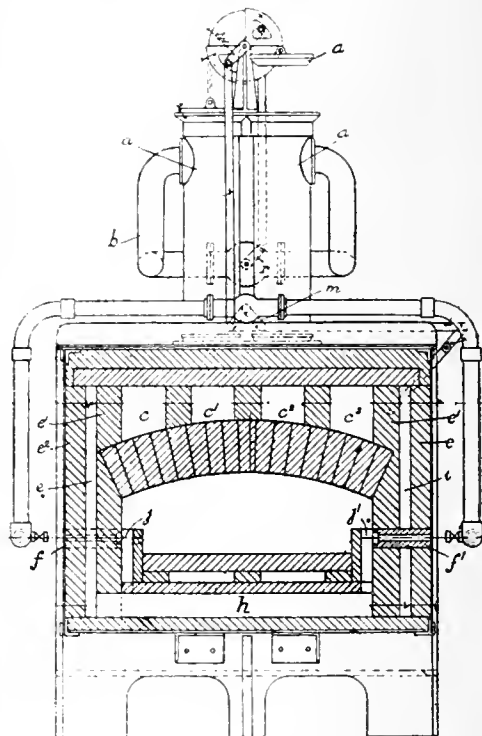
Combustion of gases in boiler furnace flues or tubes: Means for completing or promoting the —. W. Hassall, Woodville. Eng. Pat. 137,770, 18.8.19. (Appl. 20,307/19.) Addition to 128,325 (this J., 1919, 611 A).

IN order to ensure the more effective withdrawal

of the burnt dust from the flue, the block placed in the flue is tapered longitudinally, so that the annular passage is gradually restricted.—W. P.

Furnaces; Reversible gas-heated —. F. T. Renison, Sheffield. Eng. Pat. 137,977, 25.4.19. (Appl. 10,307/19.)

WITH the reversing valves in the position shown in the figure, air enters behind the reversing air valve, r, passes up the pipe, b, down the chimney,



a, to the longitudinal flue, c, in which it travels the full length of the furnace, then across to flue, c¹, again along the furnace, across to side flue, e, down which it passes to four (or other number of) transverse flues, h, to vertical passages, j¹, opening at the side of the hearth proper. The walls, e¹, e², of the side flues, e and i, are tied with bricks which are staggered so as to break up the incoming air or outgoing gases; some of these bricks, f, f¹, are formed with gas conduits in them supplied from a gas main behind the valve, m, and with the valves in the position shown the gas conduit-bricks, f, are shut off, while the conduits in f¹ are supplying gas which meets the heated air in j¹. Combustion takes place under the arch and the gases pass by vertical passages, j, down to transverse flues (not shown), which alternate with flues, h, then up through the side flue, i, twice along the furnace in c² and c³, and out to the atmosphere through the chimney, a¹, the valve-cap, a², of which is open.—B. M. V.

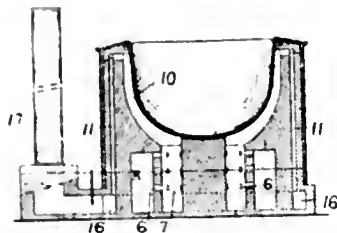
Grinding mills. O. Soder, Niederlenz, Switzerland. Eng. Pat. 137,640, 13.2.19. (Appl. 3582/19.)

IN a mill for fine grinding consisting of a grinding roller working against a grinding block, the latter is divided into segments in the direction of its width, the several segments being pivoted upon a pin which lies below the blocks (the roller running downwards as usual) and outside the angle subtended by the blocks to the centre of the roller; the object of this is that the grinding slot may taper, becoming narrower in the direction of rota-

tion. The pivot rod should be placed in such a position that the resultant of the frictional forces between the grinding blocks and roller passes through or near it; the upper edges of the grinding blocks are situated at about the point on the roller where the material would leave the latter due to centrifugal force. The grinding roller may be built up of an inner cast drum and outer grinding segments with a layer of heat insulating material between.—B. M. V.

Furnace setting. F. H. Nickle, St. Louis, Mo. U.S. Pat. 1,329,470, 3.2.20. Appl., 1.7.18.

A CONTAINER to be heated is supported in a furnace setting composed of firebrick and having concentric chambers as shown. A combustible mixture



is supplied to the intermediate annular chamber where it is burnt, and the combustion products pass through the openings, 6, to the annular chamber, 7, and thence through the space, 10, to heat the container. The gas then passes downwards through the annular space, 11, to the passage, 16, leading to the stack, 17.—W. F. F.

Refrigerating apparatus. B. E. D. Kilburn, London. From Sulzer Frères, Winterthur, Switzerland. Eng. Pat. 137,975, 23.4.19. (Appl. 10,157/19.)

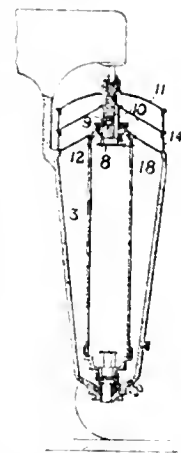
The apparatus is of the type in which the vapour of the refrigerating agent is removed from the refrigerator and compressed in an ejector by means of another working fluid circulating through the ejector, a cooler, and a pump. The refrigerating vapour is not cooled sufficiently to condense in the first cooler, but is there allowed to separate as vapour by gravity from the working fluid, and then condensed in a second cooler or condenser, whence it passes through an expansion valve back to the refrigerating coil. The same cooling water may be passed through the condenser and the cooler for the working liquid in succession. The whole apparatus (except the driving motor) may be contained in a double tank, and if a refrigerating agent is used which is lighter (when liquid) than the working fluid, a small passage may be provided from the bottom of the condenser portion of the tank to the working-fluid cooler, so that any spray of working fluid carried over into the condenser can flow back to its proper compartment. A special form of ejector is described in which the suction chamber is made large enough to collect the small quantity of working fluid contained in the nozzles, so that when the apparatus is stopped the working fluid does not flow into the refrigerator and become frozen there.—B. M. V.

Separator; Centrifugal — [for crude oil]. W. S. Estep, Cleveland, Okla. Assignor to R. V. Henry, Augusta, Kans. U.S. Pat. 1,307,000, 17.6.19. Appl., 20.4.18.

The apparatus is intended more particularly for separating foreign substances, such as particles of water, from crude mineral oil. The separating vessel is a closed cylinder rotating on its vertical axis, and the oil is supplied through a pipe which is fixed vertically in the axis of the vessel, and is provided with a number of radial discharge pipes terminating near the outer wall of the cylinder. The

bottom of the vessel is dished inwards in the form of a large cone projecting upwards to about two-thirds of the height of the vessel, and the resulting annular space is provided with large radial ridges projecting from the surface of the cone and each formed of two sloping plates forming an inverted V-shape in vertical cross-section. The outlets of the radial supply pipes are arranged above the radial troughs thus formed. As the result of centrifugal action the heavier impurity collects at the bottom of the vessel at its outer periphery, and the lighter clarified oil collects at the top and overflows into a central vertical discharge pipe surrounding the vertical shaft. The radial baffles prevent the impurities from collecting in a single mass, and thus destroying the balance of the vessel.—W. F. F.

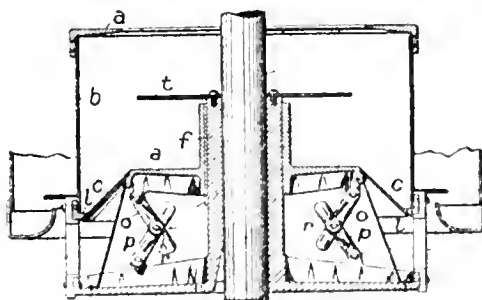
Separator; Centrifugal — P. T. Sharples, St. Davids, Pa. U.S. Pat. 1,320,119, 4.11.19. Appl., 29.10.18.



The liquids to be separated are contained in the rotating bowl, 3, which is provided at the top with a concentric ring, 8, having internal and external knife edges over which the lighter and heavier constituents respectively are discharged. The lighter constituent passes through the passage, 10, into the receptacle, 11, and the heavier constituent passes through the passage, 12, and over the weir, 9, into the receptacle, 14. Wings, 18, are provided to rotate the liquid with the bowl. This construction is adapted to maintain a constant ratio between the constituents in the separator, notwithstanding variations in the viscosity of the constituents and in the quantities discharged. The weirs, 8 and 9, are detachable, and may be replaced by others of different proportions.—W. F. F.

Centrifugal machines. E. D. Mackintosh, Brooklyn, N.Y., Assignor to S. S. Hepworth Co. and E. M. Mackintosh, New York. U.S. Pats. (A) 1,321,766 and (B) 1,321,767, 11.11.19. Appl., 30.10.18.

(A) The bottom of the centrifugal basket, *b*, consists of an inner part, *a*, sliding on the inner sleeve, *f*, and an outer frustum of a cone, *c*, the outer edge



of which is normally in contact with the lower rim, *t*, of the side wall, *b*, to close the basket. The bottom is pressed upward by a pair of toggle levers, *o*, *p*, controlled by a spring, *r*, which tends to open them and force the basket upwards, the centrifugal force also tending to open the toggles. The material to be treated is dropped on to the disc, *t*, and the solid matter is thrown on to the perforated side wall, *b*, where it collects in a layer. On stopping

the basket the solid matter by its weight forces the bottom down and is thus discharged. The bottom closes again when the basket is rotated. (b) The vertical spindle of the machine is driven from the vertical rotating shaft of a motor through a universal coupling member. The vertical shaft carries a T-shaped head, to the ends of which a frame having a depending lug at each end is pivoted to swing about the horizontal axis of the T-piece. The ends of the depending lugs engage freely with lugs projecting from a headpiece carried by the spindle of the centrifugal machine, and the headpiece rotates on ball bearings carried by a cup-shaped member which is non-rotatable, but which is supported in a spherical socket in a fixed support. The spindle of the centrifugal machine is thus free to swing slightly in any direction.

—W. F. F.

Filter-press devices. G. F. Miller, New York. U.S. Pat. 1,313,318, 19.8.19. Appl., 31.3.19.

THE filter-press is of the type in which there are two heads connected by side bars, and a movable follower operated by a screw from one of the heads to press together the filter elements, which slide on the side bars. The object is to improve the connexion between the side bars and the heads. Each bar is of channel section and fits into a recess in the side of the head. A longitudinal reinforcing block fits into each end of the channel and is provided with a T-piece overhanging the end of the channel bar. The head carries a stud which projects through a slot in the bottom of the channel bar and also through the reinforcing block, and the parts are secured against detachment by a small transverse plate screwed to the head.—W. F. F.

Drum-filter wiring. E. S. Pettis, Mill Valley, Cal. U.S. Pat. 1,327,962, 13.1.20. Appl., 5.3.19.

A FILTERING drum is surrounded by filtering material, and this in turn by a series of circumferential wires. A pair of bars are arranged on the periphery of the drum, parallel to the axis and close together, and the two ends of all the circumferential wires are attached to the two bars respectively.—W. F. F.

Hydrocarbon filter. Multistage adjustable filtering apparatus. Unitary multistage filter apparatus. H. A. Hills, Grand Rapids, Mich. U.S. Pats. (A) 1,328,044, (b) 1,328,045, and (c) 1,328,046, 13.1.20. Appl., (A) 23.7.16, (b, c) 7.5.17.

(A) A HORIZONTAL cylindrical casing is provided with a number of internal filter elements each in the form of a coaxial frustum of a cone, the cones being nested one within the other. The compartments thus formed are provided with outlets at the bottom leading to receptacles below, having transparent walls. An inlet for the unfiltered material is provided at one end of the casing and an outlet for the final filtrate at the other end. The filtering elements are mounted on an axial rod so as to be readily detachable. (b) A number of receptacles containing filtering material are provided with pipe connexions whereby the liquid to be filtered may be passed in succession upwards through the filtering material in each receptacle. Any one of the receptacles may be cut out of the series when desired. (c) The filters described in (b) are arranged in adjacent compartments in a single casing, and a heating device is provided in the bottom of each compartment.—W. F. F.

Evaporator. W. E. Garrigue (J. C. Guenther, administrator, Chicago, Ill.), Assignor to W. Garrigue and Co., Inc., New York. U.S. Pat. 1,317,488, 30.9.19. Appl., 19.2.19.

THE apparatus is of the type in which solutions to be concentrated are heated in a vertical tubular heater by means of superheated steam around the

tubes. The liquid is caused to circulate upwards through the central tubes and downwards through the outer tubes into a space below the lower tube-plate. The bottom plate of this space is concave, and is provided with a central tube leading downward to another vessel. The liquid moving radially over this plate sweeps any crystals formed into the outlet pipe, from which they pass to the vessel below. The vapour rises upwards from the tubes into a chamber above and passes into a separator at a higher level, and thence to a condenser. The bottom of the condenser is connected by a vertical tube of barometric height to an open collecting vessel, forming a liquid seal at the bottom, so that the vacuum is maintained in the system. When desired, liquid from the collecting vessel may be drawn by suction into the lower part of the evaporator through a valved pipe dipping into the liquid.—W. F. F.

[Evaporating] materials; Apparatus for treating —. C. R. Mahee, Toledo, Ohio. U.S. Pat. 1,329,786, 3.2.20. Appl., 23.10.15.

THE material is maintained in continuous movement over the inner cylindrical surface of a fixed horizontal cylinder by means of rotating baffles close to the surface. The cylinder is heated by means of an external jacket and the vapour from the treated material is withdrawn through an adjustable valve at the end of the cylinder, the valve being normally closed.—W. F. F.

Liquids; Process of concentrating —. G. T. Walker, Assignor to Colonial Chemical Co., Minneapolis, Minn. U.S. Pat. 1,330,016, 3.2.20. Appl., 17.6.18. Renewed 19.5.19.

THE liquid to be concentrated is mixed with an anhydrous salt capable of forming crystals containing water of crystallisation, and heated to the temperature at which the solubility is greatest. The solution is stirred until saturated and then cooled, when crystals are deposited, containing some of the water in the solution, which is thereby concentrated.—W. F. F.

Cooling liquids; Means for —. J. Hutchinson, Brooklyn, N.Y. U.S. Pat. 1,327,560, 6.1.20. Appl., 16.9.18.

LIQUID and gas chambers, each provided with baffles, are arranged adjacent to one another in heat-exchanging relation. Successive charges of compressed gas are expanded in the gas chamber, thereby falling in temperature and cooling the liquid, and each expanded charge is expelled before the next compressed charge is admitted.—W. F. F.

Conveyor. B. A. Parkes, Assignor to The Philadelphia Drying Machinery Co., Philadelphia, Pa. U.S. Pat. 1,328,099, 13.1.20. Appl., 8.10.17.

A SERIES of horizontal endless band conveyors are arranged one above the other, and each is composed of sections which are adapted to receive material on one face only. Each section is inverted as it approaches the end of its upper horizontal travel, and the material is transferred from it to the returning lower part of the conveyor. The inverted sections are re-inverted as they enter their lower horizontal travel, to receive the material as it falls from the upper part of the conveyor.—W. F. F.

Mixer. L. M. Brayman, Westville, N.H. U.S. Pat. 1,328,119, 13.1.20. Appl., 27.2.19.

THE mixer consists of a barrel of polygonal cross-section mounted to rotate with its axis horizontal. The angles between each end wall and the adjacent side walls are cut off within the barrel by inclined plates secured to both walls and joined to one another at their ends. The material in the barrel is thus directed away from the ends during rotation.—W. F. F.

Mixing concrete ingredients or other substances; Machines for —. F. W. Kiddie, Leamington. Eng. Pat. 137,901, 17.1.19. (Appl. 1281/19.) Addition to 124,112.

Electrical treatment of gases; Method and apparatus for —. L. Bradley, East Orange, N.J., Assignor to Research Corporation, New York. U.S. Pat. 1,329,825, 3.2.20. Appl., 21.6.16.

SEE Eng. Pat. 107,389 of 1917; this J., 1919, 61 A.

Catalytic agents for reduction or hydrogenation purposes; Process for producing —. A. Radisson (Assignor to Soc. de Stearinerie et Savonnerie de Lyon) and P. Berthon, Lyon, France. U.S. Pat. 1,330,003, 3.2.20. Appl., 18.5.17.

SEE Eng. Pat. 107,004 of 1917; this J., 1918, 431 A.

Drying, conditioning, and regulating the moisture content of hygroscopic materials; Method of and apparatus for —. W. H. Carrier, Assignor to Buffalo Forge Co., Buffalo, N.Y. U.S. Pat. 1,330,238, 10.2.20. Appl., 3.6.18.

SEE Eng. Pat. 121,080 of 1918; this J., 1919, 61 A.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Washing of —. A. Moreau. Rev. Mét., 1919, 16, 416—435.

In French practice, coal passing a 50—60 mm. screen is sent to the washery. It can be divided into two fractions, i.e., fine coal passing through a 10 mm. sieve, and the residue left on the sieve. The washing of the coarser portion can be controlled by visual inspection, but in the case of the finer material it is necessary to wash to a pre-arranged ash content, based on the constitution of the coal and on the terms of the coal specification. The author discusses from the mathematical standpoint the washing of this finer material.—A. G.

Wood charcoal; Combustion of — with nitric acid. F. C. G. Müller. Z. angew. Chem., 1920, 33, 40.

Wood charcoal was heated in a closed tube in a current of nitric acid vapour, and the resulting gases were collected and analysed. In three experiments the following results were obtained:—CO₂, 52.2—57.2; CO, 13.3—14.7; H₂, 3.4—4.6; N₂, 24.9—26.7%. The high CO content and the presence of free hydrogen are noteworthy. The liquid condensed from the issuing gases contained a considerable quantity of ammonia, the presence of which is difficult to account for, as the reaction temperature was certainly not less than 1000° C.

—E. H. R.

Carbon monoxide; Direct formation of — by burning carbon in pure oxygen. F. C. G. Müller. Z. angew. Chem., 1920, 33, 36.

A QUARTZ or hard-glass tube, 25 cm. long and not more than 13 mm. in diameter, is fixed in a vertical position and filled with pieces of charcoal of the size of linseed; the upper end of the tube is connected to a gas-holder containing oxygen and the lower end to a reservoir containing water. After the upper part of the tube has been heated with a burner, the tap on the oxygen holder is opened so that water flows from the lower reservoir at the rate of 2 litres per minute. It is important that a reduced pressure be maintained in this way in the tube. The gas collecting in the lower reservoir will contain 85% of carbon monoxide.—W. P. S.

Carbon; Combustion of —. F. C. G. Müller. Z. phys.-chem. Unterr., 1919, 32, 40—45. Chem. Zentr., 1919, 90, IV., 1120.

By burning charcoal in a stream of oxygen a considerable quantity of carbon monoxide is formed in addition to carbon dioxide, the quantity being greater the higher the temperature (see preceding abstract). The laboratory preparation of water-gas in a glass tube can be carried out in a combustion furnace at 800° C.; in three minutes 900 c.c. of gas was obtained having the composition CO, 23.4; CO₂, 10.8; H₂, 5.3; CH₄, 2.1%; and N₂, 4.8%. —B. V. S.

Liquid fuels; Spontaneous ignition-temperatures of —. H. Moore. Inst. Petroleum Tech., Jan. 20, 1920. [Advance proof.]

EXPERIMENTS with industrial liquid fuels in the apparatus previously described (this J., 1917, 109) showed that the results are not substantially affected by alteration in the rate of fuel and air or oxygen supply, by dilution of the oxygen with carbon dioxide, or by using crucibles of different materials, e.g., silica, platinum, nickel, or porcelain. The ignition-temperature in air was generally from 80° to 200° C. higher than in oxygen, the notable exception being turpentine, which yielded the same result in both atmospheres, viz., 275° C. The ignition-temperature of pure hydrogen in air was 686° (porcelain crucible), 619° (nickel do.), and 678° (silica do.) respectively, and the ignition-temperature in oxygen is given as 678°, 680°, and 677° respectively in three determinations. A number of observations were recorded on the ignition-temperatures of binary mixtures from which it was concluded that in a mixture consisting mainly of a liquid of high ignition-temperature, the addition of a small amount of liquid of low ignition-point has very little effect on the ignition-point of the mixture and *vice-versa*. In internal-combustion engines, whether of the two-stroke or four-stroke cycle, and whether operating upon the constant-pressure or constant-volume cycle, the theoretical thermal efficiency is dependent on the compression ratio. The bearing of the above observations on the limiting compression pressure allowed for a specific fuel is discussed.—A. G.

Refractories for coke ovens. W. J. Rees. Gas World, 1920, 72, Coking Sect., 9—11.

BRICKS used in coke ovens seldom attain a higher temperature than 1350° C., even in the heating flues, and the load on them is not sufficient to lower their refractoriness seriously. The corrosion of the bricks appears to be largely due to soluble salts—especially sodium sulphate and chloride—in the coal (see also following abstract). Sodium chloride volatilises at 800° C., hydrolyses at 700° C., and at these or somewhat higher temperatures it attacks the bricks forming a glassy sodium aluminium silicate and making the bricks weak, spongy, and of low resistance to abrasion. The action of hydrochloric acid (from hydrolysed salt) on iron oxide appears to result in the production of layer upon layer of hematite in the bricks; this brings about a different coefficient of expansion in some parts of the bricks and leads to rupture on heating and cooling. Corrosion by salt is more severe in ovens built of fireclay than in those built of silica bricks, partly as a result of direct action and partly due to the fireclay bricks shrinking in use and so causing open joints which expose a greater area to attack. Silica bricks, on the contrary, tend to expand and so cause serious stresses in the structural work of the oven. Suitable natural or artificial mixtures of silica and fireclay produce bricks which neither shrink nor expand. Fireclay bricks burned so as to produce a large percentage of sillimanite may prove

to be highly resistant to the salts in some coals. The catalytic effect of fireclay on hydrocarbons produced during coking with the resultant deposition of carbon in the pores of the brick and the reduction of ferric oxide with the formation of easily fusible ferrous silicate may also assist the disintegration of the bricks. Le Chatelier's work on the use of impalpable silica (this J., 1917, 963) is quoted, and the use of silica rock which gives angular fragments with 25% of impalpable silica powder is recommended. The higher thermal conductivity of (American) silica bricks compared with that of bricks made of or containing much clay is in favour of the use of silica bricks for coke-oven construction.—A. B. S.

Coke oven walls; Corrosion of — W. J. Rees. Trans. Ceram. Soc., 1918-9, 18, 431-434.

THE water used for washing a coal which seriously corroded coke-oven walls was found to contain sodium sulphate as well as sodium chloride, and it is suggested that the former may react with the coal, forming sodium carbonate which is a vigorous corrosive agent.—A. B. S.

Carbon in furnace refuse; Loss due to — C. H. Berry. Power, 1919, 500. Chem. and Met. Eng., 1920, 22, 179.

THE loss of heat due to the presence of carbon in furnace ashes depends upon the percentage of ash in the original coal and the percentage of carbon in the ash. Lb. carbon lost per lb. coal fired =

$$\frac{\% \text{ ash in coal}}{100} \times \frac{\% \text{ C in ash}}{100 - \% \text{ C in ash}}$$

Under fair working conditions not more than 20% of carbon should be present in the furnace ashes, and under good conditions not more than 10%. The corresponding heat losses will then be less than 5% and may be as low as 1%. A series of tests conducted by the U.S. Geological Survey under working conditions showed an average heat loss of nearly 5%. Practical suggestions with a view to reducing the loss include the avoidance of unnecessary raking, an ample supply of air at the dump plate, and the use of clinker grinders.—C. A. K.

Semi-coke obtained by low temperature carbonisation of coal. F. Fischer and W. Glud. Ges. Abhandl. Kenntn. Kohle, 1919, 3, 215-226. Chem. Zentr., 1919, 90, IV.; 880-881.

SEMI-COKE from low-temperature carbonisation ignites readily and burns easily without smoke, but with some flame—mainly of hydrogen. Lohberg coal gave a semi-coke with 15-16% of volatile matter. The specimen tested was not hygroscopic. It contained 1.9% N (on the ash- and moisture-free residue), corresponding to two-thirds of the nitrogen content of the original coal; when heated above 575° C. it gave up one-third of its nitrogen as ammonia. Heated in a current of steam almost the whole of the nitrogen was liberated as ammonia. The sulphur contents of the ash- and moisture-free residues of coal and coke are the same. The oxygen content fell from 8.7 in the coal to 7.5% in the coke, this being in accord with the yield of phenolic substances. The only serious shortcoming of the semi-coke is its friability, but this varies with the class of coal carbonised—some yielding a satisfactory coke (cf. F. Fischer, this J., 1920, 93 A). For use as powdered fuel this should even be an advantage.

—H. J. H.

Vegetable tissues; Extraction of — with dry and moist benzene under pressure. F. Fischer and M. Kleinstück. Ges. Abhandl. Kenntn. Kohle, 1919, 3, 301-314. Chem. Zentr., 1919, 90, IV., 940-941.

EXTRACTION experiments were made with various

kinds of wood, lignite and a similar material from Japan (*Umoregi*), peat, fern, shave grass, straw, pine needles, foliage leaves, and beech bark. Extraction with benzene under pressure at 250° C. gives far higher yields of extract than the ordinary method with a Soxhlet apparatus, e.g., in the case of pine wood 16.2% instead of 0.24%. At lower temperatures the yields are not so great, but they can be increased by the use of another suitable solvent. The extracts obtained under pressure, from wood, are at the ordinary temperature brownish-black shining solids, which can usually be softened by the warmth of the hand. They are soluble in chloroform, acetone, pyridine, acetic acid, and acetic anhydride, less soluble in ethyl and methyl alcohols, ammonia, potassium hydroxide, ether, and carbon tetrachloride, and only slightly soluble in light petroleum spirit. All give a strong lignin reaction and contain manganese. In water they become dull and grey; the aqueous extracts reduce ammoniacal silver solution, usually in the cold, and Fehling's solution on warming. Extracts made under pressure, from the other materials, show a strong green fluorescence in benzene solution. Those from peat, pine needles, straw, and foliage and fern leaves have a waxy appearance and are almost completely soluble in ether and carbon tetrachloride. Extraction with benzene under pressure in presence of water was found in many cases to give higher results than with benzene alone, though the matter extracted was entirely soluble in benzene. In the aqueous portion formic and acetic acids, formaldehyde, and acetaldehyde were detected. By a single extraction with water alone, at 250° C., yields of extract amounting to 83% in the case of poplar wood, and 85% in the case of beech wood were obtained; besides acetone and acids, catechol, furfural, formaldehyde, and acetaldehyde were present in the extracts.—J. H. L.

Lignite; Influence of the moisture-content of — on the yields of extract obtained with benzene in Soxhlet apparatus and under pressure. F. Fischer and W. Schneider. Ges. Abhandl. Kenntn. Kohle, 1919, 3, 315-324. Chem. Zentr., 1919, 90, IV., 935.

IN extracting lignite with benzene in a Soxhlet apparatus higher results are obtained in operating on an air-dry sample containing 10-20% of moisture than with a sample previously dried at 105° C., e.g., 15% of extract in the former case and 11% in the latter. Still greater differences are found in extracting with benzene under pressure at 240° C. The higher yields obtained in presence of moisture are not due to higher contents of water, ash, or humic acids in the extracts.—J. H. L.

Gaseous explosions; Calculation of radiation emitted in — from the pressure-time curves. W. T. David. Phil. Mag., 1920, 39, 66-83.

FROM an examination of a large number of photographic films on which were traced curves of pressure and of radiation emitted in explosions of inflammable mixtures of coal gas and air (this J., 1919, 707 A, 807 A) some simple equations have been established which would seem to apply within fairly wide limits of composition, density, and volume of the mixture. In mixtures containing from 9.2 to 15% of coal gas and at pressures varying from 0.5 to a little over 1.5 atm., the rate at which the walls receive radiation is at a maximum some little time before the attainment of maximum pressure, and this maximum rate is proportional to the square root of the product of the maximum rate of change of the mean gas temperature and the quantity of coal gas present in the explosion vessel measured at atmospheric temperature and pressure. The effect of variation in the size of the explosion vessel and the density of the gas mixture upon the rate and total amount of radiation is examined, and the

rate of emission of radiation during cooling discussed. The conditions governing the emission of radiation in gaseous explosions are exceedingly complex and the formulae set up, though useful as a basis for gas-engine calculations, can only be relied upon to give results correct to within 10–20%.—W. P.

Gaseous explosions; Analysis of the radiation emitted in —. W. T. David. *Phil. Mag.*, 1920, 39, 84–95. (See preceding abstract.)

From an analysis of the radiation emitted during explosion of mixtures of coal-gas and air and hydrogen and air, conclusions are drawn as to the origin of the radiation, and a theory is formulated as to the distribution of energy.—W. P.

Thermal efficiencies of production, distribution, and use of gas and electricity. D. Clerk. *Gas J.*, 1920, 149, 294–296.

A REPLY to criticisms advanced against a previous paper (this J., 1919, 104 B). The figures given were obtained by calculations based on the assumption of the lowest efficiency figure that can be adopted for coal gas generation, the gaseous product of carbonisation being debited with the whole of the heat employed in the carbonisation process.—J. S. G. T.

Viscosity of gases. M. Hofsäss. *J. Gasbeleucht.*, 1919, 62, 776–777.

THE viscosity of complex gas mixtures, such as coal gas, cannot be accurately calculated from their composition. An apparatus is illustrated which may be used for the determination of the viscosities of mixtures of illuminating gas and other gases relative to the viscosity of air by measuring the rate of flow through a fine capillary. Curves are given showing the viscosities relative to air of mixtures of coal gas with from 0 to 100% of hydrogen, methane, carbon dioxide, nitrogen, carbon monoxide, and air. Curves are likewise given showing the viscosities of mixtures in various proportions of methane and air, hydrogen and carbon dioxide, hydrogen and nitrogen, hydrogen and oxygen, and helium and argon, the three latter mixtures at 0° C., the remainder at 15° C. The following table gives the relative density, relative viscosity, and relative viscosity modulus (relative viscosity: relative density) of various gases, in each case relative to air:—

	Illuminat- ing gas.	Carbon mon- oxide. Nitrogen	Carbon dioxide.	Ethy- lene.	Benzene.
Relative density ..	0.595	0.967	1.520	0.967	2.694
Relative viscosity ..	0.738	0.965	0.840	0.574	0.417
Relative viscosity modulus ..	1.240	1.00	0.553	0.593	0.155

The viscosity of a gas affords valuable information concerning the purity of the gas.—J. S. G. T.

Coal-gas; Influence of carburetting of — on the crystallisation of naphthalene in the mains. C. Ab-der-Halden. *Chim. et Ind.*, 1920, 3, 19–23.

To determine the influence of carburetting agents on the amount of naphthalene retained in the gas at various temperatures, coal gas, saturated with naphthalene at a definite temperature, was carburetted with known amounts of various carburetting agents derived from petroleum or coal-tar and then cooled until naphthalene was deposited. It was found that after the addition of the carburetting agent the gas could be cooled several degrees lower without depositing naphthalene than before such addition. The power of

holding naphthalene in "supersaturation" in the gas depended on the boiling point of the carburetting agent rather than on its constitution, and the degree of extra cooling to which the carburetted gas could be submitted without depositing naphthalene was dependent on the amount of carburetting agent added and on the degree of saturation of the gas with naphthalene.—W. H. C.

Purification; Oxide — [of coal gas; analysis of spent oxide]. G. Weyman. *Gas J.*, 1920, 149, 301.

FERRIC and ferrous sulphides are produced by the action of hydrogen sulphide upon ferric hydroxide, and the free sulphur produced by the interaction is usually taken as a criterion of the amount of ferrous sulphide formed. The sulphur content of the purifying mass may, however, be affected by many other factors than the amount of ferrous sulphide formed. The author, adopting a suggestion by L. T. Wright, determines the ratio of sulphur to iron in the mass by decomposing the sulphides with copper sulphate solution. The sulphide is freed from hydrogen sulphide by a current of hydrogen and digested for 10 minutes at 30°–40° C. with an excess of copper sulphate solution, then rapidly filtered and washed, the filter paper being kept full of liquid. The wet copper sulphide is oxidised in the original beaker with concentrated nitric acid. The solution is diluted and excess of ammonia added. The precipitate is purified by re-solution and precipitation, the washings being added to the main bulk, which is then acidified with nitric acid and a little sulphuric acid, made up to a known volume, and a portion taken for electrolysis. The filtrate from the copper sulphide is made strongly alkaline and filtered. The precipitate is purified by re-solution and re-precipitation, dissolved in sulphuric acid, diluted to a known volume, and portions taken for reduction and titration with permanganate, the reduction being best effected with a modified Jones reductor.

—J. S. G. T.

Matita asphalt; Products obtained by the dry distillation of —. C. Nicolescu-Otin. *Bull. Acad. Roumaine*, 1916–7, 5 [3], 129–138.

MATITA asphalt was distilled in stoneware or iron stills at 730° C. Compared with petroleum, the lower fractions are much less in quantity, indicating that the simple hydrocarbons of petroleum have undergone extensive polymerisation by atmospheric action aided by the exposure of a large surface due to the percolation of the petroleum in the sandy clay-beds. The tar-fractions also have saponification values much higher than those of petroleum fractions, and the presence of cyclic hydrocarbons in the tar in such striking quantities compared with the petroleum fractions indicates the change from petroleum to bitumen, the action of pressure, temperature, oxygen, and specially of time converting the saturated and unsaturated acyclic hydrocarbons into aromatic compounds. The tar products are characterised by large contents of sulphur and nitrogen. Matita asphalt gives, on distillation, a combustible gas which approaches to that from lignite in quantity and quality. About 10 kilos. of ammonium sulphate per ton of bitumen is obtained, the asphalt containing 29% of bitumen. The coke may possibly be utilised for refining petroleum in place of fuller's earth. The quantity of tar obtained is at least 120 kilos. per ton of rock asphalt, or 450 kilos. per ton of bitumen; it contains a high proportion of aromatic compounds as well as a large amount of paraffin (30% in the fraction 200°–275° C.). The asphalt could be used as a source of paraffin, vaseline, and mineral oils of superior quality.—A. G.

Paraffin wax; Technique of crystallisation in the manufacture of —. K. Fuchs. *Petroleum* (Berlin), 1919, 14, 1281—1285. *Chem. Zentr.*, 1919, 90, IV., 938—939.

PARAFFIN WAX may be made to crystallise from any (petroleum) oil or intermediate product, however viscous, if the wax-content is increased sufficiently. The lighter paraffins give the largest crystals, and require only a relatively low degree of supersaturation for crystallisation to take place. With increase of viscosity the size of the crystals diminishes (cp. Bergel, this J., 1919, 278 A). Very satisfactory crystallisation can be obtained from oil mixtures having a viscosity of 1.17°—1.8° Engler at 50° C., a setting point of 27°—30° C., sp. gr. 0.87—0.88, and a wax-content of 20—25%. The cake (*Gatsch*) of separated paraffin should be fairly free from oil, show a well-marked crystalline texture, and yield a pressed scale with a setting point of 45°—48° C. for a wax-content of 70—80%. For a satisfactory sweating process the wax-content of the scale should not be too high, and the crystals should be large in order to provide interstices for drainage. The drainings from the sweating chambers are preferably divided into three fractions: the first, of setting-point up to 32° C., is worked up again for paraffin; the second, of setting-point 32°—38°, is filtered warm together with paraffin cake (*Gatsch*), and the third, of setting-point 38° or above, is again sweated alone or together with pressed scale.—J. H. L.

Petroleum products; [Laboratory] distillation of —. F. Bordas. *Ann. Falsif.*, 1919, 12, 346—351.

A MODIFICATION of the Regnault-de Luynes apparatus is described; the condensing system is not changed, and for petroleum spirit (petrol) the original form of still is retained. For lighting oils, the de Luynes still is provided with an opening 2 cm. above the usual side tube to facilitate the introduction of thick oils and the cleaning of the apparatus; a number of perforated plates are fitted inside the still. The latter is surrounded by a jacket, the lower part of which contains a gas burner fitted with a regulating tap; the top of the burner is provided with a metallic gauze cap and a metal plate and wire gauze are placed between the burner and the bottom of the still.—W. P. S.

Montan wax; Determination of the saponification value of —. H. Salvaterra. *Chem.-Zeit.*, 1920, 44, 129.

FOUR grms. of the sample is heated under a reflux condenser for about 5 hours with 30 c.c. of *N*/2 alcoholic potassium hydroxide solution and 20 c.c. of xylene; 25 c.c. of 6.1% barium chloride solution and 250 c.c. of boiled water are then added, the mixture heated for a further one hour on a water-bath, cooled, diluted to 520 c.c., and a quantity of the aqueous layer is drawn off, filtered, and 100 c.c. of the filtrate titrated with *N*/2 hydrochloric acid, using phenolphthalein as indicator. The end-point is quite sharp, which is not the case when it is attempted to titrate the dark-coloured saponification mixture directly.—W. P. S.

Paraffin; Oxidation of —. *Oxidation of hydrocarbons by oxygen.* C. Kelber. *Ber.*, 1920, 53, [B], 66—71.

PARAFFIN, vaseline, mineral oil distillates, and the higher fatty acids may be oxidised, rapidly and almost completely, by means of a well-divided current of oxygen at 140°—150° C., especially in the presence of insoluble manganese compounds. A preliminary example is recorded. Paraffin wax of m.pt. 50°—51° C. gave 25% of a distillate containing fatty acids of the series C_1 to C_{10} , 40—50% of a residue of higher fatty acids, and 5—10% of hydroxy-fatty acids.—J. C. W.

Light petroleum and paraffin from coal. Fischer and Glud. *See* III.

PATENTS.

Pulverised fuel; Apparatus for feeding —. P. Meider, jun., Pittsburgh, Pa. U.S. Pat. 1,328,750, 20.1.20. Appl., 23.6.19.

THE pulverised fuel and the air supply pass from a main pipe into a short pipe connected therewith. Secondary air is introduced under pressure through a pipe surrounding the short pipe and extending to the furnace.—W. P.

Coal; Flotation of —. R. F. Bacon, Pittsburgh, Pa., Assignor to Metals Recovery Co., New York. U.S. Pat. 1,329,493, 3.2.20. Appl., 27.2.17.

BITUMINOUS coal is ground to give a finely divided product with particles having sharp angular edges and bright surfaces, and is then subjected to a froth flotation separation process.—W. P.

Solidified fuel. J. M. Kessler, West Orange, Assignor to The Arlington Co., Arlington, N.J. U.S. Pat. 1,329,610, 3.2.20. Appl., 28.10.16.

A SOLIDIFIED liquid fuel composition contains nitro-cellulose, a metallic powder containing copper (e.g., bronze powder), and alcohol.—W. P.

Chamber ovens; Regenerative —. A. Klönne, Dortmund. *Ger. Pat.* 314,803, 30.1.18.

THE lower portion of the oven is designed similar to the usual horizontal retort furnaces, while in the upper portion separate sources of heat, which can be controlled independently, are arranged for each chamber, and may lie in the longitudinal axis behind each other and side by side. Separate horizontal air, combustion, and gas collecting channels are provided between the upper and the lower portions in an intermediate part of the oven, which possesses inclined coke-discharging surfaces. Separate reserve heating gas chambers, which can also be controlled independently, are arranged between the chambers of the oven and the heating cells. Since each chamber is built in a special self-contained heating cell, it may be put into or out of work, according to requirements, without endangering the work of the whole oven. In consequence of the arrangement of the air, combustion, and gas collecting channels there is always a sufficient supply of heated air and a supply of heating gas for the heating of the chambers, so that a uniform temperature is maintained.—J. F. B.

Gas producers. J. West, Southport, and W. Wild, Blackpool. *Eng. Pat.* 137,647, 18.2.19. (Appl. 4010/19.)

THE fire-grate is vertical or inclined, and is formed wholly or partly of water tubes attached to a supply header at their lower end, and delivering into a header above the grate. The steam generated is delivered into the ash-pit, whence it is drawn into the furnace with the air supply. Cooling is thus effected without the necessity for a stream of water over the grate. Clunkering of the grate is required much less frequently, resulting in a more regular and constant production of gas and an economy of labour. The header above the grate, one side of which is heated by the furnace, contains water maintained at a constant level.—W. P.

Gas producers; Working of —. W. Beswick and N. E. Rambush, Stockton-on-Tees. *Eng. Pat.* 138,003, 24.5.19. (Appl. 13,123/19.)

WATER is circulated through parts of the producer which require to be cooled, and after being heated in this manner is brought into contact, in a separate chamber, with air which is to be supplied to the producer. The air is heated and more or less saturated with water vapour, and all the necessary

steam can be obtained from the water in circulation without the latter being heated to the boiling point. A circulating pump draws water from the bottom of a tower packed with coke or the like, and delivers it to the water jacket of the producer and to the water-cooled poker. The water is then returned to the top of the tower and is distributed over the packing, meeting the air which is to be supplied to the producer. A portion of the water may be withdrawn and replaced by fresh water. —W. P.

Gas for cutting and welding purposes. J. Harris, Lakewood, Ohio, Assignor to J. R. Rose, Pittsburgh, Pa. U.S. Pat. 1,329,400, 3.2.20. Appl., 3.8.18.

A mixture of hydrogen and carbon monoxide only is used, the carbon monoxide being not less than 5% or more than 20%, by volume, of the hydrogen. —W. P.

Gas; Manufacture of —. W. F. Rittman, Assignor to Synthetic Hydrocarbon Co., Pittsburgh, Pa. U.S. Pat. 1,329,853, 3.2.20. Appl., 23.2.16. Renewed 27.5.19.

Air and steam are forced through a body of incandescent carbonaceous material, the heated gases so produced being utilised to crack hydrocarbon fluids whilst out of direct contact with them. The heated gases are then mixed with the gases produced by the cracking process. —W. P.

Gas for balloons. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 303,966, 8.9.16.

Gas for filling balloons is treated with a small quantity, e.g. up to 3%, of ammonia or other volatile base such as zinc- or dimethylamine or piperidine, to prevent deterioration of the caoutchouc in the envelope. —J. H. L.

Blast-furnace and the like gases; Cleaning of —. J. E. Weyman, Newcastle-on-Tyne. Eng. Pat. 137,586, 10.1.19. (Appl. 781/19.)

A shaft running centrally through a drum carries arms threaded thereon loosely in pairs, each pair of arms being coupled by a framework filling more or less half the diameter of the drum. The filtering material or collecting surfaces are secured to this framework. The revolving arms carry the collecting surfaces at regular intervals up one side of the vessel and allow them to fall by gravity down the other side of the drum. The arms are lifted by means of slots in the drum casing, which revolves with the shaft. Spring arms or levers in the casing enable the arms to fall with a minimum of shock, though with sufficient to shake off the dust, which collects in a hopper at the bottom. The number and spacing of the arms are such that there is always one set between the inlet and outlet for the gas in the top half of the drum, and two or more between the gas inlet and outlet in the bottom half. —W. P.

By-products from coal; Process for recovery of —. Generator A.-G., Charlottenburg. Ger. Pat. 310,121, 10.12.16.

The coal is heated in a producer to which a distillation shaft is connected. The distillation gas, after condensation, is mixed with the producer gas, and the mixture washed with ammonia liquor and sulphurous acid obtained from recovered sulphur. The object is to obtain the gas tar and oils as direct products, and to recover ammonia and sulphur simultaneously in the form of ammonium sulphate and sulphur. By the action of the hydrogen sulphide in the gas on sulphur dioxide, in presence of ammonia, a solution of ammonium sulphate is obtained, which is used to absorb sulphur dioxide and so return it to the washer as an acid liquor.

The excess of acid liquor is evaporated to give ammonium sulphate, free sulphur, and sulphur dioxide. —H. J. H.

Paraffin; Process and apparatus for producing cakes of solid crude —. A. Szekely, Vienna. Eng. Pat. 131,293, 11.8.19. (Appl. 19,727/19.) Int. Conv., 30.5.18.

The crude solid paraffin residue ("gatsch") is melted and poured into a vessel with perforated walls partially immersed in water. The perforated walls above the water level are protected by a covering until the gatsch has solidified, after which the covering is removed and sweating is accomplished *in situ*. Sieve-like tubes, extending through the containing vessel and protected in a similar way during the setting of the gatsch, may also be provided. —A. E. D.

Hydrocarbons; Process of treating —. W. M. Cross, Kansas City, Mo., Assignor to Gasoline Products Co., Inc. U.S. Pat. 1,326,851, 30.12.19. Appl., 1.6.15.

GASOLINE is manufactured from heavier hydrocarbons such as residuum and the like by subjecting the vaporised hydrocarbons to a superheating treatment and preventing the deposition of carbon in the apparatus by injecting into the heated vapour a quantity of air less than is sufficient to cause complete combustion of the carbon in the apparatus. —J. F. B.

Oil-cracking stills; Furnace for —. F. E. Wellman, Assignor to Kansas Gasoline Co., Kansas City, Kans. U.S. Pat. 1,328,468, 20.1.20. Appl., 19.1.17.

A HEATING chamber is connected with the source of heat by a number of ducts, each provided with a damper. Each damper is adjustable, and all the dampers may be regulated by a common operation. —A. E. D.

Oil refining [cracking] apparatus. A. R. Jones, Independence, Kans. U.S. Pat. 1,328,522, 20.1.20. Appl., 9.10.16.

OIL is introduced through a feed pipe into a vertical pipe forming part of a tubular still, wherein cracking takes place. The vertical pipe is connected at its upper end with an approximately horizontal tube-system, and at the lower end with a catch-pot for carbon particles. —A. E. D.

Gasoline; Apparatus for recovery of — from casing-head gas. W. R. McGinnis, Assignor to Pilsbry-Becker Engineering and Supply Co., St. Louis, Mo. U.S. Pat. 1,328,680, 20.1.20. Appl., 14.5.17.

THE gas is compressed, cooled, and passed through a primary and a secondary separator, wherein free gas and gasoline are produced. —A. E. D.

Hydrocarbon; Apparatus for cracking —. T. E. Valey, Jamestown, N.Y. U.S. Pat. 1,329,450, 3.2.20. Appl., 19.4.17.

A CRACKING retort is provided with a nozzle which is connected by automatic non-return valves to the feed tank and to the primary condenser, wherein uncracked oil is collected. —A. E. D.

Carbon - removing means [in cracking hydrocarbons]. W. F. Rittman and C. B. Dutton, Pittsburgh, Pa. U.S. Pat. 1,329,852, 3.2.20. Appl., 5.2.17.

A rod extends into a tubular retort for cracking hydrocarbons practically the whole length. The rod is furnished with scrapers, and is given a reciprocating and oscillating motion. —W. P.

Hydrocarbons; Treatment [cracking] of —. W. F. Rittman, Pittsburgh, Pa. U.S. Pat. 1,330,008, 3.2.20. Appl., 5.2.17.

HIGH-BOILING oils in the vapour phase are exposed to cracking conditions, and the product is discharged into a receptacle, wherein, at low pressure, the desired low-boiling oil is vaporised and is subsequently condensed.—A. E. D.

Oil-bearing shale; Apparatus for distilling —. H. R. Straight, Adel, Iowa. U.S. Pat. 1,330,014, 3.2.20. Appl., 31.7.18.

AN internally-heated rotating retort is provided at the upper end with a hopper and at the lower end with a discharge valve. The retort is heated by a nozzle burner, which projects into the lower end of the chamber in the axis of rotation and which is fed with air and fuel.—A. E. D.

Petroleum spirit [benzine]; Process for breaking down high-boiling hydrocarbons into — and simultaneous conversion of unsaturated into saturated hydrocarbons. F. Bergius, Hanover, and A.-G. für Petroleum-Industrie, Nürnberg. Ger. Pat. 304,348, 6.5.13.

BENZINE is produced by heating high-boiling hydrocarbons in presence of hydrogen without catalysts, the hydrogen being under a pressure of at least 20 atm. The temperature necessary is about 450° C., but varies with the raw material. The products consist almost entirely of saturated compounds; they are colourless and have only a faint odour.

—J. H. L.

Lubricating oils; Process for purifying used —. W. v. Sperl, Hof. Ger. Pat. 314,175, 19.7.18.

FOUR parts of glycerin pitch and 1 part of water are boiled together to a syrupy consistence. One part of this mixture is boiled with 20 parts of the used lubricating oil and at least two parts of water. On standing clean oil separates.—H. J. H.

Coke-retorts; Apparatus for extracting coke from —. A. J. Nerrière, St. Brieux, France. Eng. Pat. 124,719, 24.2.19. (Appl. 4604/19.) Int. Conv., 28.3.18.

Combustion in flues. Eng. Pat. 137,770. See I.

Centrifugal separator for crude oil. U.S. Pat. 1,307,000. See I.

Hydrocarbon filters. U.S. Pats. 1,328,044—6. See I.

Hydrogen. Eng. Pat. 137,340. See VII.

Hydrogen. Eng. Pat. 137,674. See VII.

Road-material. Eng. Pat. 138,017. See IX.

Gas analysing apparatus. U.S. Pat. 1,311,952. See XXII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Vegetable tissues. Fischer and Kleinstück. See IIa.

Gas and electricity. Clerk. See IIa.

Cellulose. Fischer and Schneider. See V.

PATENTS.

Coal; Low temperature destructive distillation of —. J. Swinburne, London. Eng. Pat. 137,572, 4.1.19. (Appl. 261/19.)

COAL is distilled at 200°—350° C. by means of metallic electrical resistance heaters which sub-

divide a charge in a retort. The distillation is continued for upwards of 20 hours, and the oil produced is distilled substantially without cracking. The distillate resembles crude petroleum, and a rich gas is also produced.—W. F. F.

Acetone; Manufacture of — by the dry distillation of pyrolignite of lime and other acetates. L. Rappaport, Paris. Eng. Pat. 137,558, 19.11.18. (Appl. 18,984/18.)

PYROLIGNITE of lime containing about 75% of calcium acetate is distilled on a number of superposed trays of refractory material such as porcelain or sandstone, the lowest tray being supported on rollers of similar material. Each tray is provided with side openings for the escape of gas, and the furnace and supports are so constructed that the pyrolignite and the acetone formed do not come into contact with any metal. A higher yield of acetone is thus obtained.—W. F. F.

III.—TAR AND TAR PRODUCTS.

Liquites; Production of low temperature tar from — and its use as a source of viscous oils. W. Schneider. Ges. Abhandl. Kenntn. Kohle, 1919, 2, 80—127. Chem. Zentr., 1919, 90, IV., 935—937.

Low-temperature tars were prepared from two lignites, viz., a bituminous Central German lignite from the Riebeck Montan works, containing 20—30% of moisture and 15—16% of bitumen, and a non-bituminous Rhenish lignite (Union Briquettes) containing 15% of moisture and 3—4% of bitumen, by distilling the lignites, with superheated steam, at a maximum temperature of 450° C. in apparatus similar to that used by Fischer and Glund (see this J., 1919, 492 A). The tars differed physically from corresponding products from pit coal in their consistence (cp. Fischer and Glund, this J., 1919, 942 A) and setting point (37° and 33° C. for the two lignite tars). Their specific gravities at 50° C. were 0.883—0.890 and 0.955 respectively. They were subjected to fractional distillation in superheated steam, and their composition is shown in the accompanying table:—

Low-temperature tar from 100 kilos. of lignite (dry substance).	Bituminous lignite, 24 kilos. of tar (16% creosote)	Non-bituminous lignite, 7.6 kilos. of tar (36-38% creosote)
Viscous oils (freed from phenols) ..	17.2%	14.8%
Viscosity at 20° C., ° Engler ..	13.5	18.0
" " 50° C. ..	2.9	3.6
Comprising fractions:—		
(a) of high viscosity	1.73% (V ²⁰ = 67° E.)	2.8% (V ²⁰ = 7.4° E.)
(b) of medium	13.01% (V ²⁰ = 11.4° E.)	8.9% (V ²⁰ = 8.5° E.)
(c) of low	2.47% (V ²⁰ = 5.4° E.)	3.1% (V ²⁰ = 7.5° E.)
Hard and soft paraffins (montan wax, etc.)	29.4%	13.0%
Non-viscous oils	28.9%	26.2%
Acid fraction	10.5%	24.7%
Resin	2.2%	2.3%
Pitch	3.2%	8.5%

The viscous oils in low-temperature tar from lignite are not present in the original lignite, for extraction of the latter yields only solid bitumen. In pit coal, on the other hand, viscous oils are present before distillation and pass over undecomposed into the low temperature tar.—J. H. L.

Lignite; A technically produced low-temperature tar from —. W. Schneider and H. Tropsch. Ges. Abhandl. Kenntn. Kohle, 1919, 2, 128—132. Chem. Zentr., 1919, 90, IV., 937.

A TAR from a Central German lignite, obtained in

a producer with separate ducts for the distillation gases and tar vapours on the one hand and the producer gas on the other (*cp.* Fischer and Glud, this J., 1919, 492 A), contained only 3.7% of water, and less than 0.1% of matter insoluble in hot benzene. Subjected to fractional distillation with superheated steam it gave similar results to those obtained with low-temperature tars from lignite (see preceding abstract); the yield of viscous oils possessing lubricating characters was about 18%.

—J. H. L.

Lignites; Behaviour of low-temperature tar from bituminous and non-bituminous — when distilled under ordinary pressure. W. Schneider. Ges. Abhandl. Kenntn. der Kohle, 1919, 2, 133—144. Chem. Zentr., 1919, 90, IV., 937.

Low-temperature tar from a Central German bituminous lignite, having a setting point of 38° C., was distilled at the ordinary pressure under different conditions, *viz.*, from a retort in which the vapours escaped rapidly from the heated space; rapidly from an iron still in which the escaping vapours would be more exposed to heat than in the preceding case; and very slowly from the same still. The setting points of the distillates in the three cases were 24°—25°, 15°, and 0° C. respectively. Substantially similar results were obtained with a low-temperature tar from Rhenish lignite. The breakdown of high-boiling constituents into lower-boiling products could probably be carried even further, but there is a possibility that with very slow distillation condensation of unsaturated compounds to high-molecular substances might also occur.—J. H. L.

Lignite tar oils; Production of viscous oils from non-viscous — by heating in an autoclave. W. Schneider. Ges. Abhandl. Kenntn. der Kohle, 1919, 2, 145—150. Chem. Zentr., 1919, 90, IV., 940.

The high-boiling fractions (above 250° C.) obtained by a single distillation of low-temperature lignite tar can be rendered more viscous by heating for several hours at 300° C. in an autoclave, and their bromine absorption value is diminished at the same time. The thickened oil still contains unsaturated compounds even after heating for 34 hours. The change is due not to oxidation processes, but probably to polymerisation.—J. H. L.

Coal-tar and its constituents; Thickening of low-temperature —. F. Fischer, W. Glud, and P. K. Breuer. Ges. Abhandl. Kenntn. Kohle, 1919, 2, 222—235. Chem. Zentr., 1919, 90, IV., 934—935. (See also this J., 1919, 492 A, 942 A.)

Low-temperature tar and various fractions thereof, *e.g.*, the fractions boiling below and above 300° C., the phenolic constituents, and the tar deprived of phenolic substances, were heated under pressure in contact with "steel wool" in an autoclave at 320° C. for two successive periods of 3 days each. After this treatment the tar itself, and the hydrocarbons contained in it, showed a higher viscosity and density and a higher setting point than before. The phenolic constituents thickened more rapidly and to a greater extent than the hydrocarbons, giving products resembling pitch or asphalt. It is possible to transform the hydrocarbons of low-temperature tar into viscous products having the characters of true lubricating oils. Where phenols are also present the asphalt-like substances formed remain dissolved in the oils, but in the event of this condensation of the phenolic constituents being incomplete there would remain a possibility of further thickening taking place if the products were employed as lubricants at high temperatures. To produce heat-stable lubricants it would therefore appear advisable to remove the phenols in spite of the highly viscous products to which they give rise.—J. H. L.

Light petroleum and paraffin from coal; Production of —. Low-temperature tar. F. Fischer and W. Glud. Ges. Abhandl. Kenntn. Kohle, 1919, 2, 295—342. Chem. Zentr., 1919, 90, IV., 881—882. (See this J., 1919, 561 A.)

The light fraction of the tar oil from Minden coal can be obtained almost without attendant evolution of gas by distilling the coal at 240°—260° C. It is a clear green fluorescent oil of sp. gr. 0.763 at 20° C., and bromine-absorption value 4.4, and the yield is 0.35% on the coal. The loss on acid and alkali washing is extremely small. 65 c.c. of the product gave 20 c.c. of colourless spirit at 40°—100° C., sp. gr. 0.700, bromine value 2.5, and 36 c.c. of a fraction 100°—190° C., sp. gr. 0.783, and bromine value 6.8. It consists of paraffins mixed with naphthenes and compounds poorer in hydrogen. The small bromine values show that the products differ from those of low-temperature carbonisation carried to completion. As the Minden coal belongs to one of the newer formations, the existence of these liquid hydrocarbons is characteristic of the earlier stages of coal formation. The oil here described resembles closely another which had exuded from a coal seam. The essential difference between the low-temperature tars from coal and from lignite is that the former are liquid and the latter semi-solid. Coal contains liquid paraffins ready formed, while by extraction only solid products are obtained from lignite, and these appear also in its low-temperature tar. The low-temperature tars from gas coal and Saxon lignite on redistillation show a progressive fall in viscosity and setting point. In the practical recovery of such tars from gas producers it must therefore be possible to prepare tar of any desired viscosity and setting point. By working at temperatures below 600° C. and condensing all constituents liquid at the ordinary temperature, the tars which are least decomposed are those with the highest setting point. In these tars no great proportion of pre-formed lubricating oils is present, but transformation products, the result of heating in presence or absence of catalysts, have the character of lubricating oils. Jones (this J., 1917, 3) states that naphthalene appears in tars only above 750° C., so that its presence in tar is proof that the latter is not a true low-temperature product.—H. J. H.

Lamp oil fraction of low-temperature coal tar; Liquid paraffins of —. F. Fischer and W. Glud. Ges. Abhandl. Kenntn. Kohle, 1918, 3, 39—45. Chem. Zentr., 1919, 90, IV., 882—883.

The fraction 200°—300° C. of a low-temperature tar from a gas coal (Lohberg) was treated with acetone to remove acid and basic components and cooled to -50° to -70° C., whereby a mixture of paraffins was separated which, at ordinary temperature, was a liquid, light pink in colour, with a blue fluorescence. The product was still impure and showed greater optical activity than the original oil. Further purification with concentrated and fuming sulphuric acid gave a colourless oil boiling from 150° to 336° C., which was apparently composed of paraffins in the range $C_{10}H_{22}$ to $C_{15}H_{32}$. The yield was at least 7.7% of the fraction 200°—300° C., and 1 to 1.5% of the original tar. The fraction 200°—300° C. of a low-temperature tar produced on the large scale, purified by concentrated and fuming sulphuric acid, gave 9.4% of liquid paraffins (1—1.5% on the original tar).—H. J. H.

Fuel oil and lamp oil (solar oil) from coal; Production of —. W. Glud. Ges. Abhandl. Kenntn. Kohle, 1918, 3, 46—65. Chem. Zentr., 1919, 90, IV., 883.

The fuel and lamp oils obtainable from coal were investigated by distilling up to 300° C. the low-

temperature tar prepared from a gas coal (Lohberg), lignite tar from Riebeck'sche Montanwerke, and an ordinary gas tar. The phenols were removed, and after a double washing the residue was distilled and collected in fractions. Lamp oils of the range 150° – 310° C. were obtained in yields as follows:—Low-temperature tar, 18.8%; lignite tar, 30.2%; gas tar, 16.2%. The yields of crude products up to 300° C. were 33.9%, 47%, and 25% respectively. Corresponding fractions of the low-temperature tar and the lignite tar seemed to be identical, especially the solar oil fractions, which agreed in colour, specific gravity, ultimate analysis, calorific value, viscosity, etc. The solar oil fraction of the coal tar, with sulphur content 0.17%, seems to be the purest fraction and probably a mixture of paraffins, naphthenes, olefines, and higher aromatic hydrocarbons. The oils from gas tar were very different in composition, calorific value, and specific gravity. The solar oil fraction of low-temperature tar is distinguished by a small content of unsaturated compounds.—H. J. H.

Low temperature tar from lignite; Characteristic difference between — and other tars. F. Fischer and W. Schneider. Ges. Abhandl. Kenntn. Kohle, 1918, 3, 200–212. Chem. Zentr., 1919, 90, IV., 883–884.

Low temperature lignite tar does not differ greatly in composition from the product obtained in the normal process, whereas low-temperature coal tar differs materially from gas tar. The differences with the lignite tars are quantitative rather than qualitative. It is not yet possible to lay down general characteristics of the tars from different lignites. Contrary to the experience with coal tars (this J., 1919, 941 A) the specific gravity of the tar after removal of phenols is not characteristic; the same is true of the residue on distillation to 300° C. A rapid test may be made as follows:—50 grms. of tar is dissolved in 100 c.c. of hot benzene, filtered hot, and washed with hot benzene. The benzene is distilled off and 20 grms. of the residue is stirred with 20 c.c. of light petroleum spirit (b. pt. 35° – 65° C.), filtered through a tared filter, washed with 20 c.c. of the same solvent, dried at 100° C., and weighed. Low-temperature tars from various lignites gave more than 30% of product insoluble in petroleum spirit. Other lignite tars gave less than 10%. The presence of naphthalene in a lignite tar shows that it is not a low-temperature product. Lignite tars are distinguished from coal tars by their consistency (Fischer and Gluud, preceding) and methoxyl content (Fischer and Tropsch, this J., 1919, 523 A).—H. J. H.

Nitro-compounds; Reduction of aromatic — by stannous chloride and by titanous chloride, and their volumetric estimation. D. Florentin and H. Vandenberghé. Bull. Soc. Chim., 1920, 27, 158–166.

WHILST the method described by Druce (this J., 1919, 247 A), using stannous chloride as the reducing agent, gives satisfactory results with most aromatic nitro-compounds, the authors find that with the three mononitrotoluenes, in particular *o*-nitrotoluene, the results obtained are too low. They recommend the use of titanous chloride in place of stannous chloride, the excess titanous chloride being titrated into a known volume of a standard ferric salt solution, using ammonium thiocyanate as an indicator. The results are accurate with all the nitro-compounds examined, except *o*-nitrotoluene, which gives results 3% too low.

—W. G.

Reactions of calcium hydride. Reich and Serpek. See VII.

Electrochemical oxidation of benzaldehyde and benzoic acid. Fichter and Uhl. See XX.

IV.—COLOURING MATTERS AND DYES.

2-Nitro-4-cyanobenzaldehyde and 6,6'-dicyanoindigo. S. Reich and E. Lenz. Helv. Chim. Acta, 1920, 3, 144–157.

2-NITRO-*p*-TOLUNITRILE, colourless shining needles, m.pt. 107° C., is prepared from 2-nitro-*p*-toluidine by a modification of the method of Niementowski and Noyes, and is converted by *p*-nitrosomethyl-aniline in alcoholic solution in the presence of sodium carbonate into *p*-dimethylaminophenyl-2-nitro-4-cyanophenylazomethine,

$N(CH_3)_2 \cdot C_6H_4 \cdot N : CH \cdot C_6H_3(CN)(NO_2)$, almost black needles, m. pt. 182.5° – 183° C. (decomp.), which is hydrolysed by hydrochloric acid, yielding 2-nitro-4-cyanobenzaldehyde, red needles, m.pt. 110° C. When the aldehyde is dissolved in acetone and treated with dilute sodium hydroxide solution, 6,6'-dicyanoindigo is deposited as a bluish-violet powder with metallic lustre; when heated it does not sublime but decomposes with evolution of reddish-violet vapours. After reduction with sodium hydrosulphite it dyes wool and cotton from an alkaline bath in reddish-purple shades which become blue with a tinge of violet on exposure to air, oxidation proceeding more slowly than with indigo. The shades are fast to light and milling. (See further J. Chem. Soc., 1920, i., 255.)—H. W.

Aniline; Oxidation of —. Amine oxidation. I. S. Goldschmidt. Ber., 1920, 53, [B], 28–44.

THE author holds the view that there are two initial products of the oxidation of aniline, according to the conditions. Agents which readily yield atomic oxygen, such as Caro's acid or hydrogen peroxide, produce aniline oxide, which changes into phenylhydroxylamine and then gives nitrosobenzene, nitrobenzene, azobenzene, and azoxybenzene. Aniline oxide or phenylhydroxylamine cannot account, however, for the production of benzoquinonephenyldi-imine, $C_6H_5 \cdot N : C_6H_4 : NH$, and subsequently of Emeraldine, Aniline Black, etc. Under conditions which lead to these products the initial stage is represented by the hypothetical radicle, $C_6H_5 \cdot N :$. This may, for example, polymerise to azobenzene or benzoquinonephenyldi-imine, the first demonstrable products of the oxidation of aniline by alkaline permanganate or bleaching powder, or polymerise to Emeraldine. The assumption is based on the fact that azo-compounds and quinonearyldi-imines can be isolated when aniline, and especially xylidine, eunidine, or aminodurene are oxidised by lead peroxide, and is considerably strengthened by the fact that mixed azo-compounds and mixed quinonearyldi-imines are produced when two amines are oxidised together. (See further, J. Chem. Soc., 1920, i., 226.)

—J. C. W.

Quinone-imide dyes. XII. Absorption spectra of some cyano-acridine and cyanopyrroline dyes. F. Kehrman and M. Sandoz. Ber., 1920, 53, [B], 63–66. (Compare this J., 1918, 364 A.)

ABSORPTION curves for three sets of comparable dyes are reproduced, showing that the replacement of the trivalent nitrogen atom in safranines and azoxines by the group $C \equiv N$, has practically no effect on the colour. (See further, J. Chem. Soc., 1920, ii., 142.)—J. C. W.

Coloured substances; Determination of the constitution of — from their absorption spectra. II. F. Kehrman and M. Sandoz. Helv. Chim. Acta, 1920, 3, 104–114.

ALCOHOLIC solutions of phenazine and aminophenazine are almost colourless and orange respectively, whilst similar solutions of sym. and 2,3-diaminophenazine are pale yellow and very

light yellow; this is in accordance with the author's theory that the introduction of an amino group in the *para*-position to the nitrogen of quinonimines or in the same position with respect to the methane carbon atom of triphenylmethane has a hypsochromic or bathochromic influence which depends on the basicity of the group into which it enters. The introduction of the first amino group into phenazine considerably increases its basicity, but this is not the case with the second group. Phenylated derivatives of phenazine are slightly more reddish than the corresponding non-phenylated compounds, but are chiefly noticeable for the increased intensity of colour effected by introduction of the phenyl-group. In general, the absorption curves of the bases do not present any striking anomalies, and the authors therefore attribute to them the ortho-quinonoid structure of phenazine itself. Mono- and di-aminophenazines when dissolved in fuming sulphuric acid have very similar absorption spectra, and appear to have the orthoquinonoid structure. When, however, the solutions are gradually diluted with concentrated sulphuric acid, the blood-red colour suddenly changes to green, probably owing to hydrolysis. The authors ascribe the para-quinonoid constitution to the di-acid and mono-acid salts as well as to the green tri-acid compounds. The ultra-violet spectra of the bases and of the mono-acid salts have been examined. With the former, an absorption band is invariably observed which becomes progressively displaced towards the visible violet as the chromogen becomes more highly substituted. The location of the anilino-groups, which has such a marked influence on the position of the bands in the visible spectrum, appears to have no effect on the position of the band in the ultra-violet. Isomeric substances with equal molecular weights appear to give superposable spectra in the ultra-violet.

—H. W.

Ortho-quinones; Action of o-aminothiophenol on —. I. Naphtho-6-phenothiazine. K. Stahr-foss. *Helv. Chim. Acta*, 1920, 3, 134–138.

o-AMINOTHIOPHENOL hydrochloride reacts with 4-amino-1,2-naphthoquinone in boiling 80% acetic acid solution with the formation of naphtho-6-phenothiazine hydrochloride, a chocolate-brown powder which is stable in air and readily soluble in water. The free base is an unstable yellow substance, the constitution of which follows from the conversion of the hydrochloride by concentrated sulphuric acid into a naphthophenazthione identical with that formed from *o*-aminothiophenol and 4-hydroxynaphthoquinone.—H. W.

Rhodanines. C. Grünacher. *Helv. Chim. Acta*, 1920, 3, 152–163.

IN the oxidation of rhodanine to Nencki's Rhodanine Red with ferric chloride the imino-hydrogen atom of rhodanine is not affected, since *N*-substituted rhodanines, such as *N*-phenylrhodanine, also yield red dyes under suitable conditions. On the other hand, benzylidenerhodanine is not changed by ferric chloride, so that the presence of a free methylene-group appears essential to the production of a dye. Alkylidenerhodanines react with aniline and phenylhydrazine to yield the corresponding anilides and phenylhydrazones, the sulphur of the thio-keto group being removed as hydrogen sulphide. Since aniline is comparatively readily removed from these anilides, they can be employed for condensing the rhodanine molecule with compounds containing a reactive methylene group; in this manner, benzylidenerhodanine-anilide can be condensed with a second molecule of rhodanine to yield *α*-benzylidene-*α*-rhodanal-rhodanine. Reaction with aniline or phenylhydrazine proceeds in a totally different manner with

rhodanines, such as *N*-phenylrhodanine, which contain the free methylene group; in this case, the sulphur of the thio-keto group remains unaffected and complete fission of the rhodanine ring occurs with the formation of thioureas or thiosemicarbazides, reaction occurring so readily that the process is conveniently used for the preparation of the latter class of substances. (See further *J. Chem. Soc.*, 1920, i., 252.)—H. W.

Phlorouacetophenone. K. B. Sen and P. C. Ghosh. *Chem. Soc. Trans.*, 1920, 117, 61–63.

ON heating phloroglucinol with glacial acetic acid and zinc chloride, a yellow crystalline compound is obtained, which is a pyran derivative produced by the combination of two molecules of the initially formed phlorouacetophenone. On boiling this 5,7-dihydroxy-2-*o*-*p*-trihydroxyphenyl-4-methylene-1,4-benzopyran with 10% sodium hydroxide the heterocyclic ring is broken and phlorouacetophenone regenerated. It forms white crystals, m. pt. 284°–285° C., and gives a phenylhydrazone which decomposes at 237°–240° C.—G. F. M.

Dyes and yeast cells. Von Euler and Florell. *See* XVIII.

Colour lakes. Bremner. *See* XXIII.

PATENTS.

Pyrazole-anthrone Yellow; Derivatives of —. F. Singer and A. Holl, Offenbach, Germany, Assignors to Chemical Foundation, Inc. U. S. Pat. 1,329,435, 3.2.20. Appl., 23.9.15.

See Eng. Pat. 14,103 of 1915; this J., 1916, 1101.

Sulphur dye; Blue — and process of manufacturing same. L. A. F. Haas, Assignor to Soc. Anon. l'Air Liquide, Paris. U.S. Pat. 1,329,898, 3.2.20. Appl., 5.8.18.

See Eng. Pat. 118,103 of 1918; this J., 1919, 676A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cellulose; Heating — with benzene under pressure. F. Fischer and W. Schneider. *Ges. Abhandl. Kenntn. Kohle*, 1919, 3, 287–300. *Chem. Zentr.*, 1919, 90, IV., 879. (See also Fischer and Niggemann, this J., 1919, 491A; Fischer and Kleinstück, p. 218A.)

CELLULOSE heated with benzene under pressure at 250°–260° C. is gradually decomposed, with the formation of water and products dissolved in the benzene, but scarcely any gas products. The decomposition products are about 6% greater in amount than the normal yield of tar on destructive distillation. They are dark-brown viscous liquids, and about 90% is soluble in alkalis. On acidification the solution deposits brown flakes (47.5%). The bulk of the extract is insoluble in light petroleum spirit, soluble in cold chloroform, and almost completely soluble in cold alcohol. The presence of water accelerates the process of extraction and improves the yield. Thus a double extraction of dry cellulose by dry benzene gave 2% of products, of air-dry cellulose (5.4% H₂O) 6.4%, and in presence of water 12%. By protracted heating of air-dry cellulose with benzene under pressure a yield of 12% is also attainable.—H. J. H.

Paper testing; Report on —. F. C. Clark. *Paper*, 1919, 25, 693–699, 739–743, 777–780, 829–831, 877–879.

THIS is the report of the Committee on Paper Testing presented to the Technical Association of the

Palp and Paper Industry (U.S.A.). The tests are considered under the heads of microscopical, physical, and chemical, and selected methods and apparatus are described. Microscopical examination relates principally to the fibres, their nature, proportions, and condition of beating; it comprises also the identification of the starches and specks in the paper. Physical tests comprise the following: Area, weight, bursting strength per unit area, thickness, bulk or thickness of a number of sheets, folding endurance, tensile strength, elongation at rupture, absorption, opacity, gloss or glaze, translucency, degree of sizing, retention of loading, breaking length. Chemical tests include percentage of ash, identification of loading, percentage of paraffin, sizing material, coating material, presence of acids, free chlorine, etc. A chart is given illustrating the relationships of the various tests to each other in the determination of the quality of the paper.—J. F. B.

Sulphite spirit manufacture; Influence of the design of the digester fittings on the quantity of waste liquor for —. A. D. J. Kuhn. *Woch. f. Papierfab.*, 1919, 50, 2317—2319, 2388—2391. *Chem. Zentr.*, 1919, 90, IV., 1016.

IN sulphite pulp mills which work up their waste liquors for the manufacture of spirit it is very important to have the correct proportion of fresh liquor to the quantity of wood and to employ a liquor which is rich in free sulphur dioxide. The ratio of liquor to wood depends upon the method of digestion, the species and the moisture-content of the wood, so that the quantity of waste liquor may show variations between 5 and 10 cub. m. per ton of cellulose. The digestion itself proceeds most favourably when the proportion of free sulphur dioxide is at least 70—80% of the total. A liquor rich in free acid increases the yield of sugar and alcohol. The digestion must be stopped when the concentration of sugar in the liquor has reached its maximum, because the sugar which has been formed then undergoes a gradual decomposition. Moreover, the use of a liquor of high concentration effects an economy of sulphur and steam and a higher efficiency in the digester with uniform results in the product. In order to obtain the maximum quantity of waste liquor, the digester must be drained under pressure. By blowing off under a pressure of 4—5 atm. the cellulose retains only 4 cub. m. of liquor per ton, whereas when blown off under 2—3 atm. the pulp retains 5.25 cub. m. per ton. It is also advantageous to introduce hot water on to the pulp under pressure, because in this way the water displaces the spent liquor, whereas the introduction of water without pressure merely results in the dilution of the spent liquor. The spent liquor coming from the digester under high pressure should be conducted to a pressure relief vat, where a powerful evolution of sulphur dioxide takes place, and this gas may be recovered for use in the preparation of fresh liquor.—J. F. B.

Extraction of vegetable tissues with benzene. Fischer and Kleinstück. *See* II A.

Sulphite-cellulose in tanning extracts. Knowles. *See* XV.

PATENTS.

Impregnating yarns and fabrics of paper; Process for —. O. Ruff and P. Praetorius, Breslau. Ger. Pat. 303,926, 21.3.17.

AQUEOUS solutions of phenols with formaldehyde and alkali are heated together, but not long enough to permit of the separation of insoluble products, and are then used for the impregnation of paper materials. By heating the latter further insoluble condensation products are formed. To add to the strength of the fabrics a further treatment with formaldehyde may be used.—H. J. H.

Fibres suitable for spinning from rushes ("Binsen"); Production of —. Deutsche Typha-Verwertungs-Ges.m.b.H., Charlottenburg. Ger. Pat. 308,565, 20.2.18.

THE bundle of fibres composing the rushes is disintegrated by a preliminary treatment with hot water, steam, either under pressure or not, or extremely dilute alkali, and then by the action of a strongly corrosive reagent, such as dilute caustic soda, which removes the tissues binding together the threads. The useful fibres are found in the outer parts of the plant.—H. J. H.

Textile fibres; Production of —. V. Wilke and L. Schorsch, Bielitz. Ger. Pat. 314,176, 6.1.18. Int. Conv., 25.1.17.

FOR the preparation of textile fibres from all kinds of vegetable matter—leaves, stalks, tendrils, etc., from the silk cocoon, and from spinners' waste—the raw material is treated with soap-makers' spent lye, with or without the admixture of alkali and/or other substances. The soap liquor dissolves or converts into soluble form the pectins (incrusting substances) without attacking the fibres. The dissolved matter must be removed by washing in a current of water or by boiling in water, followed sometimes by washing in dilute acid to soften the material.—H. J. H.

Paper; Process for producing sewing or basting thread from —. Vohwinkler Papier- u. Salicyl-Pergamentpapier-Industrie, Vohwinkel. Ger. Pat. 314,490, 16.4.18.

PAPER threads of suitable strength are impregnated with a mixture consisting of paraffin or stearine, linseed oil, mineral oil, and varnish, and afterwards drawn between corks to remove the excess.—J. H. L.

Yarn, ribbons, fibres, webbing, etc.; Machine for treating articles such as — with liquids. A. Schmidt, Charlottenburg. Ger. Pat. 314,721, 2.3.18.

THE material is drawn horizontally in series through the liquid contained in a tier of shallow boxes one above the other. The liquid is fed evenly into the top box and gravitates downwards from box to box after traversing each from end to end. The material may pass in the same direction as, or in the opposite direction to, the current of liquid. The arrangement has the advantage over deep baths through which the material is conducted vertically that only a small quantity of liquid is required, and that the heat losses are low when a hot liquid is used.—H. J. H.

Sulphite fibre; Process of manufacturing — and recovering sulphur dioxide. J. P. V. Fagan and H. G. Spear (Assignors to Brown Co.) and R. B. Wolf, Berlin, N.H. U.S. Pat. 1,327,666, 13.1.20. Appl., 30.4.18.

DURING the last portion of the cooking operation the gases and vapours which are usually blown with the digester contents into the blow pit are blown into a condenser from the top of the digester and the sulphur dioxide recovered.—L. L. L.

Straw, hay, wood, etc.; Treatment of — by boiling or steaming with reagents. E. Hauss, Osnabrück. Ger. Pat. 298,863, 1.1.16.

THE material is treated in a boiler with a suitable reagent in presence of air, oxygen, or a substance yielding oxygen, e.g. hydrogen peroxide. Economy of time and reagent are thereby attained.—H. J. H.

Cellulose from slightly lignified plants; Manufacture of —. M. Lüders, Magdeburg. Ger. Pat. 301,716, 30.8.16.

MATERIALS such as jute, manila, bullrush, typha,

flax straw and flax waste, hemp straw and hemp waste, nettle straw, etc., are digested in a closed vessel under pressure with a weakly acid magnesium sulphite solution, which, according to the degree of lignification of the material, may contain up to 90% of its sulphur dioxide in the combined state.—J. F. B.

Cellulose; Process for the production of — from moss or peat by heating with dilute acids. C. F. Hildebrandt, Hamburg. Ger. Pat. 311,712, 22.12.17.

Moss or peat is digested with dilute acids at a temperature below 100° C. and under atmospheric pressure, without subsequent treatment with alkaline liquors, and the process is arrested before saccharification commences. After washing, or partial or complete neutralisation, the cellulose obtained may be employed as fodder or for the production of paper, nitrocellulose, etc.—J. H. L.

Coating material [from viscose]. J. H. Young, Assignor to Aspromet Co., Pittsburgh, Pa. U.S. Pat. 1,327,933, 13.1.20. Appl., 11.9.18.

Viscosz is mixed with a soluble soapy material, the mixture having a lower surface tension than the viscose alone and being capable of spreading evenly over and firmly adhering to a sticky surface.

—B. V. S.

Viscose solutions; Preparation of improved, more stable —. R. Linkmeyer, Barby, and H. Hoyermann, Hanover. Ger. Pat. 312,392, 17.11.17.

Viscose solutions are treated with substances which contain amide or imide groups or are capable of forming such groups. Suitable substances are: Urea, derivatives of urea, cyanamide, dicyanodiamide, guanidine and its derivatives, acid amides, urethane, cyanates, thiourea, mustard oils, and condensation products of the above substances with aldoses. The viscose solutions are thereby rendered more stable and remain suitable for spinning for several weeks; the threads are characterised by good strength, but are sensitive to the action of water when they have been precipitated in a coagulating bath containing formaldehyde.—J. F. B.

Films and celluloid plates; Production of —. Deutsche Celluloid-Fabrik, Eilenburg. Ger. Pat. 314,119, 31.1.17.

NITROCELLULOSE, either dry or dehydrated by alcohol, is dissolved with a gelatinising agent in alcohol and rolled out to plates of small thickness, which are dried and treated with anhydrous solvents. Exclusion of every trace of water adds to mechanical strength.—H. J. H.

Paper, papier-mâché, and the like; Preparation of pulp or fibrous material for the manufacture of —. L. H. Skinner, London. Eng. Pat. 137,105, 30.12.18. (Appl. 21,790/18.)

FRONES of a silky nature are obtained from the tree mallow plant or from the tree mallow grafted or budded on to the stalk of a Jerusalem artichoke or sunflower. The stems are collected in the late autumn, preferably after frost; they are chopped, boiled with water for about eight hours, and reduced to pulp in a beating engine.—J. F. B.

Paper-making [from waste paper]. C. T. Crocker, Fitchburg, Mass. U.S. Pat. 1,326,849, 30.12.19. Appl., 21.10.18.

A CONTINUOUS stream of pieces of waste paper is subjected to the action of a hot alkaline solution by causing each oncoming piece of paper, as nearly separate as possible, to be submerged in and moved through the solution, to facilitate the exposure of its surfaces to the action of the solution for the purpose of removing or neutralising the ink or

other material fixed to the surface. The pieces of paper are then removed from the solution and freed from the major portion of the adherent liquid, exposed to the air to cool, and treated in a washing machine.—J. F. B.

Paper; Sizing of —. O. Ruff, Breslau. Ger. Pat. 307,691, 19.1.18.

THE paper pulp is first made neutral or alkaline, then treated with an alkaline solution of condensation products of phenols and formaldehyde, and finally acidified. The finished paper is heated for about an hour at 120° C. in a drying chamber, or passed slowly over the rollers of a drying machine heated to 120°—140° C.—J. H. L.

Paper or board; Process and machine for the manufacture of easily folded —. J. M. Voith, Heidenheim. Ger. Pat. 314,611, 25.7.18.

WEBS of paper pulp made on a flat or cylindrical machine with an uninterrupted wire surface are combined at the wet press with webs made on a machine, the wire of which is partly covered.

—J. F. B.

Cleaning material; Production of — from the waste of the paper industry. Reis and Co., Friedrichsfeld. Ger. Pat. 306,461, 18.9.17. Addition to 303,302 (this J., 1920, 15 a).

THE alkali in the liquor is partly replaced by salt, and the treatment is effected in the cold; e.g., the waste is treated with a liquor containing 10 parts of caustic soda to 60 parts of salt for three to four hours.—H. J. H.

Wood pulp; Process and apparatus for reclaiming waste products in the manufacture of —. H. K. Moore and J. T. Quinn, Berlin, N.H., Assignors to Brown Co., Portland, Me. U.S. Pat. 1,326,414, 30.12.19. Appl., 13.2.18.

WASTE liquor is "exploded" (atomised) in a furnace and the disintegrated particles are subjected to heat to cause the evaporation of the liquid content and the combustion of the combustible matter.

—J. F. B.

Cellulose and like substances; Apparatus for bleaching —. A. D. Berglind, Klosterpossen, Norway. U.S. Pat. 1,329,824, 3.2.20. Appl., 3.5.18. SEE Eng. Pat. 114,456 of 1917; this J., 1918, 296 a.

Volatile solvents. Ger. Pat. 303,396. See I.

Resinous substances. Ger. Pat. 314,118. See XIII.

Material treated with cellulose derivatives. U.S. Pat. 1,327,197. See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing mixtures of wool and staple fibre. G. Rudolph. Färber-Zeit., 1919, 30, 285—286.

THE staple fibre is dyed, at as low a temperature as possible (50°—60° C.), with direct cotton colours, which can be diazotised and developed. By this method shades fast to bleeding, washing, and milling are produced. The wool is then dyed by neutral wool dyestuffs. For general fastness the use of vat colours is recommended. With many vat colours a level tone is obtained directly; with others the wool is dyed a paler shade, and in such cases the shade is deepened by means of fast acid colours. For the production of shades fast to light the wool is dyed by means of neutral wool colours and the staple fibre by direct cotton dyes which can be after-coppered.—L. L. L.

Printing; Discharge — on staple fibre union material. O. Gaumnitz. *Färber-Zeit.*, 1919, 30, 286—287.

STAPLE fibre is reduced in strength considerably in a damp condition. To overcome this defect union yarns consisting of 25—75% of staple fibre and 75—25% of wool are produced. The raw material woven from such yarn is singed or cropped and then crabbed. It is then dyed with dischargeable colours and printed with basic dyes along with a discharge for the production of fancy goods. The colour from the staple fibre is more readily discharged than that from the wool, leaving web-like effects.—L. L. L.

Colour lakes. Brenner. See XXIII.

PATENTS.

Bleaching and similarly treating textile fabrics and yarns in rope form; Machines for —. W. Morley, Bolton, and Bleachers' Assoc., Ltd., Manchester. Eng. Pat. 136,713, 20.2.19. (Appl. 4229/19.)

THE material in rope form is fed mechanically into kiers or cisterns, along with a stream of liquor, through a hollow trunk of metal or other suitable material. The trunk is hinged or pivoted to a metal box casting or the like and receives a reciprocating motion lengthwise of the kier or cistern, and also a transverse oscillating movement. For round vessels, when desirable, a variable speed and variable throw of stroke to the trunk may be imparted in order to fit the material to the vessel. The feeding of the material into the kier or cistern along with the liquor insures thorough impregnation before entering the vessel.—L. L. L.

Bleaching composition. R. B. Kadish and T. W. Buscher, Assignors to The Kadish Manufacturing Co., Chicago, Ill. U.S. Pat. 1,327,394, 6.1.20. Appl., 11.12.17.

THE composition is obtained by mixing a fluoride, a hydroxide, and an acid sulphate of an alkali metal while in a nearly dry state.—L. L. L.

Dyeing machine. H. M. Dudley, Philadelphia, Pa. U.S. Pat. 1,327,200, 6.1.20. Appl., 13.11.17.

THE machine comprises a receptacle provided with a series of fibre chambers, within each of which is a non-perforated core. The fibre to be treated is wound upon non-perforated hollow members which slide over the cores. Means are provided at each end of the fibre chambers for compressing the fibre and also means for forcing a liquid through the compressed fibre.—L. L. L.

Dyeing machine; Skein —. H. M. Dudley, Philadelphia, Pa. U.S. Pat. 1,327,657, 13.1.20. Appl., 22.8.17.

A DYEING chamber is provided with a removable frame adapted to carry fibre skeins, with a series of foraminous pipes below the frame, and a removable top carrying a series of foraminous pipes. Means are also provided for removing liquid from the dyeing chamber below the frame and for forcing liquid through either series of pipes and through the dyeing chamber.—L. L. L.

Dyeing machine. H. M. Dudley, Philadelphia, Pa. U.S. Pats (A) 1,327,658, (B) 1,327,660, and (C) 1,327,662, 13.1.20. Appl., (A) 13.11.17, (B) 24.12.17, (C) 26.1.18.

(A) WITHIN the dye vessel is a series of cores, each carrying an extended arm supporting a series of annular members. The fibre to be dyed is wound upon hollow foraminous spindles, which slide upon the annular members. Means are provided for forcing liquid transversely through the spindles and

through the fibre and also for compressing the fibre upon the spindles. (B) The machine consists of a vessel containing a series of fibre chambers, each provided with a foraminous end and a foraminous plate, which slides within the chamber and compresses the fibre between the end and the plate. The fibre chambers are connected by a series of inwardly diverging tubes with liquid chambers, and means are provided for continuously passing liquid in opposite directions through the tubes, the foraminous members, and the fibre within the fibre chambers. (C) The machine comprises a series of fibre chambers, each provided with a foraminous spindle, upon which fibre is wound. Means are provided for forcing liquid in either direction through the series of spindles and fibre wound thereon.—L. L. L.

Fabric-treating device. H. M. Dudley, Philadelphia, Pa. U.S. Pat. 1,327,659, 13.1.20. Appl., 20.12.17.

THE device consists of a series of fabric chambers into each of which is inserted a foraminous core upon which the fabric is wound. The wound fabric is encased by a foraminous cover. Means are provided for closing all the openings of the core except those adjacent to the fabric and for forcing the liquid through the fabric in either direction.—L. L. L.

Dyeing device. H. M. Dudley, Philadelphia, Pa. U.S. Pat. 1,327,661, 13.1.20. Appl., 7.1.18.

THE device comprises a series of fabric chambers with non-perforated side walls, within each of which is a series of hollow foraminous fabric reels; the upper end of each fabric chamber and hollow reel may be closed. Liquid chambers are connected to the lower ends of the hollow reels and to the lower ends of the fabric chambers, and means are provided for passing liquid in opposite directions through the device and the fabric wound upon the reels.—L. L. L.

Fibre-treating machine. H. M. Dudley, Philadelphia, Pa. U.S. Pat. 1,327,663, 13.1.20. Appl., 13.3.18.

THE machine consists of a receptacle provided with a series of removable hollow foraminous spindles upon which the fibre is wound. Means are provided for closing one end of the spindles, for compressing the fibre in the direction of the axes of the spindles and also at right angles to the axes, and for passing liquid in either direction from the receptacle to the interior of the spindles and through the fibre.—L. L. L.

Hosiery-dyeing machine. H. M. Dudley, Philadelphia, Pa. U.S. Pat. 1,327,756, 13.1.20. Appl., 22.6.18.

THE two end plates of a dyeing receptacle are connected by a series of spaced bars revoluble within the receptacle, and a series of foraminous plates extends outwards from the bars and abuts upon the end plates, their outer extremities being connected by removable foraminous covers, which, together with the end plates, form a series of fabric chambers. Means are provided for revolving the fabric chambers and for forcing liquid through them, the chambers being maintained beneath the liquid during its passage therethrough.—L. L. L.

Woollen fabrics dyed fast to milling; Process for improving the wearing quality of —. L. Cassella und Co., Frankfurt. Ger. Pat. 303,223, 17.1.17.

THE dyed fabrics are treated with tannins or formaldehyde or both, or with tannins in combination with metallic salts. In comparing the effectiveness of these different methods of treatment the

treated fabrics may be exposed to light and weather for several months and then subjected to the biuret test. Those which have deteriorated most will give the strongest violet coloration.—J. H. L.

Dyeing yarns and fabrics of paper treated with phenol-formaldehyde condensation products; Process of —. O. Ruff, Breslau. Ger. Pat. 306,447, 1.9.17.

THE dyestuff is added to the liquid used for impregnating the material and fixed by subsequent heating. Dyestuffs suitable for this purpose are those which are fast and not precipitated in slightly alkaline baths—e.g., sulphur, basic, and substantive colours. As example is given the use of a condensation product of crude cresol, 8% caustic soda, and formaldehyde, and a solution of a sulphur colour in caustic soda and sodium sulphide.

—H. J. H.

Aniline Black; Process of producing —. A. Ehrenzweig, Hermsdorf, and M. van Delden und Co., Gronau. Ger. Pat. 314,660, 2.10.15.

ANILINE, a diamine, and chlorate are employed together with cyanogen or thiocyanogen compounds containing no iron, copper, or other heavy metals. It is thus possible to effect oxidation of the whole of the aniline or diamine and obtain great depth of colour without risk of weakening the fibre.

—J. H. L.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphur dioxide; Solubility of — in sulphuric acid. F. D. Miles and J. Fenton. Chem. Soc. Trans., 1920, 117, 59—61.

THE solubility of sulphur dioxide in sulphuric acid diminishes fairly regularly with increasing concentration of the acid to a minimum value with 86% acid, after which a sudden and rapid increase in solubility accompanies further increase in the percentage strength of the acid. The solubility of the gas at 20° C. and normal pressure was determined over a series of concentrations from 55% acid upwards, and following are some of the results:—

Sulphuric acid, per cent.	Grms. SO ₂ dissolved in 100 grms. of acid.	Sulphuric acid, per cent.	Grms. SO ₂ dissolved in 100 grms. of acid.
55.1	5.13	88.1	2.90
68.9	4.16	92.8	3.21
80.2	3.12	95.5	3.60
85.8	2.80	98.5	4.03

—G. F. M.

Sulphurous acid; Oxidation of — by ferric salts. J. Meyer. Ber., 1920, 53, [n], 77—78.

A CRITICISM of Houben's recent method for estimating acids and ferrous and ferric salts in the same solution (this J., 1920, 85 A). (See further, J. Chem. Soc., 1920, ii., 173.)—J. C. W.

Iodides; Oxidation potentiometric titration of — in the presence of chlorides and bromides. I. M. Kolthoff. Rec. Trav. Chim., 1920, 39, 208—214.

USING the potentiometric method previously described (this J., 1919, 444 A) iodides may be estimated by titration with potassium bichromate or, preferably, potassium bromate or iodate, in the presence of hydrochloric acid. Chlorides do not interfere with the titration, and the presence of relatively large amounts of bromides does not vitiate the results.—W. G.

Ammonium silicate. III. R. Schwarz and R. Sonard. Ber., 1920, 53, [n], 1—17. (Compare this J., 1919, 498 A.)

AMMONIACAL solutions of dioxadisiloxane or monosilane, or of preparations of silicic acid from various sources, provided they have not been ignited, have higher conductivities than ammonia solutions, which indicates the formation of ammonium silicate. The increment, however, is dependent on the initial proportion of water in the material employed, and the results indicate that the dissolved silicic acid exists in three forms, of molecular weights in the ratio 1:2:3. (See further J. Chem. Soc., 1920, ii., 175.)—J. C. W.

Barium sulphide; Action of water on —. E. Terres and K. Brückner. Z. Elektrochem., 1920, 26, 1—24.

A DOUBLE compound Ba(SiH₃OH)₂·5H₂O is formed when barium sulphide is extracted with water at all temperatures up to 100° C. This compound is comparatively stable, and under no circumstances can barium hydroxide be separated from the solution by crystallisation. These results explain the failure of many processes put forward for the manufacture of barium hydroxide from barium sulphide. (See also J. Chem. Soc., Apr., 1920.)

—J. F. S.

Strontium sulphide; Action of water on —. E. Terres and K. Brückner. Z. Elektrochem., 1920, 26, 25—32.

STRONTIUM sulphide is converted into strontium hydroxide and strontium hydrosulphide by the action of water, and there is no formation of an intermediate hydroxy-hydrosulphide as in the case of barium (see preceding abstract). The solution obtained by treating strontium sulphide with boiling water, on cooling deposits pure strontium hydroxide. (See also J. Chem. Soc., Apr., 1920.)

—J. F. S.

Chromium chromate. M. Z. Jovitchitch. Helv. Chim. Acta, 1920, 3, 40—46.

WHEN a solution of chromium oxide in nitric acid is evaporated until excess of acid is removed, and the residue is dissolved in water and treated with ammonia, a dark-brown, almost black, product is obtained, having the composition CrO₃·Cr₂O₃·71H₂O. The substance loses 11H₂O when placed over sulphuric acid, 21H₂O at 105° C. and 61H₂O at 205° C. It loses chromium trioxide even on protracted washing with warm water. It may be used for the preparation of pure chromium hydroxide free from alkali by treating its solution in boiling concentrated hydrochloric acid with alcohol till reduction is complete and then precipitating with an excess of ammonia. Anhydrous chromium chromate is obtained as a porous, black mass by dissolving chromium oxide in nitric acid, evaporating the solution to dryness and cautiously heating the residue to 290° C. It is readily converted by ammonia, alkali carbonate or hydroxide, or by boiling water into chromate and chromium hydroxide.

—H. W.

Chromium hydroxide; Formula of —. M. Z. Jovitchitch. Helv. Chim. Acta, 1920, 3, 46—49.

THE formula Cr₃(OH)₈O₂·10H₂O or Cr₃(OH)₁₀O·9H₂O is assigned to chromium hydroxide on account of its solubility in ammonia and the preparation from such solutions of complex salts which invariably contain 10H₂O, and also because it absorbs carbon dioxide from the air with the formation of the carbonate CO₃Cr₂(OH)₂·8H₂O. (See also J. Chem. Soc., 1920, ii., 179.)—H. W.

Calcium hydride; Some reactions of —. S. Reich and H. O. Serpek. *Helv. Chim. Acta*, 1920, 3, 138–141.

CARBON monoxide is reduced by calcium hydride at a dull red heat with formation of hydrogen, methane, and formaldehyde. When a mixture of the hydride with sodium carbonate or bicarbonate is gently heated, formic acid is produced. Ammonia is formed in small amount when nitrogen is passed over calcium hydride at about 500° C., but the main reaction appears to consist in the formation of calcium nitride. Acetone reacts energetically with finely divided calcium hydride giving mesityl oxide and a cyclic substance, b. pt. 226°–236° C. Calcium hydride appears to act as a reducing agent only at very high temperatures, and its use in the hydrogenation of organic substances must be very limited, particularly as its action, when occurring, appears to lead preferably to condensation products. (See also *J. Chem. Soc.*, 1920, i., 249.)

—H. W.

Lead borates. H. V. Thompson. *Trans. Ceram. Soc.*, 1918–9, 18, 510–511.

ABEGG (*Handb. der anorg. Chem.*, 1909, 3, pt. ii.) records four lead borates, but the author finds that there are only two of definite chemical composition, viz., the triborate, $\text{PbO} \cdot 3\text{B}_2\text{O}_3$, formed by fusing a mixture of lead carbonate and boric acid and washing out the excess of boric acid with water, and the metaborate, $\text{Pb}(\text{BO}_2)_2 \cdot \text{H}_2\text{O}$, formed by precipitating a solution of a lead salt with an excess of a concentrated solution of borax. The precipitate produced by mixing cold solutions of lead nitrate and borax is of indefinite composition. The polyborate, $2\text{PbO} \cdot 3\text{B}_2\text{O}_3 \cdot 4\text{H}_2\text{O}$, mentioned by Abegg is not formed under the conditions mentioned, the metaborate being produced. (See also *J. Chem. Soc.*, 1920, ii., 176.)—A. B. S.

Antimony trisulphide; Formation of the black modification of —. F. de Bacho. *Annali Chim. Appl.*, 1919, 12, 143–152.

WHEN red antimony trisulphide was dried for an hour at different temperatures between 150° and 300° C. no material differences were observed in the loss of weight at the different temperatures (1·12 to 0·87%); the change to the black modification occurred at 212° C. In a current of hydrogen sulphide the change to the black modification occurred at 207° C. By adding antimony trichloride to the red sulphide the temperature at which the change took place in carbon dioxide was lowered to 115° C. Metallic silver also has a favourable influence on the conversion. Ammonium chloride and antimony chloride promote the change to the black form when the red sulphide is boiled with water. (See also *J. Chem. Soc.*, 1920, ii., 183.)—C. A. M.

Metastannic acid; Soluble —. H. Kreis. *Schweiz. Chem.-Zeit.*, 1919, 389–390.

DURING the analysis of samples of tinned iron it was observed that no deposition of metastannic acid occurred when the turnings were dissolved in dilute nitric acid (sp. gr. 1·23). This was proved to be due to the protective action of colloidal ferric hydroxide formed by hydrolysis of the nitrate, and it was found that the presence of three parts of iron prevented the precipitation of ten parts of tin. (Compare Antony and Mondolfo, this *J.*, 1898, 1186.) (See also *J. Chem. Soc.*, 1920, ii., 182.)

—A. R. P.

Bismuth; Peroxides of —. R. R. Le G. Worley and P. W. Robertson. *Chem. Soc. Trans.*, 1920, 117, 63–67.

VARIOUS peroxides of bismuth were obtained by the action of chlorine, ammonium persulphate, or

potassium ferricyanide on bismuth trioxide in presence of boiling alkali hydroxide of different concentrations. In dilute solutions bismuth tetroxide was the only product, and was isolated from admixed trioxide and occluded alkali by repeated grinding with glacial acetic acid. Four modifications were recognised, viz., Bi_2O_4 (brown), Bi_2O_4 (purplish black), $\text{Bi}_2\text{O}_4 \cdot \text{H}_2\text{O}$ (brown), and $\text{Bi}_2\text{O}_4 \cdot \text{H}_2\text{O}$ (purplish black). All decompose at 160° C. and are soluble in nitric acid (sp. gr. 1·2) with liberation of oxygen. Another variety of tetroxide, $\text{Bi}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ (yellow), is formed together with a pentoxide, $\text{Bi}_2\text{O}_5 \cdot \text{H}_2\text{O}$, by the action of chlorine on Bi_2O_3 in boiling concentrated alkali. Finally a hexoxide, Bi_2O_6 , was obtained in small quantities along with Bi_2O_4 by the action of ammonium persulphate or potassium ferricyanide on the trioxide suspended in boiling concentrated alkali. It was isolated by continued extraction with warm nitric acid (sp. gr. 1·2), in which it is insoluble. The hexoxide slowly loses oxygen at ordinary temperatures and darkens in colour.

—G. F. M.

Cerium carbide; Behaviour of — towards nitrogen. F. Fichter and C. Schölly. *Helv. Chim. Acta*, 1920, 3, 164–172.

FINELY-divided cerium carbide may be prepared by heating a mixture of cerium dioxide (1 mol.) and carbon (6 atoms) in a graphite tube in an electric vacuum-oven at 1600° C. In spite of its apparent similarity to calcium carbide, it does not yield at 1250° C. with nitrogen any compound which contains both carbon and nitrogen, but the carbon is replaced by nitrogen with the formation of cerium nitride. The possibility of the formation of cyanides and cyanamides from carbides appears to exist solely in the groups of the alkali and alkaline-earth metals and not in that of the earth metals; it does not depend on the type of carbide, but on the nature of the metal. Cerium carbide also reacts with ammonia at 1250° C. yielding the nitride, but the action is slower and less complete than when nitrogen is used.—H. W.

Uranium; Violet compounds of —. J. Aloy and E. Rodier. *Bull. Soc. Chim.*, 1920, 27, 101–105.

THE production of violet compounds of uranium described by Aloy (this *J.*, 1901, 480) is shown to be a general reaction, and does not necessarily involve the action of light. Such compounds are obtained whenever a uranous and a uranic salt are present together at the requisite temperature in a neutral or feebly acid medium. (See further *J. Chem. Soc.*, 1920, ii., 182.)—W. G.

Zirconium monoxide; Question of the existence of —. R. Schwarz and H. Deisler. *Ber.*, 1920, 53, [B], 1. (Compare this *J.*, 1920, 62 A.)

A RECOGNITION of similar work by Wedekind (this *J.*, 1913, 197).—J. C. W.

Carbon monoxide. Müller. See IIa.

Phosphoric acid. Stutzer. See XXIII.

Arsenious anhydride. De Bacho. See XXIII.

PATENTS.

Sulphuric acid; Apparatus for making —. H. V. Welch. Assignor to International Precipitation Co., Los Angeles, Cal. U.S. Pat. 1,328,552, 20.1.20. Appl., 1.10.18.

SULPHURIC acid mist present in chamber gases is removed by circulating the gases through an electrical precipitator connected to the chamber by inlet and outlet tubes, and provided with means for maintaining a film of dilute sulphuric acid in contact with the circulating gases.—L. A. C.

Hydrofluoric acid; Process for the removal of — from phosphoric acid. W. H. Ross, Washington, D.C. U.S. Pat. 1,329,273, 27.1.20. Appl., 26.7.19.
 Calcium chloride, equivalent in amount to the fluorine present, is added to the concentrated phosphoric acid solution thereby precipitating the fluorine.—S. S. A.

Ammonia; Production of — from atmospheric nitrogen. General Chemical Co., Assignees of F. W. de Jahn, New York, Eng. Pat. 120,516, 28.2.18. (Appl. 3607/18.) Int. Conv., 3.11.17.

REGULATED quantities of steam and air are introduced into an incandescent material to obtain a gaseous mixture of approximately 1 vol. of nitrogen to 3 vols. of gas consisting of hydrogen and carbon monoxide, and the mixture, after removal of dirt, sulphur, and the like, is passed together with regulated quantities of steam over a catalyst at the required temperature and at the ordinary pressure to oxidise carbon monoxide to carbon dioxide and to form hydrogen. The resulting mixture is subjected to a pressure not exceeding 1100 lb., and whilst under pressure carbon dioxide and sulphur impurities are removed by water scrubbing, carbon monoxide and other sulphur impurities are eliminated by treatment with caustic soda, and oxygen and carbon monoxide by means of cuprous material such as cuprous ammonium carbonate free from chlorides or sulphates. Traces of moisture are then removed, and the residual mixture containing nitrogen and hydrogen is passed over a catalyst consisting of iron or other suitable metal together with sodium, at a temperature of 450° C. under a pressure of not more than 1400 lb. The accumulated inert rare gases are removed from the ammonia produced. The pressure of the gases is used in raising the hot alkali solution required for the removal of the carbon monoxide.—S. S. A.

[*Synthesis of ammonia.*] *Process of making a mixture of nitrogen and hydrogen.* C. Ellis, Montclair, N.J., Assignor to C. S. Lutkins, Rye, N.Y. U.S. Pat. 1,327,029, 6.1.20. Appl., 18.5.16.

A MIXTURE of water-gas and producer-gas is prepared in a single operation, and as much as possible of the contained carbon monoxide is removed by liquefaction. The residual gas is passed over lime and an iron compound to remove sulphur, and sufficient hydrogen is then added to produce a mixture containing hydrogen and nitrogen in the proportion of 3 to 1 by volume. The added hydrogen is previously treated to remove substances injurious to catalysts employed in the synthesis of ammonia.

—W. E. F. P.

Ammonia; Production of synthetic — from air or nitrogen. R. F. Gardiner, Clarendon, Va. U.S. Pat. 1,323,082, 13.1.20. Appl., 13.1.19.

Air is brought into contact with an alkaline solution containing a metal from which nascent hydrogen is being evolved.—S. S. A.

Ammonia; Synthetic production of —. H. C. Greenwood, London. U.S. Pat. 1,329,397, 3.2.20. Appl., 15.2.18.

Is the synthetic production of ammonia, the incoming gases are heated by the issuing gases in a heat interchanger through which at least one of the gas streams flows at a velocity greater than the critical velocity.—S. S. A.

Ammonium salts; Production of —. W. S. Curphey, London. Eng. Pat. 137,315, 21.8.18. (Appl. 13,596/18.)

For the production of ammonium salts, e.g., ammonium sulphate or a mixed product ("Burkheiser salt") containing sulphate and sulphite, ammoniacal liquor is heated in an apparatus comprising two distillation vessels combined to form a

two-stage still. The gas evolved in the first stage contains a portion of the ammonia together with hydrogen sulphide, carbon dioxide, and at least part of the volatile organic impurities, and is absorbed in an acid liquor, e.g., sulphuric acid. The products of the reaction are withdrawn from the absorber and filtered, and the still acid liquor is passed into a second absorber and to it a solution of sulphurous acid or bisulphites may be added. The ammoniacal liquor which has been heated in the first stage is passed through a device, in which it is mixed with lime, into the second distillation vessel, and the relatively pure ammonia obtained at this stage is absorbed in the liquor contained in the second absorber. The precipitated salts obtained are removed from the absorber and drained, and the liquor is collected and returned to the first absorber.—S. S. A.

Ammonia; Manufacture of salts of —. Amsterdamsche Superfosfaatfabriek, Amsterdam, Holland. Eng. Pat. 138,002, 21.5.19. (Appl. 13,108/19.) Int. Conv., 10.2.19.

AMMONIA is absorbed in a concentrated solution of the required ammonium salt contained in a cooled iron absorber which is connected to a column and scrubber through which a portion of the ammonium salt solution, cooled to atmospheric temperature, is circulated for the recovery of any ammonia which escapes solution in the absorber. The resulting ammoniacal solution of the salt is passed into a wooden vat, neutralised with acid, and pumped thence to an evaporating plant.—S. S. A.

Ammonium sulphate; Production [purification] of —. W. A. Sloss, Toledo, Ohio. U.S. Pat. 1,328,312, 20.1.20. Appl., 22.5.19.

COMMERCIAL ammonium sulphate is purified by treatment with ammonia at a temperature above that at which pyridine becomes volatile.

—W. E. F. P.

Caustic potash; Manufacture of —. C. L. Higgins, and The United Alkali Co., Ltd., Liverpool. Eng. Pat. 137,632, 6.2.19. (Appl. 2912/19.)

To prevent the deleterious action of caustic potash solution on metallic evaporating apparatus, the surface of the apparatus which comes into contact with the caustic potash is made of or covered with gold.—S. S. A.

Iron compounds; Process of manufacturing —. R. S. Penniman, jun., and N. M. Zoph, Berkeley, Cal., Assignors to West Coast Kalsomine Co., Cal. U.S. Pat. 1,327,061, 6.1.20. Appl., 20.6.17.

METALLIC iron is immersed in a solution of a ferrous salt, the solution is heated, and an oxidising agent is introduced.—S. S. A.

Potassium; Process of obtaining combined — from greensand. T. C. Meadows, New York, M. Hauber, jun., West Haverstraw, and H. W. Charlton, New York. U.S. Pat. 1,327,161, 6.1.20. Appl., 6.12.18.

A MIXTURE of greensand and lime is digested with an aqueous solution containing sufficient calcium chloride to react with substantially all the potassium present in the greensand, at a pressure not exceeding 200 lb. per sq. in. and at a corresponding temperature, until a solution containing more than 2% of potassium chloride is obtained together with a valuable sludge material.—S. S. A.

Potassium; Process of producing combined — from greensand. H. W. Charlton, New York, Assignor to American Potash Corporation. U.S. Pat. 1,329,369, 3.2.20. Appl., 12.11.18.

POTASSIUM compounds are extracted from green-

sand by agitating a mixture of the material with more than 50% of its weight of lime and more than three times its weight of water under a pressure exceeding 150 lb. per sq. in.—L. A. C.

Potassium salts; Process of obtaining — from liquids containing the same. Process of obtaining potassium salts from saline waters. Process of recovering potassium sodium sulphate and other potassium salts from saline waters. G. B. Burnham, Berkeley, Cal. U.S. Pats. (A) 1,328,416, (B) 1,328,417, and (C) 1,328,418, 20.1.20. Appl., (A) 18.9.16, (B and C) 1.7.18.

(A) A solution containing sodium, potassium, sulphate, and chloride ions is evaporated to approximate saturation with potassium sodium sulphate, and then cooled to below 44° C. to saturation with potassium chloride; the salts deposited are removed and the solution is evaporated at about 44° C. again to saturation with potassium sodium sulphate and cooled to approximate saturation with potassium chloride. After separation of the deposited salts, the solution is cooled further, and potassium chloride is deposited in crystalline form. (B) A solution containing sodium, potassium, chloride, sulphate, and carbonate ions is evaporated to crystallise out hanksite, potassium sodium sulphate, and potassium sodium sulphate together with a carbonate, in successive stages; the crystals deposited are separated after each evaporation and the final liquor is cooled, whereby potassium chloride is deposited. (C) A solution containing potassium, sodium, chloride, and sulphate ions is cooled to crystallise out Glauber salt; the liquor, after separation of the crystals, is evaporated in two stages, the first to crystallise out sodium chloride, and the second to crystallise out sodium chloride and potassium sodium sulphate.—L. A. C.

Wood ashes; Apparatus for extracting lye from —. W. P. D. Moross and J. C. Costello, Chattanooga, Tenn. U.S. Pat. 1,328,096, 13.1.20. Appl., 2.8.17.

THE apparatus comprises a tray or receptacle on which the ashes are placed, provided with a movable lid which serves as a deflector during agitation. The ashes are mixed with water and agitated by steam introduced into the receptacle through a series of jets. The liquor drains into a steam-heated gutter and is collected.—S. S. A.

Calcium carbonate; Process of grinding — and product thereof. J. H. Ryan, Assignor to D. R. Seaman, Chicago, Ill. U.S. Pat. 1,328,299, 20.1.20. Appl., 24.3.19.

CALCIUM carbonate in a relatively fine condition is ground in the presence of sufficient water to form an emulsion or plastic mass with the very fine particles present and hold the coarser particles in suspension, whereby the whole of the material is reduced to an impalpable powder.—L. A. C.

Borax; Process of recovering — from saline waters. G. B. Burnham, Borosolway, Cal. U.S. Pat. 1,328,614, 20.1.20. Appl., 30.6.19.

A solution containing sodium sulphate, carbonate, and borate is cooled to crystallise the sodium sulphate and carbonate; the crystals are separated, and the liquor maintained cold, whereby crystals of borax are deposited.—L. A. C.

Calcium fluoride precipitate; Process of obtaining —. W. S. Chase, Lakewood, Ohio, Assignor to National Carbon Co. U.S. Pat. 1,329,072, 27.1.20. Appl., 1.3.17.

AN insoluble metallic fluoride, e.g., calcium fluoride, is precipitated by adding a concentrated solution of some other compound of the metal to a concentrated solution of hydrofluoric acid.—S. S. A.

Cyanide-producing apparatus; Process of producing cyanide. W. D. Mount, Saltville, Va., Assignor to Nitrogen Products Co., Providence, R.I. U.S. Pats. (A) 1,329,721 and (B) 1,329,722, 3.2.20. Appl., 26.4.18 and 4.10.18.

(A) THE apparatus comprises a cyanising retort provided with means for gradually moving the treated material out of the retort at a temperature below that at which it congeals, and a mechanically operated toothed device for breaking off the material in fragments as ejected from the retort. (B) A material, part of which is in the solid phase, is so disposed as to provide interstices throughout its mass. It is heated, and treated by passing a substance in the gaseous phase through the interstices, whereby a partial reaction is promoted between the two substances, with formation of a compound which tends to liquefy at the temperature employed; by causing a circulatory movement of the resulting mass this compound is cooled and congealed. The mass is then broken into fragments which are cooled in a stream of nitrogen to a temperature at which the reaction product is unoxidisable.—S. S. A.

Thorium; Art of recovering —. O. N. Berndt, Assignor to Lindsay Light Co., Chicago, Ill. U.S. Pat. 1,329,747, 3.2.20. Appl., 26.6.19.

A PHOSPHATIC thorium compound is treated with fuming sulphuric acid, thereby converting the thorium into an insoluble compound.—S. S. A.

Permanganate; Process for the production of —. R. E. Wilson, Cambridge, Mass. U.S. Pat. 1,330,032, 3.2.20. Appl., 27.9.18.

A CATALYST is employed to accelerate the reaction in which more highly oxidised manganese compounds are prepared from less oxidised compounds by treatment with a halogen.—S. S. A.

Hydrogen; Manufacture of —. H. E. F. Gool-Adams and H. C. Greenwood, London, Eng. Pat. 137,340, 24.1.18. (Appl. 1383/18.)

HYDROGEN practically free from carbon monoxide is obtained by passing a mixture of water-gas (or similar gas) and steam through a series of catalyst chambers separated by intermediate lime containers, the alternate catalysis (oxidation of carbon monoxide) and absorption (of carbon dioxide) being effected without fall of temperature by passing the gas mixture through a heat-interchanger before and after the series of operations.—W. E. F. P.

Hydrogen gas; Means for charging retorts or apparatus with spathic ore for the manufacture of —. W. J. and W. R. Bates, Silverdale, Staffs. Eng. Pat. 137,674, 13.3.19. Appl. 6252/19.)

PERFORATED metal cages, of appropriate mesh, are employed as containers for graded spathic ore. The charged cages are placed one above the other in the retort, so that the fineness of the ore decreases from the top downwards. (Reference is directed to Eng. Pat. 12,117 of 1912; this J., 1912, 1079.)

—W. E. F. P.

Minerals [sulphur] from ores; Apparatus for extracting —. G. M. Shires, Houston, Tex. U.S. Pat. 1,328,210, 13.1.20. Appl., 29.11.18.

AN apparatus for extracting sulphur from ore consists of a number of connected heater drums, arranged one above the other, and a cylindrical container disposed beneath and communicating with the bottom drum. A heating fluid is introduced into each of the drums, and means are provided for charging the ore into the top vessel and for separating the sulphur from the gangue in the container.—W. E. F. P.

Graphite [from kish]; Process for producing flake — S. W. Osgood, Assignor to N. C. Kenner, Chicago, Ill. U.S. Pat. 1,328,845, 27.1.20. Appl., 1.3.16.

Improve flake graphite, separated from kish, is treated to remove iron particles and gritty material and then graded by air flotation.—W. E. F. P.

Iodine; Apparatus for purifying — R. M. Carter, Baltimore, Md., Assignor to U.S. Industrial Alcohol Co. U.S. Pat. 1,329,457, 3.2.20. Appl., 21.12.18.

A vapourising vessel is provided with means for introducing a heated gaseous medium and with an internal condenser through which cold water is circulated.—W. E. F. P.

Cyanogen compounds; Process of producing — J. C. Clancy, Freeport, N.Y., Assignor to The Nitrogen Corporation, Providence, R.I. U.S. Pat. 1,329,652, 3.2.20. Appl., 12.6.17.

SEE Eng. Pat. 133,095 of 1918; this J., 1919, 900 A.

Packing material resistant to chlorine. Eng. Pat. 131,871. See I.

Gas for balloons. Ger. Pat. 303,966. See HA.

VIII.—GLASS; CERAMICS.

Refractories for glassworks' use; Specifications for — W. J. Rees. Trans. Ceram. Soc., 1918-9, 18, 420—431.

THE Refractories Research and Specifications Committee of the Society of Glass Technology have drawn up provisional specifications for clays for use in glass manufacture (this J., 1919, 137 A). In the discussion W. R. Ormandy pointed out that the specification for glass pots is too severe, that it places too much emphasis on the total porosity and too little on the size of the pores. J. W. Mellor confirmed this, and considered that the proposed specification sets up impracticable ideals, and that it would immobilise manufacturing processes.

—A. B. S.

Blue glass. J. W. Mellor. Trans. Ceram. Soc., 1918-9, 18, 478—480.

WHEN glass is stained blue by the addition of cobalt oxide to the batch, an incandescent filament viewed through it appears red. The addition of a small percentage of chromium oxide neutralises the red rays so that only blue rays are transmitted. Photographs of the absorption spectra are given.

—A. B. S.

Silica bricks; Constitution and micro-structure of — and changes involved through repeated burnings at high temperatures. H. Insley and A. A. Klein. U.S. Bureau of Standards, Tech. Paper No. 124, July 11, 1919. 31 pages.

COMMERCIAL silica bricks which have been reheated repeatedly by use in kilns contain quartz, cristobalite, and tridymite, with small amounts of pseudowollastonite (CaSiO_3) and glass. The greater the proportion of flux present the more rapid is the inversion of the silica, but 6% or more lime makes the bricks too fusible. The lime added during manufacture acts more as a flux than as a bond, the strength of the bricks being chiefly due to the interlocking crystals of quartz, cristobalite, and tridymite. The extent to which a silica brick will expand when in use may be judged from the extent to which the quartz has been converted into cristobalite or tridymite. Bricks low in quartz should show very little expansion in use. The complete

inversion of quartz to cristobalite gives an increase in volume of 13.6% ; the inversion into tridymite gives an increase of 16.8% . The permanent expansion of most silica bricks is only 10 or 11% by volume. The proportion of unaltered quartz may be estimated by grinding the bricks until the powder will pass through a 200-mesh sieve, immersing the powder in a liquid of refractive index 1.51, viewing it through a microscope magnifying 400 diameters and fitted with a camera having a plain glass plate carrying a sheet of tracing paper instead of the usual ground glass screen. The outlines of the grains are traced on the paper and then cut out; those having an index of refraction higher than the liquid are weighed separately; they represent the proportion of quartz present. The grains are next immersed in a liquid having an index of refraction equal to the mean index of either tridymite or cristobalite, whichever is in excess in the specimen of brick being examined, in which case the corresponding grains will be rendered invisible. Thus, if the first test showed 30% of quartz and 70% of cristobalite plus tridymite, and it was judged that there was more cristobalite than tridymite, the grains would be immersed in a liquid with a refractive index of 1.485 in which the cristobalite is invisible. If the grains of quartz plus tridymite left were in the proportions of 60:40, the actual amount of tridymite would be $40 \div 60 \times 30 = 20\%$, and the brick would be composed of quartz 30%, tridymite 20%, and cristobalite 50%. Five determinations should be made on each sample. The error may reach 5% or even higher, as glass and calcium metasilicate have higher indices than quartz, and are therefore included as quartz. When using a commercial mixture of silica rock and lime, the constitution of the bricks produced is dependent on the duration and temperature of the burning; the longer the heating at high temperatures (below 1470° C.) the greater the amount of tridymite present. An all-tridymite brick would be technically ideal, but too costly. Detailed descriptions of 14 bricks made by different American manufacturers and one German manufacturer are given. All these bricks have a porphyritic structure made up of phenocrysts or coarse fragments and ground mass, this structure being entirely due to the method of grinding the material.

—A. B. S.

Silica bricks; Factors influencing the properties of — (I.) A. Scott. Trans. Ceram. Soc., 1918-9, 18, 481—496.

A STUDY of the effects of the impurities in silica bricks on the inversion of quartz to cristobalite and tridymite, and on the properties of silica bricks generally. Vein-quartz was used as the standard on account of its purity, though it is unsuitable for the manufacture of silica bricks; this quartz was mixed with about 2.5% of other material and compressed into bricks which were then fired in a silica brick kiln at cones 11, 16, and 19 respectively, and afterwards examined. The inversion of the quartz is affected by the nature and amount of impurities, the grain-size of the quartz, and the temperature and duration of firing, and is increased if the firing temperature is increased or the period of firing is prolonged, especially at cone 20 (1530° C.). Iron oxide and lime appear to have the greatest influence of all single oxides, whilst alumina has a very small effect. The effect of a second oxide is to facilitate the inversion, especially with iron oxide and either lime or alumina. The best results are obtained when the bonding material forms a relatively fusible mixture with finely divided silica. Carbon appears to have no influence on the inversion. Both the apparent and true sp. gr. vary directly as the firing temperature. The porosity varies irregularly when only one metallic oxide is present, but re-

gularly when two or more are present. After-expansion or permanent increase in volume must be determined by reheating to at least cone 20. Heating to cone 26 for 2 hours appears to complete the inversion of quartz in small test-pieces. Mixtures of silica with lime and iron oxide give the smallest expansions; the next lowest are those with silica and either iron oxide or lime separately. Alumina and titanite oxide both give high values, but if another oxide is present the values are much lowered. The expansion with a fireclay bond is low. The presence of carbon does not greatly alter the after-expansion; in three out of four cases tried the bond containing carbon gave slightly lower values. The liability to spalling may be judged from the ratio of expansion determined by direct measurement to the expansion calculated from the sp. gr. determinations. In general, bonds containing magnesia have less spalling tendency than those containing lime. The presence of carbon, alumina, and fireclay seems to increase the spalling tendency. The greatest reduction in refractoriness occurs on the addition of lime or magnesia or a mixture of lime, iron oxide, and either carbon or titanium oxide. When the only metallic oxide present is lime or magnesia the reduced refractoriness is due to the fusion of the silicate. When alumina or iron oxide is present, or both, the matrix of fine silica and the rock fragments fuse simultaneously, and at the same temperature as pure silica. Fireclay acts similarly. Carbon in the presence of ferric oxide slightly reduces the refractoriness.—A. B. S.

Silica products; Manufacture of —. A. Bigot. Trans. Ceram. Soc., 1918-9, 18, 354-361. (See this J., 1918, 335 A, 695 A.)

Rock crystal, fused quartz, chalk flint, and chalcedony fall to powder on prolonged heating at 1450°-1650° C., and are unsuitable for silica bricks. Quartzites, ferruginous sandstones (3-8% Fe₂O₃), and aluminous rocks, on the contrary, remain compact, even when heated for several days at 1500° C. The behaviour of the quartzites is attributed to the small proportion of clay present, which unites the particles of silica and prevents them from falling apart. The following tests etc. are recommended: complete analysis (there should be 2-5% of alumina); porosity test, i.e., increase in weight after immersion for 24 hours in water; expansion on heating: materials which expand 10% are superior to those which expand 15%; crushing test curve by Bodin's method (this J., 1918, 695 A); compactness of the material after heating to 1710° C.; if it remains compact the expansion, porosity, and loss on heating are determined. The selected rock should be ground in a jaw crusher followed by two pairs of crushing rolls, so as to produce angular grains—the largest of which should pass through a No. 3 or No. 4 sieve—together with a suitable proportion of fine powder. Water and 2% of lime are added to the paste and the mixture pugged for 15-20 mins. The paste is moulded by hand or in hydraulic presses, dried rapidly, raised to 1500° C. in 5 or 6 hours, held at this temperature for 4 hours and then burned at as high a temperature as possible. The glassy mass produced by combination of the lime with the clay and fine silica should not be too fusible or the bricks will not be sufficiently refractory, and will be too sensitive to changes in temperature. Hydraulically pressed bricks are less porous and more durable than those moulded by hand in a small lever press.—A. B. S.

Silica materials; New experiments on —. A. Bigot. Trans. Ceram. Soc., 1918-9, 18, 362.

Pure silica, including fused hyaline quartz, other types of quartz, and chalk flints fall to powder when heated for four hours at 1450° C., and are, therefore, unsuitable for the manufacture of silica

bricks. Silica materials (including some quartzites and quartzose rocks and some dense flints) containing 2-5% of clay, iron oxide, and a small proportion of magnesia and lime do not fall to pieces on prolonged heating, and are suitable for making silica bricks if their expansion at 1710° C. is not too great (about 10%, see preceding abstract). Silica rocks which contain more than 3% Al₂O₃ become too fusible after the addition of 2% of lime, and so are not suitable for the manufacture of silica bricks.—A. B. S.

Silica brick from the roof of an open-hearth furnace. J. E. Stead. Trans. Ceram. Soc., 1918-9, 18, 389-398.

A SILICA brick taken from the roof of an open-hearth furnace at Middlesbrough had been reduced during use from 12×6×3 in. to 12×2½×2½ in. The results of the chemical and microscopical examination of the brick confirmed those obtained by E. Rengade (this J., 1918, 372 A).—A. B. S.

Silica brick from roof of a basic open hearth furnace after 135 charges; Seasoned —. C. S. Graham. Trans. Ceram. Soc., 1918-9, 18, 399-406.

EXAMINATION of a silica brick taken from the transverse centre of the roof of a 45-ton basic open-hearth steel furnace after 135 charges gave results confirming those of Stead and Rengade (see preceding abstract).—A. B. S.

Refractory materials; Standardisation of tests for —. (Part III.) *Penetrative action of slags.* Report of Committee. Trans. Ceram. Soc., 1918-1919, 18, 516-517.

THE method of Nesbit and Bell (Proc. Amer. Soc. Testing Materials, 1918, 18, 345) is recommended. A ring of stoneware is cemented to the firebrick under test by means of a thin film of grog and sodium silicate. The receptacle so formed is filled with slag, and the whole is fired at the desired temperature for the desired time (say, 4 hrs.). The block and its contents are afterwards sawn in half and examined.—A. B. S.

Clay; Osmotic purification of —. W. R. Ormandy. Trans. Ceram. Soc., 1918-9, 18, 327-339.

WHEN a clay slip is subjected to the action of an electric current it collects at one pole in a purer form, and is dried to a considerable extent even when collected under water. The rate of collection is increased by adding a little alkali (or sometimes organic matter) to the slip. Under suitable conditions such treatment produces cakes of ball clay containing only 18% of water. The remarkable dryness of the clay is due to the particles forming a series of capillaries through which the water, travelling in a contrary direction to the clay, is expelled at a considerable velocity. On adding a suitable amount of soda or other suitable electrolyte the positive particles are neutralised and precipitated whilst the negative ones remain in suspension, and so may be run off and separated. The purified slip is then passed into an osmosis machine in which the nearly dry clay is deposited as described. The product is more plastic, and has a greater shrinkage than crude clay, and it sinters at a temperature as much as 300° C. lower.

—A. B. S.

[Earthenware] bodies; Preparation of —. R. L. Johnson. Trans. Ceram. Soc., 1918-9, 18, 321-326.

IT is possible to produce equally good bodies or ware from materials ground in cylinder mills as from those ground in pan-mills, but there are marked differences in cost. For flint of good quality and washed to a fine degree, the cost of grinding

in a pan-mill is no more than for grinding in a cylinder mill producing a material of the same quality. For a coarser, lawned product cylinder grinding is cheaper. Crazing may result if coarser flint replaces a finer one in an earthenware body and glaze. The setting of flint in the ark or blunger may be prevented by finer washing. The construction of the filter-presses used may have a serious effect on the plasticity of the clay. Wooden presses are preferable; they should produce a thin, uniform cake. The cakes should be stored for 24 hours before use. To make a really good white earthenware body it is necessary to develop the plasticity of the material as fully as possible, and this is difficult when a large proportion of non-plastic or milled material is present. Both ball and china clays should be blunged to a thick slip, avoiding unduly prolonged blunging. The flint and stone should be washed very fine and stored as long as possible in a slip state (32 oz. or more per pint). Any scrap material added should be in the form of a thicker or heavier slip than the newly mixed body, as few white scraps as possible being used.

—A. B. S.

Bone china body; Recent research on the —. J. W. Mellor. Trans. Ceram. Soc., 1918-9, 18, 497-509.

BONE china is composed of bone ash (tricalcium phosphate), china clay, and Cornish stone. On grinding the Cornish stone and bone ash with water and on evaporating the water used in preparing the body slip, hydrolysis occurs, and the product becomes plastic. Bone china body cannot be matured by prolonged storage. When bone china body is fired in a kiln the shrinkage is roughly proportional to the temperature, up to the finishing point, after which the body expands on further heating. This expansion is due to the dissociation of the bone ash or its decomposition by carbon, under reducing conditions, with evolution of phosphorus. There is less than 1½% of iron in a bone china body, but in firing the ware it combines with phosphorus from the bone ash forming greenish-blue, blue, or brown iron phosphate. Blue china can readily be made by enclosing charcoal or other organic matter in the sagger in which the ware is fired or by using a bone ash containing sufficient carbonaceous matter. It can also be obtained with bone ash free from carbon and with ware fired wholly in an oxidising atmosphere. In some samples of bone ash the carbonaceous matter burns out readily and at a temperature much below the decomposition point of bone ash; other samples give trouble because vitrification of the ware sets in before all the carbonaceous matter has burnt away; this difference in behaviour appears to be due to the manner in which the bones are calcined. It is shown that the range of white bone china bodies is extremely limited, and that outside this range an extraordinary variety of tints is developed. The smaller the proportion of stone and the greater the proportion of clay in a bone-china body the greater is the tendency to form blue ware. The presence of a high proportion of alkalis thus hinders the conversion of calcium phosphate into iron phosphate. If, however, the proportion of Cornish stone is excessive the body blisters or "bloats." Even the use of a more moist sample of stone may spoil the ware by disturbing the balance essential to white ware, and blue china must have been very frequently produced by assuming that the Cornish stone used contained only 10% of water when, in reality, it contained 20% or more. A greater variation is permissible in the bone ash used than with either stone or clay. When the stone is deficient in feldspar, the blue china is in so critical a condition after passing through the glost oven that oxidation may occur in the enamel kiln with pro-

duction of brown stains of ferric phosphate. Sometimes white iron phosphate in chinaware is so sensitive that it oxidises to the brown form on mere exposure to air. This brown ware is very liable to spit in the enamel kiln (this J., 1917, 550). The variation in the composition of mixtures forming a satisfactory glazed bone china is very limited, due to the fact that glazed ware rich in clay, i.e., glazed blued ware, tends to craze badly, and also to the tendency to blister of ware which is rich in stone and bone ash. Alumina and iron and alkalis appear to facilitate the decomposition of the bone, but they fix the phosphate before it has time to blister. Alkalis also aid in vitrifying the body, but the free quartz in the stone acts as an acid decomposing the bone ash and any aluminium phosphate.

—A. B. S.

Bone ash; Note on apatite substituted for — [in bone china bodies]. N. B. Davis. Trans. Ceram. Soc., 1918-9, 18, 378-381.

CANADIAN mica-apatite containing CaO 55.60%, P₂O₅ 39.24%, SiO₂, Fe₂O₃, Al₂O₃ each 0.72%, MgO 0.35%, Na₂O, K₂O each 0.25%, Cl 0.5% and F 3.10% was substituted for bone ash in four typical china bodies. The mixture showed excellent casting properties and plasticity, but all the pieces were deformed when fired at cone 12 (1350° C.). The greatest translucency was developed at cone 9 (1280° C.).

—A. B. S.

Recuperative furnace. Rosenhain and Coad-Pryor. See I.

Refractories for coke-ovens. Rees. See IIA.

Corrosion of coke-oven walls. Rees. See IIA.

Lead borates. Thompson. See VII.

Mica schist. Boswell. See X.

Zinc furnace refractories. Audley. See X.

Zinc retorts. Scott. See X.

Coagulation of clay. Odén. See XVI.

PATENTS.

Glass articles; Machine for re-melting [fire-polishing] —. M. Mathy, Liège, Belgium. Eng. Pat. 137,660. 27.2.19. (Appl. 4956/19.)

A MACHINE for superficially remelting or fire-polishing glassware consists of a vertical cylinder down which is passed a current of gas heated by surface combustion in a disc of porous refractory material in the upper part of the cylinder. Means are provided for rapidly inserting and removing the glassware.—A. B. S.

Furnace for continuous sheet-glass drawing. M. J. Owens, Assignor to The Libbey-Owens Sheet Glass Co., Toledo, Ohio. U.S. Pat. 1,327,405. 6.1.20. Appl., 18.11.18.

MOLTEN glass flows from a melting tank through a refining tank to a sheet-drawing source. The refining tank is provided with means for retarding the flow of hot gases from the melting tank over the molten glass to the outlet end of the refining tank.—J. W. D.

Glass; Method of staining —. J. W. Hasburg, Chicago, Ill. U.S. Pat. 1,328,833. 27.1.20. Appl., 18.6.19.

GLASS or ceramic ware is decorated by applying a porous ground coat followed by a colouring material which penetrates it, and then firing the ware.

—A. B. S.

Refractory material. E. D. Frohman, Pittsburgh, Pa. U.S. Pat. 1,327,758. 13.1.20. Appl., 26.4.19. The material consists of a mixture of finely-divided

dry fireclay, silicious material, and "sulphite pitch."—L. L. L.

Refractory material and process of making same. T. Sheard, Lakewood, and H. E. Wetherbee, Cleveland Heights, Ohio, Assignors to The Dolomite Products Co., Cleveland, Ohio. U.S. Pat. 1,329,431, 3.2.20. Appl., 19.9.18.

DOLOMITE is calcined, and a portion of the lime present is converted into a solution of calcium chloride, which is separated. The residual solid is suitable for use as a refractory material.—A. B. S.

Plastic [insulating] composition. F. Richard and F. M. Brady, Cleveland, Ohio. U.S. Pat. 1,329,094, 27.1.20. Appl., 2.5.17.

A PLASTIC, insulating composition is prepared by mixing silica and carbon with lesser quantities of alumina and ferrous oxide, together with water and a binding agent, drying the mixture at 100° F. (38° C.), then heating to 800°–900° F. (430°–480° C.) to burn off the carbon, and finally heating to incipient fusion of the ferric oxide, leaving the greater part of the silica unfused.—A. B. S.

Porcelain, stoneware and the like; Process of firing —. E. Enke, Breslau. Ger. Pat. 314,585, 2.11.15.

HIGHLY-HEATED secondary air is introduced under high pressure into the fire-gases as they leave the combustion chamber. The exhaust gases are drawn away exclusively by this hot secondary air so that the furnace works without a chimney draught. The air introduced under high pressure causes a better mixing of the smoke particles, and consequently a better combustion, the latter being also improved by the fact that the secondary air introduces additional heat.—J. F. B.

Drying machine for clay. U.S. Pat. 1,329,082. See 1.

IX.—BUILDING MATERIALS.

Salt mixtures as preservatives for wood against fungus and decay. F. Moll. Z. angew. Chem., 1920, 33, 39–40.

THE author gives a short history of the development of the use of inorganic salts for impregnating wood for preserving it against decay, the attacks of insects and fire. Wolman introduced the use of salts of organic acids and reduced the temperature of treatment. Subsequently he patented the use of salts of heavy metals with sodium fluoride (Eng. Pat. 17,534 of 1906; this J., 1907, 968). He also combined with the use of sodium fluoride that of water-soluble organic compounds known to have antiseptic properties (Eng. Pat. 19,241 of 1906; this J., 1907, 529). Wolman's mixtures can be varied in many ways for different purposes. To fix such substances as phenol, naphthol, and cresol in the wood, a chroming treatment is now employed. The type of mixture frequently used at present contains 70% of sodium fluoride and 30% of phenol-chromium salt compound. A chronological bibliography is given.—E. H. R.

PATENTS.

Cement; Mixing of — and apparatus therefor. N. C. Johnson, New York. Eng. Pat. 127,560, 9.4.19. (Appl. 9030/19.) Int. Conv., 23.5.18.

BATCHES of cement, with or without other ingredients, are mixed with water in an upright bowl which is rotated at such a speed—1500 revs. per min.—as to cause the mixture to be driven centrifugally upwards, outwards, and along the walls of the bowl at a high velocity until it is deflected

by concave baffles and projected in a smoothly-flowing stream to the centre of the bowl. The paths taken by the contents may be varied by altering the shape of the bowl, and the number, shape, and position of the deflectors. The crushing strengths of mortars and concretes prepared as described are stated to be considerably superior to those of similar products mixed by hand.—A. B. S.

Road or like covering material; Composition for use as a dust-proof and waterproof — and the method of making the same. R. Illemann, Glasgow. Eng. Pat. 138,017, 20.6.19. (Appl. 15,534/19.)

A BITUMINOUS composition for surfacing roads is made by adding bituminous oil or tar or a mixture of non-oxidising oil, such as creosote, and pitch to dried stone-dust, heating to remove light volatile oils, and then agitating so as to obtain a uniform mixture. (Reference is directed to Eng. Pats. 1737 of 1871, 17,483 of 1887, 17,153 of 1890, 21,043 of 1905, 3749 and 24,829 of 1906, 9929 of 1909, 25,465 of 1910, and 1455 of 1913 (this J., 1909, 1315; 1911, 1388; 1914, 201.)—A. B. S.

(A, C, D, G—K) *Lumber; Process of drying* —. (B) *Sterilised wood.* (E) *Sapless composite wood.* (F) *Process for treating and drying wood.* F. K. Fish, jun., New York. U.S. Pats. (A) 1,328,505, (B) 1,328,506, (C) 1,328,507, (D) 1,328,655, (E) 1,328,656, (F) 1,328,657, (G) 1,328,658, (H) 1,328,659, (I) 1,328,660, (J) 1,328,661, and (K) 1,328,662, 20.1.20. Appl., (A) 20.9.16, (B) 27.9.16, (C) 18.1.18, (D) 23.4.19, (E) 30.4.19, (F) 21.11.19, (G, J) 22.3.17, (H, I) 8.3.17, (K) 17.12.19. Renewed (A) 9.11.18, (B) 18.4.19, (C) 13.8.19, (G—J) 13.12.19.

(A) Wood is immersed in superheated water under pressure until its soluble contents are dissolved and the wood completely sterilised; it is afterwards dried under conditions which prevent hardening and enable the resins to permeate the individual fibres, whilst "tanning colloidal substances" extracted from the wood are deposited on the fibres. (B) A tanned wood, having its gummy sap-matters removed and its natural preservatives (tannins) uniformly distributed. (C) Wood is immersed in superheated water and then heated so as to dry it without hardening the resins. The wood may be subjected to a vacuum prior to immersion. (D) Timber is subjected simultaneously to live steam under pressure and to a dry heat, and then to a dry heat whilst periodically removing the accumulated moisture. (E) A wood from which the gummy sap-matters have been removed and the individual fibres permanently enlarged by impregnation with the natural preservatives in the wood and with a foreign preservative. (F) Wood which has been sweated and softened by heat is subjected consecutively to a vacuum, steam, a further vacuum, and hot water to remove all soluble constituents. The water is then removed and the wood dried. (G) Wood is heated by radiation, sweated, subjected consecutively to steam under pressure, vacuum, superheated water, a further vacuum, to dry heat at a high temperature, and finally to a moderately-heated moist atmosphere. (H) Wood is subjected to a moderately-heated moist atmosphere without pressure, then to saturated steam until the sappy matters are dissolved; the pressure is then gradually reduced to below atmospheric pressure, and the wood is finally dried at atmospheric pressure without heat. (I) Wood is subjected to live steam under pressure, then to a partial vacuum, and is finally dried in a moderately-heated moist atmosphere at atmospheric pressure. (J) Wood is mixed with a heat-absorbent, then sweated, surrounded by steam, subjected to dry heat, and finally cooled in a moderately-heated moist atmosphere. (K) Wood is placed in a sweating chamber and subjected to a moderately-hot, moist atmosphere; it is then

subjected successively to steam under pressure in an autoclave, to dry heat in an "equalising chamber," and to a moist moderately-heated atmosphere in a cooling chamber.—A. B. S.

Wood; Method of bleaching —. E. Bateman, Madison, Wis. U.S. Pat. 1,329,284, 27.1.20. Appl., 8.9.19.

Wood is bleached by means of a solution of concentrated nitric acid and potassium chlorate.

—A. B. S.

Paint oils for concrete. Eng. Pat. 137,878. See XIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Mica schist for lining cupolas and steel converters. P. G. H. Boswell. Trans. Ceram. Soc., 1918-19, 18, 382-388.

IN America mica schist is used instead of ganister for lining cupolas and steel converters. The material is of Cambrian age, fairly hard, foliated, but fine grained and compact. Microscopical examination shows that the material is a quartz-schist rather than a mica schist; it contains 69% of quartz, a considerable amount of muscovite mica, and some potash feldspar. The rock contains SiO_2 81.55%, Al_2O_3 10.01%, Fe_2O_3 2.21%, $\text{Na}_2\text{O} + \text{K}_2\text{O}$ 3.46%, $\text{CaO} + \text{MgO}$ 0.41%. It is built into the walls of converters and cupolas inside the firebrick casing, the blocks being set with the cleavage edges forming the inner wall. Fireclay is generally used as mortar, but occasionally the crushed rock is employed. The face becomes glazed in use, and it is claimed that slag and clinker do not adhere to it. As somewhat similar quartz mica schists occur in the Moine Series in Scotland, and possibly in Anglesea, the author suggests that they may be used for the same purpose.—A. B. S.

Electric versus open-hearth [steel] furnace practice. E. H. Ballard. Blast Furnace and Steel Plant, 1920, 8, 62.

A COMPARISON of the cost of electric and open-hearth steel made from actual running costs of a 6-ton Héroult electric furnace and a 20-ton oil-fired open-hearth furnace. As the furnaces were only run for comparison over short periods, in working out the costs certain deductions and allowances were made on the actual costs, in order that the figures presented should permit the best comparison, assuming both furnaces were operating under normal and reasonably full-time conditions. A summary of the costs is as follows:—

	Electric furnace.	Open-hearth furnace.
	Net ton.	Net ton.
	\$	\$
Total metal cost	9.52	14.28
Special metals	1.79	3.52
Expense items	21.58	6.60
Molten metal cost in ladle	36.57	32.55

The steel made was an alloy steel, and the metal made in the electric furnace was superior in chemical and physical requirements to the open-hearth steel.—J. W. D.

Electric furnace smelting; Problems in —. F. Holmgren. Trans. Swedish Iron Inst., 1919. Chem. and Met. Eng., 1920, 22, 165-172.

INVESTIGATIONS have been made on existing furnaces in Sweden in order to determine the best

arrangement of electrodes and leads, and the relation between the electrical and metallurgical behaviour of the furnace. A considerable reduction of the losses in the conductors was effected by connecting the transformer leads to adjacent electrodes, thus reducing the resulting electro-magnetic fields. The heat supply at the different electrodes is often irregular, due to an uneven energy supply or to an unsuitable mixture (excess of coal or ore) surrounding the electrode. The electrodes should be controlled individually to attain regular working of the furnace as a whole. Electric furnaces may be operated with any of the standard frequencies now in use, and the output of a furnace may be increased by increasing the current intake by means of larger or a greater number of electrodes. Too high a voltage is not permissible on account of the reducing action on the silica walls of the furnace. 30-40% of the losses from the transformer to the electrode may be traced to faulty contacts. The shorter the leads, the better will be the operation from the electrical point of view.—C. A. K.

Zinc retorts; Microstructure of —. A. Scott. Trans. Ceram. Soc., 1918-9, 18, 512-515.

AN examination of thin sections of various parts of a used zinc retort showed that the outer yellowish-gray portion consists of sporadic quartz grains in an opaque matrix of calcined clay, which in part is replaced by a clear glass containing innumerable microlites of hexagonal zinc orthosilicate with some sillimanite. As the blue portion of the retort is approached these microlites increase in number, the amount of quartz which has been converted into tridymite also becomes greater, and small isotropic octahedra of zinc spinel appear. In the blue material the alumina and zinc form zinc spinel, whilst the silica appears partly as tridymite and partly as zinc and iron silicates. The grog particles have been attacked externally, but retain an unaltered core. The quartz has been wholly inverted to tridymite, and a concentration of willemite needles often surrounds the latter. Two types of zinc silicate are present: the orthosilicate (willemite) and small quantities of the metasilicate. Some sections show brown crystals of fayalite in which part of the iron has been replaced by zinc. When a mixture of zinc oxide and iron pyrites was allowed to impinge on to a silica brick at 1400° C., a thin section of the brick so treated showed that the effect was confined to the matrix, the coarser fragments being unaltered. The quartz in the matrix was completely inverted to tridymite; the iron pyrites had been oxidised to magnetite, and the zinc oxide had formed prismatic crystals of zinc silicate agreeing with Gaubert's mineral (willemite with 10% of iron oxide). The spinel crystals which form one of the chief constituents of used zinc retorts vary in colour from deep blue to light green, and are identical with gahnite, ZnAl_2O_4 .—A. B. S.

Zinc furnace refractories. J. A. Audley. Trans. Ceram. Soc., 1918-9, 18, 468-477. (See this J., 1918, 702 A.)

FURTHER information based on the work of Brandhorst (this J., 1901, 91), Mühlhauser (this J., 1918, 471 A, 658 A; 1919, 42 A), and others is given. It is suggested that in coating retorts with zirconia a series of coatings, each containing more zirconia than its predecessor, should be employed so as to overcome the lack of adhesion of pure zirconia to the retorts.—A. B. S.

Aluminium dross; Analysis of —. H. Hiller. Z. angew. Chem., 1920, 33, 35-36.

ONE gm. of the finely-divided sample is boiled in a flask with 20 c.c. of water to decompose any carbide which may be present, and the flask is then connected with an apparatus in which the hydrogen liberated when the sample is treated with dilute sul-

phuric acid is passed over heated copper oxide; the water formed is collected in the usual form of calcium chloride tube and weighed. Before the determination is commenced the air is displaced from the whole apparatus by means of a current of carbon dioxide passed previously through sodium carbonate solution. The hydrogen is dried by passing it through sulphuric acid and calcium chloride tubes before it enters the combustion tube: 541 grms. of water is equivalent to 542 grms. of aluminium.—W. P. S.

Potassium anhydrotellurate; Application of the reduction of — by zinc to the treatment of tetradymite and to the estimation of tellurium in this mineral. P. Hulot. *Bull. Soc. Chim.*, 1920, 27, 100—101. (See this J., 1920, 189 A.)

The tetradymite is first treated with dilute hydrochloric acid to remove the calcareous gangue, and the residual material is dissolved in hot, concentrated nitric acid, the solution being subsequently evaporated to dryness. The residue is fused with twice its weight of potassium nitrate. The resulting mass contains potassium sulphate, potassium selenate, bismuth oxide, and potassium anhydrotellurate. The two first-named salts are extracted with boiling water and the bismuth oxide with dilute hydrochloric acid. The residue after washing with dilute hydrochloric acid is suspended in the same acid and zinc is added. The whole of the tellurium is deposited in a pure state and may be filtered off, dried, and weighed after the whole of the zinc has dissolved.—W. G.

Soluble metastannic acid. Kreis. See VII.

Silica brick from roof of open-hearth furnace. (1) Stead. (2) Graham. See VIII.

Iron filings. Hindley. See XX.

PATENTS.

Steel smelting furnaces. J. W. Fuller, and Fuller Engineering Co., Catasauqua, Pa., U.S.A. Eng. Pat. 137,574, 4.1.19. (Appl. 278/19.)

THE furnace is constructed with slag pockets, each communicating with an end of the furnace, regenerators, precipitating chambers communicating with the regenerators and the slag pockets, and means for permitting air to enter the regenerators to be preheated. Each regenerator has a lower tier of removable checker-work bricks, and arches supporting the same, superimposed arches with permanent checker-work brick supported upon the latter, and nozzles through which gaseous or pulverulent solid fuel, commingled with preheated air, is introduced at the opposite ends of the furnace.—J. W. D.

Steel; Process of casting —. D. T. Main, Winnipeg, Canada. U.S. Pat. 1,327,398, 6.1.20. Appl., 17.6.18.

BROKEN pieces of high-speed steel are melted in a crucible. 1% of manganese and 0.5% of silicon are added to the molten mass, and the metal is cast in previously prepared moulds.—L. L. L.

Steel ingots; Process for casting —. R. G. Coates, Pasadena, Cal., Assignor to Valley Mould and Iron Corp., New York. U.S. Pat. 1,327,987, 13.1.20. Appl., 20.8.17.

THE steel is poured into shallow moulds and the temperature is then controlled by radiant heat so that the isothermal zone of fusion measured at a mid-transverse cross-section gradually advances inwards and upwards from coincidence with the surfaces of the mould to coincidence with the top surface of the steel mass.—J. W. D.

Iron or steel surfaces; Process of oxidising —. T. Rondelli, Turin, Italy. Eng. Pat. 137,436, 26.3.19. (Appl. 7617/19.)

AN oxide of a metal such as lead, electro-negative to iron, is added to an alkaline bath so as to produce oxidation of the iron or steel surface, with separation of the metal of the oxide, without using an electric current.—J. W. D.

Rust preventing and arresting solution. W. H. Allen, Detroit, Mich. U.S. Pat. 1,329,573, 3.2.20. Appl., 25.9.19.

IRON and steel surfaces are treated with a solution consisting of 25 parts of pure phosphoric acid, 20 parts of glucose, and 45 parts of water.—J. W. D.

Metallurgical reduction process. Norsk Hydro-Elektrisk Kvaestofaktieselskab, Christiania, Norway. Eng. Pat. 126,951, 23.4.19. (Appl. 10,131/19.) Int. Conv., 15.5.18.

CARBON dioxide is passed upwards through a shaft furnace containing metal sulphides heated above 600° C. Carbon monoxide and sulphur dioxide are formed, and the ore is converted to oxide. The mixed gases are passed through scrubbers to remove the sulphur dioxide, and the carbon monoxide is then utilised in a second furnace for the reduction of oxide ore. The exhaust gases from the second furnace may be led through a fresh charge of sulphide ore for the re-production of carbon monoxide.—T. St.

Blast furnaces; Heating of air to be supplied to —. Comp. des Forges et Acieries de la Marine et d'Homecourt, Paris. Eng. Pat. 128,582, 19.6.19. (Appl. 15,471/19.) Int. Conv., 20.6.18.

BLAST-FURNACE gas to be burned in a Cowper stove is mixed as it leaves the top of the furnace with a hydrocarburetted gas or mixture of hydrocarburetted gases, or liquid fuels such as tar or oil, with the object of raising the temperature of combustion and accelerating the exchange of heat in the stove.—J. W. D.

Cupolas. W. H. and D. H. Wood, Birmingham. Eng. Pat. 137,399, 2.8.19. (Appl. 2705/19.)

THE tuyères are arranged in two or more horizontal planes with a separate air belt of pressed steel for each set of tuyères in the same plane. The tuyère-boxes have hinged covers operated from outside the cupola, and from the bottom of each box an overflow pipe closed by a fusible plug passes to the outside of the air belt.—J. W. D.

Annealing furnace. L. Wolffgram, Erie, Pa. U.S. Pat. 1,329,873, 3.2.20. Appl., 24.5.18.

THE main heating chamber is surrounded by an outer chamber which extends over the top, back, and bottom of the main chamber. A heating agent is introduced into the upper part of the outer chamber at the front and also into the main chamber at the front. The two streams of hot gas unite in the outer chamber behind the main chamber, and the gas then passes under the main chamber through a controlling device to the stack.—W. F. F.

Ore briquettes; Manufacture of —. H. G. Hills, Bramhall, and E. Wheeler, Cheadle Hulme. Eng. Pat. 137,626, 1.2.19. (Appl. 2566/19.)

FINELY divided iron sand or other ore is mixed with bituminous matter and the mixture subjected to distillation in open shallow trays, during which the bituminous matter is coked and forms a binding material for the ore; the distillation products are recovered. Lime, salt, or manganese may be mixed with the ore so as to modify the by-products or to produce special qualities in the ultimate metal.—J. W. D.

Coating metal upon metal or other conductive flat surfaces for electrotyping for printing work and other like purposes, specially applicable to sound reproducing records and the like. T. R. Harris, Harlington. Eng. Pat. 137,695, 31.3.19. (Appl. 8023/19.)

THE surface to be coated is cleaned and immersed for about five minutes in a solution containing 1 lb. of arsenious acid, 1 lb. of potassium cyanide, $\frac{1}{2}$ lb. of sodium hydroxide, and 1 lb. of acetic acid per gallon of solution. By passing a current of 4 amps. at 4 volts a thin deposit of arsenic is formed, and this is then coated with nickel to a thickness of about 0.002 in. A strengthening layer of copper is applied by electrolytic means and the plate is stripped off. The nickel facing imparts a greater degree of resistance to wear than is obtained with copper shells.—C. A. K.

Liriviating and washing granular or pulverulent ore; Apparatus for —. G. Gröndal, Djursholm, Sweden. Eng. Pat. 137,930, 12.2.19. (Appl. 3464/19.)

VATS with curved bottoms are placed side by side at different levels. A shaft over each vat carries oscillating arms, to which are connected angular feeding and stirring devices. The ore is transported from one vat to the next higher vat, and the solvents or washing agents, which are admitted by several inlets, pass from vat to vat by conduits in an opposite direction to the ore.—J. W. D.

[Aluminium] *solder; Method of making —.* J. Salm, Assignor to C. L. A. Whitney, Albany, N.Y. U.S. Pat. 1,326,971, 6.1.20. Appl., 3.8.18.

A RELATIVELY small quantity of salt is added to melted aluminium, then a relatively large quantity of tin is added, the heat is allowed to decrease, the product covered to prevent oxidation, and zinc in excess of the aluminium and in considerably less quantity than the tin added.—A. de W.

Solder for aluminium. J. Segura, New York. U.S. Pat. 1,328,694, 20.1.20. Appl., 21.10.19.

A SOLDER for aluminium consists of 75–95 parts of lead, 185–205 parts of tin, 185–205 parts of zinc, 5–25 parts of antimony, and 5–25 parts of "white metal" (Sn 80–90, Sb 5–15, and Cu 3–10%).

—C. A. K.

Smelting system; Hermetic unit —. W. McA. Johnson, Hartford, Conn. U.S. Pat. 1,327,282, 6.1.20. Appl., 18.2.15.

AN intermediate readily oxidisable product is prepared by heating the charge and is transferred out of contact with air from the furnace in which it was formed to the actual smelting furnace.—T. St.

Copper alloy; Surface-coloured article of —. Process of surface-colouring articles of copper alloy. T. C. Merriman, Assignor to Winchester Repeating Arms Co., New Haven, Conn. U.S. Pats. 1,327,400 and 1,327,401, 6.1.20. Appl., 5.8.19.

THE articles to be coloured are dipped in an acid oxidising bath and afterwards in an acid solution containing sulphide and an arsenious compound giving a surface finish of black colour.—J. W. D.

Ores; Process for chemical separation of —. A. T. Elliott, Assignor to L. M. Freeman, Los Angeles, Cal. U.S. Pat. 1,327,536, 6.1.20. Appl., 20.2.18.

THE ore is leached with hydrochloric acid and the separated solution treated to obtain a chloride.

This is then decomposed by heat and the chlorine so obtained is utilised in re-producing the leaching solution.—T. St.

[Copper ores;] *Metallurgical process [for the treatment of —]* L. W. E. Greenawalt, Denver, Colo. U.S. Pat. 1,328,666, 20.1.20. Appl., 5.8.18.

COPPER ores are treated with sulphurous acid in the presence of a solution of copper sulphate. Copper is deposited from the mixed solution of copper sulphate and sulphite by electrolysis, and the regenerated sulphuric acid solution is employed to complete the extraction of copper from the treated ore.—C. A. K.

Brass and similar scrap; Method of melting —. Electric furnaces. Method of and means for melting brass and similar scrap. W. R. Clark, Assignor to Bridgeport Brass Co., Bridgeport, Conn. U.S. Pats. (A) 1,328,712, (B) 1,328,713, and (C) 1,328,714, 20.1.20. Appl., (A) 6.5.18, (B) 31.5.18, and (C) 13.6.18.

(A) THE metal is melted in an electric induction furnace having a secondary channel, and the upper surface is heated by independent means to prevent solidification thereof. (B) An electric resistance element is provided in the furnace chamber above the charge to prevent the chilling of the surface of the charge. (C) A preheated side chamber is attached to the melting furnace, which is fed by a slow, continuous advancement of the metal scrap through the preheating chamber.—C. A. K.

Concentrating ores; Method and apparatus for —. J. M. Callow, Salt Lake City, Utah, Assignor to Pneumatic Process Flotation Co., New York. U.S. Pat. 1,329,335, 27.1.20. Appl., 1.1.14.

AN ore mass consisting of metalliferous and non-metalliferous ingredients is formed into a pulp with water. The pulp then flows through a receptacle and forms therein a pulp body of gradually increasing depth. Air under different pressures is delivered to the pulp at different levels, and the rising bubbles of air adhere to and convey the metalliferous particles to the top of the pulp from which they are caused to flow laterally and are collected.

—J. W. D.

Molybdenum; Recovery of — from its ores. H. A. Doerner, Meriden, Conn. U.S. Pat. 1,329,380, 3.2.20. Appl., 14.4.19.

A CURRENT of chlorine gas is passed through molybdenum-containing materials, such as ores and concentrates, maintained at about 500° C., and the resulting gases are treated for the recovery of molybdenum compounds.—J. W. D.

Arc-welding electrode. R. E. Kinkad, Assignor to The Lincoln Electric Co., Cleveland, Ohio. U.S. Pat. 1,329,530, 3.2.20. Appl., 1.11.19.

FINELY divided metallic iron is supplied along the length of the negative iron electrode for the purpose of supplying iron vapour for the maintenance of the arc during the welding operation.—C. A. K.

Furnaces. Eng. Pat. 137,628. See I.

Blast-furnace gas. Eng. Pat. 137,586. See IIa.

Gas for welding. U.S. Pat. 1,329,400. See IIa.

Flake graphite from kish. U.S. Pat. 1,328,845. See VII.

XI.—ELECTRO-CHEMISTRY.

Electrochemical oxidation of benzaldehyde and benzoic acid. Fichter and Uhl. See XX.

PATENTS.

High-frequency coil [for use in melting refractory material]; Artificially cooled —. E. F. Northrup, Princeton, N.J., Assignor to The Ajax Metal Co., Philadelphia, Pa. U.S. Pat. 1,328,336, 20.1.20. Appl., 18.1.19.

In a process for melting refractory material by electric inductive heating the material is enclosed within a coiled conductor, through which a high-frequency current is passed. The conductor is cooled artificially to reduce its resistance, improve its insulation, and permit closer coupling.
—W. E. F. P.

Distillation of coal. Eng. Pat. 137,572. See IIb.

Sulphuric acid. U.S. Pat. 1,328,552. See VII.

Insulating composition. U.S. Pat. 1,329,084. See VIII.

Electric furnace. U.S. Pat. 1,328,713. See X.

Arc-welding electrode. U.S. Pat. 1,329,530. See X.

Extracting oils. U.S. Pat. 1,326,968. See XII.

XII.—FATS; OILS; WAXES.

Olive oil; Adulteration of — [with tea-seed oil]. J. Cofman-Nicoresti. Pharm. J., 1920, 104, 139—140.

Much of the olive oil now on the market is largely adulterated with tea-seed oil. The commercial varieties of this oil sold at the present time include the Chinese product, expressed from the seeds of *Thea sasanqua* (this J., 1908, 451), Assam oil, and Japanese oil (this J., 1908, 948). A similar oil (tsubaki oil) is expressed from the seeds of *Thea japonica* (this J., 1908, 454). All these oils agree closely in physical and chemical characters, the iodine value ranging from 80.1 to 92.7. Four samples of commercial tea-seed oil showed: Sp. gr. 0.9173—0.9198; saponif. value, 179.5—189.4; and iodine value, 81.7—88.92. Tea-seed oil does not give any coloration in Halphen's or the Baudouin test, but on shaking 10 c.c. with 10 c.c. of a mixture of equal parts by weight of strong sulphuric acid, concentrated nitric acid, and water and keeping the tube for 20 mins. in boiling water the oil layer becomes pink. In the case of mixtures of olive oil with less than 20% of tea-seed oil the pink colour is very faint, but by adding 10% of tea-seed oil a coloration may be obtained. A control test with pure olive oil containing 10% of tea-seed oil should give no pink coloration under the same conditions.
—C. A. M.

Gleditschia (black locust) seeds; Oil from and enzymic properties of —. F. Kryz. Oesterr. Chem.-Zeit., 1919, 22, 167—169.

The seeds of the black locust (*Gleditschia triacanthus*, L.) yielded on extraction with ether a viscous yellow neutral oil, which had a faint odour but no taste. The seeds contained a catalase, a peroxidase, and a lipase. On mixing 2 grms. of the finely powdered and sifted seeds with 20 grms. of neutral olive oil and 10 c.c. of neutralised xylene water, and leaving the mixture at 20° C. for four days, the

amount of fatty acids liberated corresponded to 11.0 c.c. of N/2 sodium hydroxide solution. No better results were obtained by adding acid, manganous sulphate, or powdered nickel to the powdered seed, which additions have been found to stimulate the lipolytic activity of castor seed. The lipase of *Gleditschia* seeds is much less active than the castor seed enzyme.—C. A. M.

Oleates and stearates of certain metals. M. d'A. Albuquerque. Revista Chim. pura e appl., 1916, 1. [Reprint.] 8 pages.

The oleates and stearates of certain heavy metals were obtained by precipitating solutions of their salts with alkali oleate and stearate. The absorption spectra and the oxidation by hydrogen peroxide of the ethereal solutions of the metallic soaps were investigated. (See also J. Chem. Soc., 1920, i., 216.)—W. R. S.

Oxidation of paraffin. Kelber. See IIa.

Lipase of germinated barley. Maestrini. See XVIII.

PATENTS.

Oils; Process of extracting — from fatty substances. G. D. Rogers, Gloucester, Mass. U.S. Pat. 1,326,968, 6.1.20. Appl., 7.3.19.

The material is comminuted, mixed with a saline electrolyte, and the mixture passed through and electrolysed in a conduit provided with electrodes of opposite polarity extending longitudinally there-through, the mixture then being passed through a device for effecting separation of the liberated oil.
—A. de W.

Fatty and oily matters; Process for the extraction of — from wet sludges. L. D. Jones, Philadelphia, Pa., Assignor to The Sharples Specialty Co., West Chester, Pa. U.S. Pat. 1,328,047, 13.1.20. Appl., 11.12.18.

The sludge is treated with a solvent to extract the fatty and oily matter, and the resulting mixture, with the aqueous solution, is treated in a centrifugal apparatus to separate the oily solution from the aqueous solution and from the insoluble materials. The insoluble material is floated off.
—W. F. F.

Oils; Process for producing fatty — of low solubility in petroleum spirit. Chem. Fabr. Flörsheim Dr. H. Noerdlinger, Flörsheim. Ger. Pat. 302,113, 6.3.17.

FATTY oils, such as rape, olive, sesamé, and linseed oils are treated first with air, by known methods, and then with steam, preferably superheated, and under pressure if necessary. The treatment with air is carried out at temperatures between 100° C. and that at which spontaneous heating commences. The products are only slightly soluble in petroleum spirit, and, owing to their high viscosity, are suitable for lubricating purposes.—J. H. L.

Glycerin solutions; Process for obtaining concentrated — from dilute, highly impure solutions. K. Löfl, Berlin. Ger. Pat. 314,446, 6.10.18.

The solutions are treated with alcohol or other solvent for glycerol, and frozen. If, for example, a solution containing 3% of glycerol and up to 10% of mineral salts, besides proteins, carbohydrates, etc., is gradually frozen, ice crystals collect at the surface and the impurities are deposited as a precipitate. Methyl or amyl alcohol, acetone, furfural, or epichlorhydrin may be used instead of ethyl alcohol.—J. H. L.

XIII.—PAINTS ; PIGMENTS ; VARNISHES ; RESINS.

Resinates; Molten metallic —. P. Nicolardot and C. Collignier. Bull. Soc. Chim., 1920, 27, 166—172.

A NUMBER of metallic resinates have been prepared from a sample of colophony by heating it with varying amounts of the corresponding metallic oxides. The apparent acidity, by titration with alcoholic potassium hydroxide, the yield, the amount of the product insoluble in turpentine, and the loss of resin in the process were determined. In all cases 100 grms. of resin was used.

Metallic oxide.		Yield.	Resin lost.	Appa- rent acid value of product.	Amount of product insoluble in turpentine.
Nature.	Weight used.				
	grms.	grms.	%		%
Litharge	20	100	20	65.5	1.80
Litharge	29	119.5	9.5	37.0	2.40
Litharge	31	119.5	11.5	34.2	5.50
Litharge	34	121.5	12.5	28.9	6.70
Quicklime	6	80.5	25.5	47.4	8.80
Slaked lime	7.93	92.5	13.5	60.8	4.30
Slaked lime	12.32	96.0	11.5	29.3	6.40
Manganese dioxide	6.0	90.5	15.5	143.0	4.90
Manganese dioxide	7.5	91.5	16.9	123.4	6.30
Manganese dioxide	8.5	77.0	31.5	101.0	7.10
Cobalt oxide	4.5	87.0	17.5	110.3	22.90
Cobalt oxide	5.5	87.0	18.5	119.6	19.80
Cobalt oxide	6.5	94.5	12.0	118.5	31.50
Cobalt oxide	5.0				
Slaked lime	1.5	84.0	26.0	89.7	9.83
Litharge	10.0				
Manganese dioxide	4.0	103.0	11.0	92.5	4.79
Litharge	9.0				
Manganese dioxide	2.5				
Slaked lime	2.5	97.0	17.9	89.7	1.40

Solubility determinations show that turpentine is the best solvent of these resinates, chloroform and benzene being the next best solvents. The introduction of a small amount of lime materially assists the combination of the other oxides and yields more soluble driers. The loss of resin increases with the temperature to which the mixture of oxide and resin is heated.—W. G.

Antimony trisulphide. De Bacho. See VII.

Colour lakes. Brenner. See XXIII.

PATENTS.

Paints, especially for use as undercoats. W. E. Wright, Bay Village, Ohio, U.S.A. Eng. Pat. 137,475, 5.6.19. (Appl. 14,346/19.)

A READY-MIXED paint consists of lead and/or zinc pigments 100 parts, tung oil 4—14 parts, an oil adapted to retard drying of the tung oil (preferably soya bean oil) 7—35 parts, a drying oil other than tung oil (preferably linseed oil) 5—15 parts, and a volatile thinner 18—24 parts. The use of soya bean oil to retard drying of tung oil is specifically claimed. The pigment is ground in the drying oil other than tung oil, one-half of the thinner is added, and then the mixed tung oil and retarder (soya bean oil) before adding the remainder of the thinner.—A. de W.

Paint oils suitable for cement and concrete; Manufacture of —. F. Boehm, Ltd., and C. A. Reihl, London. Eng. Pat. 137,578, 10.12.18. (Appl. 20,513/18.)

VEGETABLE or animal oil is polymerised to produce a solid, elastic mass which is dissolved in epichlorohydrin, dichlorohydrin, tetrachloroethane, or penta-

chloroethane. A metal linoleate is added to the solution, which is then thinned with turpentine or turpentine substitute. Examples are given in which cotton seed oil and a mixture of soya bean and linseed oils are employed.—W. E. F. P.

Painted or varnished articles; Apparatus for and art of treating [drying] —. C. M. Cumliffe, Assignor to American Blower Co., Detroit, Mich. U.S. Pats. (a) 1,327,313 and (b) 1,327,314, 6.1.20. Appl., 27.12.16.

(A) A DRYING chamber has an air inlet near the top, and means for deflecting the air horizontally so that it will settle down "in mass formation" upon the article within the container. The top wall of the container is spaced vertically from the article to be treated, and the space below the deflector is unobstructed, so that the air meets the article after losing substantially all its velocity. (b) Varnished articles are dried in the lower portion of a chamber as described under (A).—A. de W.

Shellac surrogate and process of producing same. J. R. Köhler, Stockholm. U.S. Pat. 1,327,332, 6.1.20. Appl., 22.4.18.

AMORPHOUS resin acids separated from the natural resin of conifers are combined with glycerin to form a shellac substitute. (See also Eng. Pat. 120,729 of 1918; this J., 1919, 428 A.)—A. de W.

Resinous substances of vegetable origin suitable for impregnating substitutes for textile fibres; Process for preparing —. C. S. Fuchs, Heppenheim. Ger. Pat. 314,418, 14.2.18.

LIGNIC acids obtained by alkaline treatment of vegetable materials are converted into resin-like substances by the action of oxidising agents, e.g., chlorine. The products obtained by the action of halogens on the lignic acids derived from straw are readily soluble in acetone, amyl acetate, and "Propol" (Bayer), and form a hard, tenacious, lustrous surface after evaporation of the solvent. —J. H. L.

Resin-soap produced by heating comminuted wood with sodium carbonate; Process for purifying —. A. Lange, Tomaszow, Poland. Ger. Pat. 314,415, 30.4.18.

THE alkaline liquor obtained by heating comminuted wood with sodium carbonate deposits on cooling a sludge containing resin soap and lignin. This is separated from the supernatant liquor and warmed with sodium hydroxide, whereupon the soap forms large flocks, which are collected and treated with a small quantity of sodium peroxide to oxidise any lignin still present. The liquor containing dissolved lignin is evaporated to recover the excess of soda by crystallisation, and may then be used as a binding agent in the manufacture of briquettes.—J. H. L.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Rubber-like masses; Process for producing soft and hard —. H. Otto Traun's Forschungslaboratorium, G.m.b.H., Hamburg. Ger. Pat. 314,560, 25.2.15.

OILS are mixed with fatty acids in emulsifying (homogenising) apparatus and treated meanwhile with sulphur chloride alone or together with neutral organic liquids. After the reaction is complete the product is washed, neutralised, and heated with or without addition of a filling material. Hard or soft products may thus be prepared from linseed oil, oleic acid, and 10 rubber solution.—J. H. L.

Rubber- or leather-like masses from yeast and formaldehyde; Manufacture of —. H. Blücher, Leipzig-Gohlis, and E. Krause, Berlin-Steglitz. Ger. Pat. 314,728, 20.11.15. (See also Ger. Pat. 280,597; this J., 1916. 430.)

THE materials, consisting of yeast and formaldehyde, with or without the addition of proteins, phenols, oils or glycerin, are mixed with glue in such quantity that the pressed articles remain soft and flexible after cooling. The plastic masses are pressed up with textile fibres or fabrics or with metal parts. The properties of the finished product, especially its hardness, depend principally on the pressure employed. The higher the pressure the more nearly the product approaches in its physical properties to leather. The lower the pressure the more nearly it resembles rubber. If very little or no moulding pressure be employed, soft elastic articles are obtained which have a consistence similar to that of pure Para rubber.

—J. F. B.

Gas for balloons. Ger. Pat. 303,966. See Ha.

XV.—LEATHER; BONE; HORN; GLUE.

Mimosa bark and [tanning] extract; Sugars in —. A. Harvey. J. Soc. Leather Trades' Chem., 1920, 4, 11—13.

FOUR samples of Natal mimosa bark contained 1.5–2.9% of disaccharides and 1.6–1.9% of monosaccharides, which is greater than the sugar content of any other tanning material except pine bark extract. The total amount of sugar in mimosa extracts appears to increase with increasing extraction pressure. Probably hydrolysis of disaccharides to monosaccharides takes place during the manufacture of the extracts, especially when high pressures are employed. Pure unadulterated mimosa extracts yielded 2.2–4.4% of monosaccharides and 3.2–7.2% of disaccharides. The Customs authorities levied duty on extracts containing 4% of disaccharides.—D. W.

Sulphite cellulose; Detection of — in tanning extracts. G. E. Knowles. J. Soc. Leather Trades' Chem., 1920, 4, 13—15.

PURE solubilised quebracho extracts give precipitates with hydrochloric acid, but not with organic acids. Formic acid has been substituted for hydrochloric acid in a modified form of the Procter-Hirst test, and has proved more satisfactory. A positive test with this modified procedure does not necessarily indicate sulphite-cellulose, as synthetic tannins give a similar precipitate.—D. W.

[Tannery] lime liquors; Analysis of —. F. C. Thompson and W. R. Atkin. J. Soc. Leather Trades' Chem., 1920, 4, 15—17.

THE precipitates obtained by neutralising lime liquors with acids or by adding excess of acid have hitherto been assumed to be due to dissolved hide substance. The authors, however, have shown them to be due entirely to dissolved keratinous matter derived from the hair and epidermis, and not to dissolved hide substance. About one-half of the dissolved keratinous matter is precipitated. Solutions obtained by treating gelatin and hide powder with lime, or lime and sodium sulphide, did not give any precipitate on neutralising or adding excess of acid.—D. W.

PATENTS.

Cellulose derivatives; Material [leather etc.] treated with —. J. J. Byers, Brookline, Assignor to Products Syndicate, Inc., Boston, Mass. U.S. Pat. 1,327,197, 6.1.20. Appl., 10.1.17.

LEATHER or other fibrous material, after drying,

is impregnated throughout with a cement which includes soluble nitrocellulose and a non-oxidising oil.—L. L. L.

Strings made from animal tendons and products made therefrom; Process for increasing the elasticity of —. H. Grätzer, Vienna. Ger. Pat. 305,196, 22.10.15. Int. Conv., 29.9.15.

THE threads of other products are drawn through hot water or treated with steam, whereby their elasticity is increased, and the increase is rendered permanent by a further treatment with glycerin. The process may be preceded by a preliminary tanning of the material, but a complete tannage is to be avoided if the elasticity is to be maintained by treatment with glycerin. By tanning the material completely it contracts extensively, acquiring a compact structure and great mechanical strength, exceeding in this respect even leather.

—H. J. H.

Leather-like masses. Ger. Pat. 314,728. See XIV.

XVI.—SOILS; FERTILISERS.

Clay; Coagulation of — and the protective effect of humic acid. S. Odén. J. Landw., 1919, 67, 177—208.

EXPERIMENTS were made with suspensions of two clays of different character, purified by dialysis, and diluted with conductivity water to about 1% concentration. Varying amounts of colloidal humic acid were added to 10 c.c. of each suspension and the effect on coagulation noted. The minimum amount of humic acid required to produce a protective effect on the addition of ammonium nitrate solution as coagulant was 0.065 mgrm. in one case and 0.14 mgrm. in the other. Much larger amounts of humic acid were required to prevent coagulation with barium nitrate and cesium chloride. The with ammonium nitrate and lithium chloride. The protective effect of humic acid appears to be of the same order as that of other colloids, but is greatly influenced by the concentration of the electrolytes present. The effect appears to be specific in character for different clays and electrolytes.

—J. H. J.

Field experiments; Relation between the size of plots and the errors of single observations in —. H. Vageler. J. Landw., 1919, 67, 97—108.

AN experiment was made with 128 field plots each of rye, oats, potatoes, and kale, the size of the plots being 25 sq. m. in the case of rye and oats, 7.5 sq. m. in the case of potatoes, and 5 sq. m. in the case of kale. The crops obtained on each plot were weighed. Various methods of calculating the mean value and the experimental error were worked out, and it is shown that the deductions to be drawn vary according to the method of computation employed.—J. H. J.

Crop production; An experience in —. W. H. Jordan and G. W. Churchill. New York Agric. Exp. Stat., Bull. 465, June, 1919, 1—20.

AN experiment in crop production is described in which crops of corn (maize), oats, wheat, and grass, clover and timothy, have been grown in a 4-year rotation for 16 years. The fertilisers used were farmyard manure, a complete fertiliser, and an acid phosphate (superphosphate) with a minimum amount of nitrogen, while two plots received no fertiliser. The largest crop was given by the farmyard manure, closely followed by the complete fertiliser; these two plots gave a 56% increase (dry weight) over the plots without fertiliser. The plot with the incomplete fertiliser gave a 33% increase over the unmanured plots. The grass plots growing

clover gave a larger yield of dry matter than those growing timothy grass, although the timothy maintained as good yields as the clover on the unmanured plots. Analyses of the soil were made, but no relation was found between the results of a soil analysis and the fertility. The cheapest fertiliser as regards crop value was superphosphate with a trace of sodium nitrate added.—J. H. A.

Nitrification of barnyard manure-nitrogen in arable soil; Influence of time on the —. C. Barthel and N. Bengtsson. *Soil Sci.*, 1919, 8, 243—258.

THE addition of calcium carbonate to neutral or acid cultivated soils does not cause any favourable influence on the nitrification of the nitrogen in applied farmyard manure, but, on the contrary, where excessive dressings (equivalent to 2% of calcium oxide) were applied an impeding effect was noticed. The authors have examined various methods for the estimation of ammonia-nitrogen in soils, and conclude that none of them permits of an accurate quantitative extraction of ammonia from any and every soil. Boussingault's method by direct distillation with magnesium oxide is probably the most satisfactory.—W. G.

Guano from Sardinia. M. Gina. *Gazz. Chim. Ital.*, 1919, 49, II., 246—249.

THE occurrence of veins of tricalcium phosphate, resembling ordinary phosphorite, in the bat guano from the Borutta grotto at Sassari, Sardinia, is regarded as confirmation of the view that many phosphatic deposits owe their origin to deposits of guano. (See also *J. Chem. Soc.*, 1920, i., 276.) —T. H. P.

Moisture in solid substrata; Relation of — to physiological salt balance for plants and to the relative plant-producing value of various salt proportions. J. W. Shive. *J. Agric. Res.*, 1920, 18, 357—378.

IN sand cultures varying the moisture content from 40 to 80% had no effect on the optimum physiological balance of the nutrient solutions for production. Good physiological balance and optimum total concentration of a nutrient solution are not the only factors, as the actual plant-producing value of any fertiliser treatment is largely determined by the moisture conditions of the substratum. The highest yields of tops and roots and high transpiration rates were obtained with a moisture content of 60%.—W. G.

Lime; Effect of — upon the sodium chloride tolerance of wheat seedlings. J. A. Le Clerc and J. F. Breazeale. *J. Agric. Res.*, 1920, 18, 347—356.

VERY small amounts of calcium oxide or sulphate overcame the toxic effects of sodium chloride or sodium sulphate. Magnesium sulphate and barium chloride were slightly antagonistic to sodium chloride. Potassium chloride, sodium nitrate, sodium phosphate, ferric chloride, and alum had no effect on the toxicity of sodium chloride. (See further *J. Chem. Soc.*, Apr., 1920.)—W. G.

Cereal seeds; Treatment of — by dry heat. D. Atanasoff and A. G. Johnson. *J. Agric. Res.*, 1920, 18, 379—390.

BARLEY, wheat, rye, and oat seeds, especially when of good quality and well-dried, are able to withstand exposure to dry heat at 100° C. for thirty hours. Seed infections from bacterial blight of barley (*B. translucens*) and the bacterial blight of oats (*Pseudomonas arena*) may be both eliminated by this treatment. A number of seed-borne fungoid diseases such as wheat scab (*Gibberella saubinetii* and *Fusarium* spp.), primary infections only, and spot-blotch of barley (*Helminthosporium sativum*)

are practically eliminated by this dry heat treatment, and other diseases such as net-blotch (*H. teres*), stripe disease (*H. gramineum*) of barley, and *Helminthosporium* blotch of oats (*H. avenae-sativae*), as well as loose smut of barley and smuts of oats, are also markedly reduced without materially injuring the germination of the seed. —W. G.

Plant juices; Factors affecting the acidity or hydrogen-ion concentration of —. C. B. Clevenger. *Soil Sci.*, 1919, 8, 227—242.

CERTAIN factors must be taken into account in determining the hydrogen-ion concentration of plant juices (see page 249 A). The expressed juice usually becomes more acid on standing, while plants when cut and allowed to stand some time before the juice is expressed usually become more alkaline. Further, the acidities of the leaves, stems, and roots show marked variations over a 24-hour period. The acidity of the leaves and stems is highest in the morning and decreases during the day, while the acidity of the roots is highest during the day. The acidity of the soil also influences the acidity of the different parts of plants grown on the soil. Oats, buckwheat, soya beans, and cow peas were grown on acid soils and on the same soils after liming, and it was found that, with the exception of buckwheat, the tops of the plants grown on the limed soil were usually more acid than those grown on the unlimed soil. The acidity of the roots of the plants appeared to be correlated with that of the soil. The type of fertiliser also appears to have some influence on the acidity of the plants, as is roughly indicated by comparative trials with sodium nitrate and ammonium nitrate.—W. G.

Plant juices. Clevenger. See XXIII.

Bacteria. Conn and Breed. See XIXB.

PATENT.

Ammonium nitrate fertilisers. Norsk Hydro-Elektrisk Kvaestofaktieselskab, Christiania, Norway. Eng. Pat. 129,974, 25.6.19. (Appl. 16,021/19.) Int. Conv., 19.7.18. (See also Eng. Pat. 117,823; this J., 1918, 556 A.)

AN easily decomposable phosphate is finely ground and 1 part of the powdered substance is mixed with 2—10 parts of ammonium nitrate. The mixture is moistened with water, and is then heated with continuous stirring until it becomes granular. —J. H. J.

XVII.—SUGARS; STARCHES; GUMS.

[*Sugar cane milling; Colloidal water, apparent and real imbibition, and the coefficient of mixing* [in —]. C. Müller. *Bull. Assoc. Chim. Sucri.*, 1919, 37, 118—121.

THE fact that successive crushings of sugar cane yield juices of decreasing sugar-content and that the juice remaining in the bagasse is poorer in sugar than the last mill juice, has been attributed to the presence, in cane, of "colloidal water" held by certain constituents and distinct from the saccharine juice. Methods of calculating the proportion of "colloidal water," the imbibition, i.e., the degree of dilution of the extracted juice corresponding to the use of a given quantity of maceration water, and the "coefficient of mixing," i.e., the sugar: water ratio in the last mill juice, expressed as a percentage of the sugar: water ratio in the final bagasse, are illustrated by examples. The "coefficient of mixing" indicates the efficiency of maceration. It may be as low as 43 when excessive amounts of maceration water are used, e.g.,

35—36 of the weight of the cane, whilst coefficients of 68—70 may be obtained by well-conducted maceration with only 18—20% of maceration water (referred to cane), and without any diminution in the yield of sugar extracted.—J. H. L.

New hydrazones of some monosaccharides [*m*-tolylhydrazones of *l*-arabinose, rhamnose, fucose, *d*-galactose, and *d*-mannose]. A. W. van der Haar. Rec. Trav. Chim., 1920, 39, 191—193.

With *m*-tolylhydrazine, arabinose, rhamnose, fucose, and *d*-galactose gave well-defined hydrazones; *d*-mannose gave an amorphous hydrazone; the hydrazone from dextrose could not be obtained pure and xylose and levulose gave negative results. (See further J. Chem. Soc., Apr., 1920.)—W. G.

Inversion of sucrose. André. See XIXa.

Liquefaction of starch. Bettinger. See XVIII.

Inulin in artichoke tubers. Colin. See XIXa.

XVIII.—FERMENTATION INDUSTRIES.

Starch; Determination of the value of different acids for the liquefaction of —. P. Bettinger. Bull. Assoc. Chim. Sucr., 1919, 37, 126—131.

In distilleries which employ Mucors as saccharifying agents, raw cereal mashers are commonly cooked under pressure in presence of a small quantity of hydrochloric acid to effect liquefaction of the starch. Experiments described indicate that in order to produce satisfactory liquefaction in mashers containing 1000 kilos. of ground cereal and 40 hectolitres of water, by heating at 120° C. for 30 mins., the following quantities of ordinary commercial hydrochloric acid must be added:—12 litres for rice mashers, 14 litres for maize, manioc, or millet mashers, and 16 for barley mashers. Chemically equivalent amounts of sulphuric acid produce less complete liquefaction under similar conditions, whilst oxalic, tartaric, and acetic acids act much more feebly in the order given. A part of the added acid is neutralised by alkaline constituents of the mash, and a further part (in the case of mineral acids) is replaced by weaker acids displaced from their salts. If allowance is made for these factors the liquefying activities of the various acids may be accounted for by their relative degrees of ionisation.—J. H. L.

Enzymes. I. Amylase of germinated barley. D. Maestrini. Atti R. Accad. Lincei, 1919, 28, II., 393—394.

THE amylase of germinated barley may be extracted by means of distilled water, but a more active extract is obtained if the water contains 0.003 mol. % of acetic acid; if a highly-active solution is required the extraction should occupy at least 6 hours. By this extract commercial soluble starch is highly degraded, even in a neutral medium, but barley, potato, or maize starch is not converted unless first gelatinised and unless the medium has a certain acidity, namely, 0.003 mol. % of acetic acid. The nature of the starch is without appreciable influence on the amylolytic activity. Hydrochloric and acetic acids activate the amylolytic decomposition to similar degrees. Potassium hydroxide in the concentration 0.003 mol. % paralyses the amylolytic activity in 10 hours. The yield of reducing sugar resulting from the action of the amylase has its maximum value at about 45° C., and diminishes to zero at about 70° C.—T. H. P.

Enzymes. II. Protease and lipase of germinated barley. D. Maestrini. Atti R. Accad. Lincei, 1919, 28, II., 456—458.

THE protease of germinated barley cannot be extracted by water. If distilled water is used, the barley-water emulsion shows no proteolytic activity, but the latter becomes marked if the water contains 0.003 mol. % of acetic acid. If very high enzymic activity is desired, the contact of the meal with the acidified water must occupy at least 6 hours at 20°—30° C. If the emulsion is rendered neutral towards litmus by means of potassium hydroxide and then acidified with hydrochloric acid to the extent of 0.003 mol. %, its proteolytic activity is diminished; the activity is also greatly lowered if 0.003 mol. % of potassium hydroxide is dissolved in the neutralised emulsion. The enzyme acts best at 45°—50° C., and is destroyed at 52°—55° C. Germinated barley contains also a lipolytic enzyme, the presence of which is demonstrable only in the emulsion and not in the filtrate. Emulsions prepared with distilled water exhibit only very feeble lipolytic activity, whereas those made with 0.003 mol. % acetic acid solution are highly active. When treated with this enzyme for 48 hours at 31°—40° C. sweet almond oil shows an acid value, expressed in mgrms. of potassium hydroxide, somewhat greater than 3 and an ester value which falls to 150. At the concentration 0.003 mol. % hydrochloric acid weakens the action of the lipase. The optimal temperature is about 45° C., and the temperature at which the enzyme is destroyed about 55° C.

T. H. P.

Glycerophosphatase of seeds. A. Nemeš. Bull. Soc. Chim., 1920, 27, 153—158.

It has been shown (Biochem.-Zeits., 1919, 93, 94) that glycerophosphatase is very widespread in plant cells and in the tissues of ungerminated seeds. The author finds that this enzyme acts best in a medium having an acidity equal to 0.06N, and that the reaction is bimolecular.—W. G.

Yeast cells; Behaviour of certain dyes towards —. H. von Euler and N. Florell. Arkiv Kem. Min. Geol., 1918—1919, 7, No. 18, 1—27.

DYES are absorbed by yeast in two ways. Some dyes penetrate the cell and are retained therein, others are adsorbed on the surface of the cell. In the former case the cell shows the stain under the microscope; in the latter the stain can only be detected in reflected light. The surface adsorption is the more common phenomenon of the two, and is controlled to a large degree by the previous preparation, especially washing, of the yeast. The absorption in both cases is influenced by such factors as the concentration of the dye, temperature, hydrogen ion concentration, and the presence of a fermentable sugar in the solution. The staining of the yeast with Chrysoidine (diaminoazobenzene hydrochloride), Janus Blue (safraninazo- β -naphthol hydrochloride), Ponceau 3R (sodium salt of cumidineazonaphtholdisulphonic acid), and Methylene Blue impairs its fermenting capacity.—S. S. Z.

Invertase and fermentation enzymes in a top yeast. H. von Euler and E. Moberg. Arkiv Kem. Min. Geol., 1918—1919, 7, No. 12, 1—17.

THE production and the activity of the invertase of a top yeast was only influenced to a small extent by a protoplasmic poison such as chloroform. In the top yeast investigated it was not possible to augment the invertase by Euler's method to the extent previously done in bottom yeasts. Comparative experiments showed that on drying practically the entire invertase content was retained by the dried yeast. The amount of alcohol and carbon dioxide produced from a certain quantity of sugar

in alkaline solution by dried and fresh yeast was approximately the same, and was not influenced by the addition of co-enzyme. The co-enzyme previously heated with sodium hydroxide became, however, more active. The author concludes that besides the co-enzyme and phosphate, fermentation in alkaline solution requires another activator, most probably of the nature of an alkali salt.—S. S. Z.

Yeast; Growth of — in alkaline solutions. Chemical enzyme studies. H. von Euler and O. Svanberg. Arkiv. Kem. Min. Geol., 1918–1919, 7, No. 11, 1–13.

THE maximum alkalinity of the medium in which a Frobberg bottom yeast grew was between p_H 7.7 and p_H 8. A distillery top yeast (S.B. II.) showed decided growth at p_H 7.3. The maximum, however, tolerated by growing yeast depends to a great extent on the quantity of cells seeded. The maximum alkalinity for *Saccharomyces ellipsoideus* and *Pseudosaccharomyces apiculatus* was p_H 7.9 and p_H 7.6 respectively. The highest limit of alkalinity tolerated by *Aspergillus niger* was found to be p_H 9.—S. S. Z.

Methyl and ethyl alcohols; Differentiation of —. T. Sabalitschka. Pharm. Zentr., 1920, 61, 78–79.

THE author agrees with Pannwitz (this J., 1920, 38A) that the copper sulphate test is useless in the presence of water. The test may, however, be of some use for the detection of methyl alcohol in absolute alcohol.—W. P. S.

Sulphite spirit. Kuhn. See V.

Bacteria. Conn and Breed. See XIXb.

Yeast nucleic acid. Levene. See XX.

PATENTS.

Yeast; Preservation of —. J. R. Robertson, Stockton-on-Tees. Eng. Pat. 137,131, 21.1.19. (Appl. 1845/19.)

LIGN yeast is mixed with powdered peat or peat moss, sawdust or wood fibre and charcoal; instead of peat or peat moss, powdered spent tannery bark may be used. The ingredients are mixed in the dry form and consist approximately of powdered peat, peat moss or powdered bark, 45 lb.; wood flour or fine sawdust, 45 lb.; powdered charcoal, 10 lb. About 2–3 lb. of the mixed powder is stirred into 1 gallon of liquid yeast until the mixture becomes fairly thick; it is then wrapped in suitable fabric and pressed.—J. F. B.

Yeast; Process for precipitating — in fermented liquids, especially fermented worts of aero-yeast factories. Verein der Spiritusfabrikanten in Deutschland, Berlin. Ger. Pat. 300,661, 27.6.15.

THE fermented wort is rendered slightly alkaline to cause the yeast to flocculate.—J. H. L.

Beer; Process and apparatus for the production of —. K. Plesch, Hohenaschau. Ger. Pat. 311,461, 24.8.16.

SEE this J., 1919, 86A; before the final boiling of the filtered wort the cold water hop extract previously made is added.—J. H. L.

Glycerin solutions. Ger. Pat. 314,446. See XII.

Rubber- or leather-like masses from yeast. Ger. Pat. 314,728. See XIV.

Yeast. Ger. Pat. 299,649. See XIXa.

Tobacco. Ger. Pat. 311,391. See XX.

XIXA.—FOODS.

Mixed meats, sausages, etc.; Determination of added water in —. R. Leclercq. Ann. Valsil., 1919, 12, 356–367.

ACCORDING to French law sausages and similar products must not contain more than 75% of water (calculated on the fat-free substance); in the case of smoked products the maximum limit for water is 85%. Since 81% of water has been found to be present in meats, the author considers that the limit of 75% is too low, and that, in any case, the quantity of water in meats varies so widely that results of analysis may lead to erroneous conclusions as to the presence of added water.

—W. P. S.

Potatoes; Manufacture of so-called compressed —. G. Wiegner and H. Mehlhorn. J. Landw., 1919, 67, 151–170.

COMPRESSED potatoes are prepared by pressing out the greater part of the water from fresh potatoes and then drying the product. 100 kilos. of fresh potatoes after pressing yields 22.5 kilos. of compressed potatoes containing 12% of moisture. The product has a starch value of 71%, and contains 15% of crude protein or 0.08% of pure albumin. By direct drying, without compression, 100 kilos. of fresh potatoes yields 25.2 kilos. of product with 12% of moisture. This product has a starch value of 69%, and contains 6% of crude protein or 2.3% of pure albumin. Detailed analyses of the chemical composition of compressed potatoes are given.

—J. H. J.

Fat-soluble vitamins. Comparative nutritive value of white and yellow maize. H. Steenbock and P. W. Boutwell. J. Biol. Chem., 1920, 41, 81–96.

THE occurrence of yellow pigment and the growth-promoting property attributed to the presence of the fat-soluble vitamins seem to be intimately associated in the maize kernel.—J. C. D.

Inulin in artichoke tubers; Origin and transformation of —. H. Colin. Bull. Assoc. Chim. Sucri., 1919, 37, 121–126.

IN the Jerusalem artichoke (*Helianthus tuberosus*) the formation of inulin takes place in the stem and is completed in the tubers, the raw material consisting mainly of dextro-rotatory carbohydrates supplied by the leaves. The inulin-content of the tubers remains practically constant from August to November, but afterwards diminishes. (See also J. Chem. Soc., Apr., 1920.)—J. H. L.

Sucrose in orange juice; Inversion of —. G. André. Comptes rend., 1920, 170, 292–295. (See this J., 1920, 201A.)

THE hydrolysing action of the citric acid present in orange juice on the sucrose also present is very marked at the ordinary temperature. The action of the invertase present is also marked, but much less intense than that of the citric acid.—W. G.

Plant juices. Clevenger. See XVI. and XXIII.

Indole. Zoller. See XXIII.

PATENTS.

Bread, cakes, and the like; Manufacture of —. W. H. Stephenson, Rock Ferry, Cheshire. Eng. Pat. 137,365. (Appls. 132, 7.1.19 and 13,820, 31.5.19.)

DORTON for bread making is improved by adding to the flour a mixture of one part of ammonium chloride and 5–7 parts of ammonium sulphate, in the proportion of 1 oz. to the sack. If the flour is

made from a hard wheat, a mixture of 1 part of ammonium sulphate to 4 parts of chloride is used.
—J. H. J.

Food; Process of treating —. A. Babendreer, Ocean Springs, Miss., Assignor to Whole Grain Wheat Co., Phenix, Ariz. U.S. Pat. 1,327,220, 6.1.20. Appl., 17.9.17.

A CLOSED receptacle is filled with matured grain and liquid, and is heated with continuous movement until the grain is cooked. The volume of liquid used is adjusted so that at the completion of the process the grain almost completely fills the receptacle.—J. H. J.

Milk; Process of treating —. H. Buel, Seattle, Wash. U.S. Pat. 1,327,308, 6.1.20. Appl., 3.4.16.

A CULTURE of lactic acid bacteria is added to sterilised milk, and the milk is incubated until a bacterial count shows the desired number of organisms per c.c., and until a sufficient degree of acidity is produced to restore to the milk its flavour and digestibility. The milk is then maintained under the conditions obtaining for new milk.—J. H. J.

Yeast for fodder; Production of —. Verein der Spiritus-Fabrikanten in Deutschland, Berlin. Ger. Pat. 299,649, 2.4.15.

YEAST is grown, by the aeration process, in suitably acidified liquids containing sugar and human or animal urine, to which may be added mineral salts to supply phosphoric acid, lime, etc. The nitrogen of urine is directly assimilable by yeast (cp. Bokorny, this J., 1917, 1284), but if necessary it may first be converted into ammonium carbonate by a preliminary bacterial fermentation. In place of sugar, molasses or products such as brewers' grains, distillers' wash, or waste from starch factories may be used after saccharification with sulphuric acid.—J. H. L.

Drying apparatus. Eng. Pat. 123,984. See I.

Cellulose from peat etc. Ger. Pat. 314,712. See V.

XIXB.—WATER PURIFICATION; SANITATION.

Bacteria; Use of the nitrate-reduction test in characterising —. H. J. Conn and R. S. Breed. New York Agric. Exp. Stat., Tech. Bull. 73, June, 1919. 21 pages.

THE value of the reduction of nitrate-broth test was investigated with the colon group of bacilli. It was found that at least 0.5% of peptone must be present in the medium to ensure vigorous growth. Under these circumstances all members of the group rapidly reduced nitrate to nitrite. With *B. cereus* some strains were found which constantly failed to act on nitrates, while others were inconsistent. *B. fluorescens* required to be in vigorous growth before reducing nitrates consistently, and also no other source of nitrogen must be present. *B. caudatus* reduced nitrate to nitrite, which was converted into ammonia as fast as produced. The general conclusion is drawn that the test can only be done on media free from other sources of nitrogen, and that it is too liable to variation to make it useful for routine work.—J. H. J.

PATENT.

Fatty matter from wet sludges. U.S. Pat. 1,328,917. See XII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Yeast nucleic acid; Structure of —. P. A. Levene. J. Biol. Chem., 1920, 41, 19–23.

THE preparation of crystalline cytidinephosphoric acid is described. The mononucleotides tend to form mixed crystals which may, on analysis, be mistaken for polynucleotides. (See further J. Chem. Soc., 1920, i., 259.)—J. C. D.

Indole; Influence of hydrogen ion concentration upon the volatility of — from aqueous solution. H. F. Zoller. J. Biol. Chem., 1920, 41, 37–44.

THE most rapid volatilisation of indole from aqueous solutions occurs when the solution possesses a hydrogen ion concentration of from $p_H=8.0$ to $p_H=10.5$.—J. C. D.

Salvarsan and neosalvarsan; Detection of arsenic in —. Utz. Pharm. Zentr., 1920, 61, 39–42.

ABOUT 0.05 grm. of the sample is dissolved by warming with 15 drops of concentrated sulphuric acid and a few crystals of ammonium persulphate are added from time to time until the mixture remains colourless; the solution is then diluted with a small quantity of water and 1 c.c. of stannous chloride solution is added. The solution soon becomes yellow in colour and later the arsenic forms a brown flocculent precipitate. The organic matter may also be destroyed by heating the sample with concentrated hydrogen peroxide solution and a small quantity of ferrous sulphate before the arsenic is detected by means of the stannous chloride test. Another test consists in treating a portion of the substance with hydrochloric acid in a platinum basin and adding a fragment of zinc; a brown stain appears on the platinum. If a small quantity of salvarsan or neosalvarsan is dissolved in water, treated with concentrated sodium thiosulphate solution, and the mixture acidified with hydrochloric acid, a white turbidity appears almost at once, and this, after a time, is coloured bright yellow owing to the formation of arsenic sulphide. The latter may be collected and identified by the usual tests.—W. P. S.

Benzaldehyde and benzoic acid; Electrochemical oxidation of —. F. Fichter and E. Uhl. Helv. Chim. Acta, 1920, 3, 22–39.

THE electrochemical oxidation of these substances at platinum anodes has been studied with particular reference to the products formed in minor quantity. Toluene is converted successively into benzyl alcohol and benzaldehyde; the latter is transformed into dibenzylidene peroxide hydrate.

$C_6H_5CH(OH).O_2.(HO)CH.C_6H_5$, which converts unchanged benzaldehyde into benzoic acid. A portion of the latter yields quinol, another part gives successively salicylic acid, 2,5-dihydroxybenzoic acid, and 2,4,5-trihydroxybenzoic acid, whilst a third portion gives rise to *p*-hydroxybenzoic acid, which yields quinone on the one hand and 3,4-dihydroxybenzoic acid and 2,4,5-trihydroxybenzoic acid on the other hand. These products are also susceptible to further oxidation. (See also J. Chem. Soc., 1920, i., 234.)—H. W.

Furfural; Catalytic reduction of —. H. Pringsheim and H. Noth. Ber., 1920, 53, [B], 114–118.

THE reduction of furfural by Sabatier's method appears to follow three courses concurrently: (i.) reduction of the aldehyde group as far as methyl; (ii.) elimination of the aldehyde group with the formation of furan, and hydrogenation of the ring in both cases (i) and (ii.); (iii.) rupture of the ring and formation of pentan- β -ol and methyl propyl ketone.—J. C. W.

Ethers; Catalytic preparation of — by the dry way. A. Mailhe and F. de Godon. Bull. Soc. Chim., 1920, 27, 121—126.

THE method previously described (this J., 1919, 9624) for the preparation of ether from alcohol, calcined alum being the catalyst, has been successfully used for the preparation of a number of simple and mixed aliphatic ethers from the corresponding alcohols, up to isoamyl alcohol. Isopropyl and isobutyl alcohols only gave very small yields of the corresponding ethers.—W. G.

Aldehydes and ketones; General method of preparation of —. Dehydrogenation of primary and secondary alcohols by catalytic oxidation. C. Moureu and G. Mignone. Comptes rend., 1920, 170, 258—261.

FINELY divided silver, deposited on asbestos by precipitation from silver nitrate with formaldehyde, is an excellent catalyst for the oxidation of primary and secondary alcohols to aldehydes and ketones respectively. The vapour of the alcohol mixed with slightly less than the theoretical amount of air is passed over the catalyst at 230°—300° C. In some cases the action is so intense as to result in very marked local rise in temperature of the catalyst, with consequent destruction of some of the products of oxidation. In this case it is better to conduct the operation in two stages, first passing the alcohol vapour mixed with half the requisite volume of air over the catalyst, and then mixing the issuing vapours with the rest of the air and passing them over a second lot of the catalyst. Yields varying from 60—96% of theory were obtained from methyl, ethyl, butyl, amyl, allyl, benzyl, cinnamyl, isopropyl, and secondary butyl alcohols by this method.—W. G.

Iron filings for pharmaceutical use; Analysis of — H. Hindley. Pharm. J., 1920, 104, 117.

DETERMINATION of the iron in iron filings by the official B.P. method for *ferrum reductum*, in which the iron is made to react with copper sulphate and the resulting ferrous sulphate titrated with potassium permanganate solution, gives concordant and high results. This appears to be due to the presence or formation of hydrogen, which reacts with the phosphorus in the iron to form hydrogen phosphide. By prolonged boiling, however, the hydrogen phosphide is eliminated and accurate results are obtained.—C. A. M.

Reactions of calcium hydride. Reich and Serpek. See VII.

Indole. Zoller. See XXII.

PATENTS.

Esters of ethylenic halogenhydrins; Manufacture of —. Soc. Chim. des Usines du Rhône, Paris. Eng. Pat. 128,911, 7.6.19. (Appl. 14,519/19.) Int. Conv., 26.6.18.

ETHYLENIC halogenhydrin esters of the general formula $R\cdot COOCH_2\cdot CH_2\cdot X$, where R is an alkyl or aryl group, and X a halogen, are prepared by the interaction of ethylene oxide and an acidyl halide in equi-molecular proportions, at a suitable temperature depending on the particular acid chloride or bromide used. The reaction, which is extremely regular and gives quantitative yields, is conveniently brought about by passing gaseous ethylene oxide through the acidyl halide at atmospheric pressure. Examples are given of the preparation of β -chloroethyl acetate from acetyl chloride at its boiling-point, β -chloroethyl chloroacetate from chloroacetyl chloride at 100° C., β -chloroethyl benzoate from benzoyl chloride at 190°—240° C., β -chloroethyl *p*-nitrobenzoate from *p*-nitrobenzoyl chloride at 190°—240° C., and

β -bromoethyl acetate from equimolecular proportions of acetyl bromide and ethylene oxide, kept for several hours at ordinary temperature.—G. F. M.

Nitrites; Manufacture of aliphatic —. P. R. de Wilde, Geneva, Switzerland. Eng. Pat. 131,304, 29.9.19. (Appl. 23,881/19.) Int. Conv., 25.9.18.

NITRATES of aliphatic alcohols are produced by the reducing action of sulphur dioxide on solutions of the alcohols in pure or diluted nitric acid. *Example*—74 grms. of isobutyl alcohol dissolved in 100 grms. of nitric acid (10° B., sp. gr. 1.384) is treated with gaseous sulphur dioxide until the odour of the gas becomes apparent. A yield of 85 grms. of isobutyl nitrite is obtained. Alternatively, a saturated alcoholic solution of sulphur dioxide may be mixed with the nitric acid, a similar result being obtained.—G. F. M.

3,6-Diaminoacridine; Manufacture of —. W. P. Thompson, Liverpool. From Poulenc Frères and R. Meyer, Paris. Eng. Pat. 137,214, 1.5.19. (Appl. 10,797/19.)

m-PHENYLENEDIAMINE is melted with formic acid or oxalic acid and glycerol or other polyhydric alcohol and a condensing agent, such as zinc or calcium chloride, and diaminoacridine is isolated from the resulting solid mass by treatment with water and ammonia, and may be purified through its sulphate, which is slightly soluble in very dilute sulphuric acid. *Example*—6 kilos of *m*-phenylenediamine, 7 kilos of crude oxalic acid, 10 kilos of fused zinc chloride, and 10 kilos of industrial glycerin (28° B., sp. gr. 1.24) are heated at 130° C., and when evolution of carbon dioxide has ceased the temperature is raised to 150°—170° C. and maintained so for 2 hours to complete the reaction.—G. F. M.

Pyrocatechin [protocatechuic] aldehyde; Preparation of —. L. Schmidt, Munich, Germany. U.S. Pat. 1,326,973, 6.1.20. Appl., 31.3.17.

PIPERONAL is treated with thionyl chloride and chlorine, and the dichloropiperonyl chloride thus formed, on hydrolysis with water, yields protocatechuic aldehyde. (See also Ger. Pat. 278,788; this J., 1915, 249.)—G. F. M.

Chloropierin; Process for producing —. R. J. King, Stamford, Conn. U.S. Pat. 1,327,714, 13.1.20. Appl., 26.11.17.

A SOLUTION of calcium picrate is added to a mixture of bleaching powder and water.—G. F. M.

Tobacco; Process for improving — and removing nicotine. R. von Rothenburg, Darmstadt. Ger. Pat. 314,391, 3.5.16. Addition to Ger. Pat. 301,439.

TOBACCO is treated, during fermentation, with metabolic products of specific tobacco organisms and, at a later stage, with fatty acids and solvents for nicotine. The bacterial cultures may be grown in infusions of leaves of *Prunus odorata* (Dwarf cherry). (Cp. Eng. Pat. 121,598; this J., 1919, 963 A.)—J. H. L.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic rendering of contrast; The fundamental law for the true —. F. F. Renwick. Phil. Mag., 1920, 39, 151—151.

SINCE the characteristic curve of a plate expresses the relationship existing under certain conditions between the light gradations of an object and those of the resulting negative, and a similar relationship between the negative and the print is obtained from the characteristic curve of the printing material, a

combination of these curves will show the relationship between the object and the print. This method was referred to by the author in 1916 (this J., 1916, 1272), and in view of a recent reference by Slade and Porter (Phil. Mag., 1919, 38, 187; this J., 1919, 600A) is now explained in detail. Two instances are given, a neutral-tinted grainless wedge being taken as the object, this giving a very convenient means of impressing a known series of exposures, changing in the same way as do the values of log E along the exposure axis of a characteristic curve. In one case the correspondence between print and object is deduced from known curves for negative and positive material, and in the other the requisite curve of the positive material to obtain a desired print from a known negative material.—B. V. S.

Photographic development; Chemical induction in —. I. Induction and the Watkins factor. S. E. Sheppard and G. Meyer. Communication from The Research Laboratory of the Eastman Kodak Co. Phot. J., 1920, 60, 12—21.

THE accelerating effect of small quantities of potassium iodide on developers, first observed by Lainer in 1891, is only slight except with developers, such as glycin and quinol, which have low reduction potentials, or high induction values. In most cases the effect is merely on the induction period, the Watkins factor being increased. With a quinol-carbonate developer, however, there is also a marked alteration in the degree of development (5), particularly at low temperatures. The accelerating effect starts at very low iodide concentrations, rises to a maximum at which it remains over a certain range, and then rapidly falls to a minus quantity; with quinol, for instance, the maximum effect occurs at concentrations from $N/1000$ to $N/100$, and retardation is obtained at $N/35$ upwards. The possible nature of the iodide action is discussed, especially in connection with the suggestion, previously made, that the first stage in development is formation of a complex of silver halide and reducer. Experiments on the adsorption of safranin by silver bromide, without and with iodide treatment, are adduced in support of this view. Plates bathed in dilute potassium iodide solutions ($N/1000$ to $N/100$), washed and then developed give fog. If such a plate, after washing, is fixed and then developed in an acid developer a black, coarse-grained fog appears, different from the usual fine-grained image obtained by physical development. It is suggested that the fogging effect is due to a nucleus infection of the silver bromide grain.—B. V. S.

PATENTS.

Colour screen plates or films for photography in natural colours; Manufacture of —. M. Wieland, Berlin-Tempelhof, Germany. Eng. Pat. 137,502, 29.8.19. (Appl. 21,263/19.)

A PHENOL-FORMALDEHYDE condensation product is used as the medium in a multi-colour screen. A suitable colouring material is added to the phenol and formaldehyde and the mixture atomised before the condensation is completed. An intimate mixture of different coloured globules so obtained is dusted on to a prepared plate, the excess dusted off, and the plate then heated so that the globules soften and coalesce, and the hardening process completed by heat, and, if necessary, by pressure. A final coat of colourless condensation product is then applied. The resulting screen is unaffected by heat and ordinary solvents.—B. V. S.

Manuscript, typewritten or printed matter, drawn [photographically] —. S. J. Waters, Esher. [photographically] —. S. J. Waters, Esher. U.S. Pat. 1,327,931, 13.1.20. Appl., 9.11.18.

A STENCIL for the reproduction of manuscript etc.

is obtained by printing through the matter, or through a negative of it on to a thin sensitised film, transferring the film to a temporary support for development etc., and then transferring it to a final backing of fine texture.—B. V. S.

Photographic printing paper and process of making the same. T. P. Middleton, Assignor to Kerotype, Ltd., London. U.S. Pat. 1,329,918, 3.2.20. Appl., 21.4.19.

SEE Eng. Pat. 126,149 of 1918; this J., 1919, 441 A.

XXII.—EXPLOSIVES; MATCHES.

Melinite; Estimation of incompletely nitrated phenols in the mother liquors from — by means of bromine. Marquoyrol and P. Carré. Bull. Soc. Chim., 1920, 27, 127—138.

THE mother liquors from the manufacture of melinite contain sodium 2,4-dinitrophenol-*o*-sulphonate and sodium 2,6-dinitrophenol-*p*-sulphonate in varying proportions, together with some mononitrophenolsulphonates and a little picric acid. The action of bromine on either of the dinitrophenolsulphonates in aqueous solution depends upon the temperature and duration of contact with the bromine, and the amount of bromine added in excess. The mononitrophenolsulphonates tend to give dibromoderivatives and the picric acid present is acted on by the bromine. All of these changes tend to vitiate the results obtained by Jolibois' method for the estimation of incompletely nitrated phenol in the mother liquors from phenol. The results by this method are always too high.—W. G.

Melinite; Estimation of incompletely nitrated phenol in the mother liquors from —. Ratio between the composition of these mother liquors and the yield of melinite. Marquoyrol and P. Carré. Bull. Soc. Chim., 1920, 27, 138—140.

ONE HUNDRED c.c. of the mother liquor is concentrated until the boiling-point reaches 125°C ., then 25 c.c. of nitric acid (40°B ., sp. gr. 1.384) is added, the mixture is heated for 15—20 mins. at 115° — 120°C ., and then 10 c.c. more of nitric acid is added and the heating continued until the boiling-point again reaches 125°C .. The liquid is diluted to 100 c.c. with water, the picric acid is collected with the aid of the pump, washed five times with 4 c.c. of water, and titrated with $N/10$ sodium hydroxide, using methyl red as an indicator. It is necessary to add a correction of 0.12—0.16 gm. for the picric acid dissolved during the washing of the precipitate. The results are always a little low. To control the yield from the manufacturing process, the sulphuric acid in the mother liquors must also be determined. In a given case the mother liquors contained 472.7 grms. of sulphuric acid per litre and a quantity of incompletely nitrated phenol equal to 11.9 grms. of picric acid per litre. In the preparation of the phenolsulphonic acids, 100 grms. of phenol required 465 grms. of actual sulphuric acid. Thus in this process there was a quantity of non-nitrated phenol corresponding to $11.9 \times 465 = 472.7$ grms. of picric acid.—W. G.

Picric acid; Transformation of dinitrophenolsulphonic acids into —. Marquoyrol, P. Carré, and P. Lorette. Bull. Soc. Chim., 1920, 27, 110—143.

THE transformation of 2,6-dinitrophenol-*p*-sulphonic acid into picric acid is more rapid than that of 2,4-dinitrophenol-*o*-sulphonic acid. Thus in the manufacture of picric acid it is preferable to use a sulphonated phenol mixture containing the maximum amount of phenol-2,4-disulphonic acid.—W. G.

Picric acid; Preparation of — by the nitric acid process, using nitric acids of different concentrations. Marqueyrot, P. Carre, and P. Lorient. Bull. Soc. Chim., 1920, 27, 143—148.

It is possible to obtain good yields of picric acid in the laboratory by sulphonating 100 grms. of phenol with 350 grms. of 92.5° sulphuric acid on a water bath for three hours and nitrating the mixture with five molecular proportions of nitric acid (31.5° B., sp. gr. 1.28) under such conditions that the nitric acid added is utilised completely. Under laboratory conditions it does not seem possible to use less than these proportions of sulphuric and nitric acids.—W. G.

Nitro-compounds. Florentin and Vandenberghe. See III.

PATENTS.

Hexanitrodiphenylamine; Process of making —. J. Marshall, Swarthmore, Pa., Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,326,947, 6.1.20. Appl., 20.3.19.

2,4-DINITRODIPHENYLAMINE is nitrated with a mixture of nitric and sulphuric acids containing 30% of nitric acid to 2,4,2',4'-tetranitrodiphenylamine, which is then further nitrated to 2,4,6,2',4',6'-hexanitrodiphenylamine by treatment at an elevated temperature with a nitrating mixture containing 20% of nitric acid.—G. F. M.

Explosive. L. O. Bryan and W. R. Swint, Assignors to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,327,859, 33.1.20. Appl., 18.3.19.

THE explosive contains as the main ingredient trinitrotoluol, together with nitroglycerin and a non-explosive inorganic oxygen-carrying salt.—T St.

Nitrostarch explosive and method of manufacturing same. Method of manufacturing nitrostarch explosives. W. O. Snelling and W. R. Laws, Assignors to Trojan Powder Co., Allentown, Pa. U.S. Pats. 1,329,211 and 1,329,212, 27.1.20. Appl., 22.6.18 and 3.9.19.

NITROSTARCH is coated with a heavy mineral oil and then treated with an organic stabilising agent soluble in the oil.—W. J. W.

Nitrocarbohydrates; Chemical process for making —. G. R. Anchors, Landing, N.J. U.S. Pat. 1,329,353, 3.2.20. Appl., 6.8.18.

CARBOHYDRATES are pulfed, then nitrated, and the products are treated with salts of fatty acids.—W. J. W.

Explosive composition. W. G. Hudson, Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,329,525, 3.2.20. Appl., 18.2.18.

AN azide and colloidal nitrocellulose are used as ingredients in explosive compositions.—W. J. W.

Explosives; Method of charging containers with high —. C. A. Woodbury, Middletown township, Pa., Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,329,566, 3.2.20. Appl., 23.5.18.

CONTAINERS are filled with a mixture prepared by dissolving picric acid in melted trinitrotoluene and then dissolving trinitroxylenes in the solution, the mixture being kept at a temperature above the melting point of trinitrotoluene during the operation.—W. J. W.

[Nitrocellulose;] Process of gelatinising [liquids containing —], and product thereof. J. E. Crane, Newark, N.J., Assignor to The Arlington Company, Arlington, N.J. U.S. Pat. 1,329,583, 3.2.20. Appl., 2.11.16.

BASIC lead acetate is added to liquids containing nitrocellulose as a gelatinising agent.—W. J. W.

Explosive; Process for producing a propellant — relatively insensitive to shock. Zentralstelle für wissenschaftliche-technische Untersuchungen, G.m.b.H., Neubabelsberg. Ger. Pat. 298,539, 15.6.16.

THE nitro-compound, $C_6H_5N_2O_6$, obtained by nitration of hexamethylenetetramine, is incorporated with nitrocellulose, if necessary with the aid of volatile solvents such as ether-alcohol mixtures or acetone or of non-volatile or slightly volatile nitro-compounds which gelatinise nitrocellulose, e.g., di- or trinitro-benzene, -toluene, or -anisole.—J. H. L.

Match-head composition. J. R. Mardick, New York. U.S. Pat. 1,329,537, 3.2.20. Appl., 20.5.19.

A PERCHLORATE is employed as the oxidising agent in match compositions.—W. J. W.

Volatile solvents. Ger. Pat. 303,396. See I.

XXIII.—ANALYSIS.

Hydrogen ion concentration; Determination of —. J. W. M. Bunker. J. Biol. Chem., 1920, 44, 11—11.

A MODIFIED form of bubbling electrode used in a closed vessel is described.—J. C. D.

Plant juices; Accurate determination of the hydrogen-ion concentration of — by means of the hydrogen electrode. C. B. Clevenger. Soil Sci., 1919, 8, 217—226.

AN apparatus is described, the essential details of which are a modified Clark electrode vessel, adapted for work with plant juices, and calomel electrodes immersed in a constant temperature water bath. The apparatus is so arranged that the plant juice is saturated with hydrogen outside the electrode vessel and only comes into contact with the electrodes just prior to the readings being taken. Contact between the plant juice and the saturated solution of potassium chloride is made by means of a scratch round the cock connecting the two, as the contact potential develops quite rapidly if the connection is made by opening the cock wide. Duplicate measurements usually agree within 0.1 millivolt and constant potentials are maintained for several minutes. The plant juice is prepared by macerating the tissue, wrapping it in a muslin cloth, and expressing the juice with a press. By this means a juice free from coarse colloidal and other material is obtained.—W. G.

Arsenious anhydride; Use of — in volumetric analysis. F. de Bache. Annali Chim. Appl., 1912, 12, 136—142.

THE volumetric method of Namias (Gazz. Chim. Ital., 1892, 22, 508) consisting in adding an excess of arsenious anhydride to any oxidising agent, in the presence of ammonium acetate, and titrating the excess in the hot liquid by means of iodine in the presence of acetic acid, using starch paste as indicator, is found to give inaccurate results.

—C. A. M.

Arsenious anhydride; New volumetric reduction methods with —. F. de Bache. Annali Chim. Appl., 1919, 12, 153—171.

THE method depends upon treating the oxidising agent with an excess of arsenious acid solution in the presence of hydrochloric acid and titrating the excess with potassium bromate solution. In some cases the solution requires boiling to effect complete oxidation of the arsenious acid, and in order to avoid loss of arsenic chloride by volatilisation the proportion of hydrochloric acid must not exceed 12.8%. In the case of potassium permanganate

solution the oxidation is complete in a short time at the ordinary temperature. For the reduction of potassium chlorate in boiling solution a large excess of the arsenious acid solution is required. For the analysis of bleaching powder and hypochlorites the proportion of hydrochloric acid should be reduced to 5%. By increasing the acid concentration to 12% and effecting the reduction at the boiling point both chlorates and hypochlorites will react. Perchlorates do not interfere with the determination. Chromates are completely reduced in about an hour at the ordinary temperature or preferably at boiling point. The method may be applied to insoluble chromates, such as lead chromate.—C. A. M.

Cobalt; A sensitive reagent for —. I. Bellucci. Gazz. Chim. Ital., 1919, 49, ii., 294–298.

By means of α -nitroso- β -naphthol, 1 mgrm. of dissolved cobalt in 1–2 litres of water may be detected colorimetrically, and by means of β -nitroso- α -naphthol 1 mgrm. in about 17 litres of water. Dimethylglyoxime permits of the detection of 1 mgrm. of nickel in 4–5 litres of water. (See also Atack, this J., 1915, 641; Jones, this J., 1918, 630A; Bellucci and Chincini, this J., 1920, 46A.)—T. H. P.

Phosphoric acid; Determination of — as ammonium phosphomolybdate. A. Stutzer. Landw. Versuchs-Stat., 1919, 91, 251–264.

THE following procedure is recommended:—From 15 to 25 c.c. of the phosphate solution, containing not more than 0.25 grm. P_2O_5 , is mixed with 25 c.c. of nitric acid (sp. gr. 1.20) containing 3% by vol. of sulphuric acid, the mixture is heated to about 80° C., and 100 c.c. of molybdic acid solution (molybdic acid, 5, ammonium sulphate, 25 grms., ammonia, sp. gr. 0.96, 20 c.c., nitric acid, sp. gr. 1.2, 65% c.c., water to 100 c.c.) is added, the whole is cooled, the precipitate collected on a weighed asbestos filter, washed with cold 2% ammonium nitrate solution, then with alcohol or acetone, dried for 1 hr. at 100° C., and weighed. The factor for calculating the weight of the precipitate into P_2O_5 is 0.03513.—W. P. S.

Oxygen; Determination of — with cuprammonium solution. W. Hachnel and M. Mugdan. Z. angew. Chem., 1920, 33, 35.

SATURATED ammonium chloride solution may be used in the preparation of the cuprammonium solution used for the absorption of oxygen, and possesses the advantage over ammonium carbonate that the residual gas, after removal of ammonia by sulphuric acid, does not contain any carbon dioxide derived from the cuprammonium solution.

—W. P. S.

Sulphur; Apparatus for the determination of —. C. Hofrichter. Chem.-Zeit., 1920, 44, 110.

AN apparatus for the determination of sulphur by the evolution method consists of a reaction flask provided with a short vertical condenser; the stem of a funnel extends through the condenser to the bottom of the flask, and a side tube leads from the top of the condenser to a vessel containing the absorption solution. The material is placed in the flask, hydrochloric acid is added through the funnel, and the top of the funnel is then closed by a bent bulb tube containing a few drops of water; this valve prevents the absorption solution from being drawn back into the flask when the pressure in the latter decreases from any cause. All parts of the apparatus are of glass, and are connected by ground-in joints.—W. P. S.

Mercury; Determination of —. H. B. Gordon. Analyst, 1920, 45, 41–46.

MERCURY in very dilute acid solution may be deter-

mined by depositing it on copper gauze, then heating the copper in hydrogen and noting the loss in weight due to the volatilisation of the mercury. The volatilised mercury may be converted into mercuric iodide by the action of iodine vapour. As little as 0.1 mgrm. of mercury in 2 litres of solution may be detected and determined. The presence of nitrates does not interfere; arsenic, bismuth, antimony, and silver interfere to some extent, but it is probable that by heating the copper gauze carefully to avoid volatilising these elements with the mercury the method might be used in their presence.—W. P. S.

Colour lakes; Theory of —. [Detection and determination of cobalt, copper, and other metals.]

C. Brenner. Helv. Chim. Acta, 1920, 3, 90–103. ATTEMPTS are described to base methods for the detection and estimation of small amounts of cobalt and copper on the colorations which the metallic ions give with nitrosonaphthols. α -Nitroso- β -naphthol is unsuitable for this purpose in aqueous solution since the lake is too easily precipitated, whilst chloroform or carbon bisulphide solutions (in which cobalt nitrosite is freely soluble) are only adapted to the detection and not to the estimation of cobalt. On the other hand, the solubility of the lake in water can be sufficiently increased by introducing one or more sulphonic groups into the naphthol molecule. The colour of the lake depends on the number and position of the hydroxy- and sulphonic-groups; the yellowest shade is given by the nitroso-derivative of R acid, the deepest red with nitroso-1-naphthol-5-sulphonic acid, and the darkest shade with nitroso-1,8-dihydroxynaphthalene-3,6-disulphonic acid (chromotrope acid). Chromotrope acid can be used for the micro-titration of cobalt since the yellow ammoniacal solution of nitrosochromotrope acid yields an immediate intense-blue coloration with the cobalt ion, in the formation of which 2 mols. of the acid react with an atom of cobalt; when the cobalt ions have been completely converted into this compound, further addition of the acid causes a change in shade from blue to red. The method is fairly accurate, but it cannot be used for cobalt in the presence of nickel, although the latter alone may also be estimated by this reagent. The micro-titration of copper can be effected similarly, but a larger excess of the reagent is required to give a definite end-point. Other substances with properties similar to those of nitrosochromotrope acid are found among azo-dyes which are used with metallic mordants, and among those which, in dyeing, are subjected to after-chroming. Thus, Diamond Black F gives a deep-blue solution in aqueous alkali, which yields differently coloured lakes with ammoniacal solutions of the most diverse metals; when the metallic ion is completely united with the dye, addition of a further quantity of the latter causes a change in shade towards blue. The possible applicability of the method is illustrated by the instance of copper. The possibilities of the formation of differently coloured complex compounds have been investigated for twenty different metals and sixteen dyes; such compounds appear to be formed most frequently with magnesium, the alkaline-earth metals, and those of the iron group. Chromium is allied in this respect with the least reactive metals of the nitrogen family. The capability of the dye to form differently coloured complex compounds which are soluble in ammonia appears to depend on the presence of a hydroxy-group in the *ortho*- or *para*-position to the azo-group, and, as a result of extensive experiments with a number of dyes, it is possible to give methods for the micro-chemical estimation of copper, silver, magnesium, zinc, cadmium, calcium, strontium, barium, iron, nickel, and cobalt. (See further J. Chem. Soc., 1920, ii., 194.)—H. W.

Chlorine or bromine; Estimation of organic — by the chromic acid method. P. W. Robertson. Chem. News, 1920, 120, 51.

CERTAIN modifications are suggested to overcome minor difficulties encountered in the chromic acid method of estimating organic chlorine or bromine (this J., 1915, 819). The chromic acid may be replaced by halogen-free potassium bichromate, and a freshly prepared, cold solution of sodium peroxide is preferable as an absorbing medium. Instead of $N/10$ solutions, $N/20$ solutions of silver nitrate and ammonium thiocyanate are recommended. In the case of compounds, particularly liquids, which react explosively with the oxidising mixture the best procedure is to cover the substance with a large amount of powdered bichromate and run the previously cooled sulphuric acid very rapidly into the reaction vessel through a wide funnel by means of a suction pump connected with the absorption tube. The reaction vessel is cooled in ice until the initial violence of the oxidation has moderated.—G. F. M.

Fehling's solution; Behaviour of — in light. I. Bolin and G. Linder. Z. physik. Chem., 1919, 93, 721—736.

WHEN Fehling's solution in glass vessels is illuminated with the light from a carbon arc, cuprous oxide is produced. The reaction has no temperature coefficient and there is no evolution of gas. When the light is shut off the reaction ceases. In quartz vessels the same reaction occurs, but it has a temperature coefficient of 1.2, and there is an evolution of gas after illumination for several hours. On shutting off the light the reaction continues for some time. (See also J. Chem. Soc., 1920, ii., 144.)—J. F. S.

Indole; Quantitative estimation of — in biological media. H. F. Zoller. J. Biol. Chem., 1920, 41, 25—36.

By utilising a modification of the nitroso-reaction for indole a colorimetric method for the quantitative estimation of that substance has been elaborated. The colour produced by applying the modified nitroso-test to the distillate from the solution containing indole is compared with that given by solutions containing known quantities. (See further J. Chem. Soc., 1920, ii., 203.)—J. C. D.

Spent oxide. Weyman. See IIa.

Petroleum products. Bordas. See IIa.

Montan wax. Salvaterra. See IIa.

Nitro-compounds. Florentin and Vandenberghe. See III.

Paper-testing. Clark. See V.

Oxidation of sulphurous acid. Meyer. See VII.

Iodides. Kolthoff. See VII.

Soluble metastannic acid. Kreis. See VII.

Refractory materials. See VIII.

Aluminium dross. Hiller. See X.

Tellurium in tetradymite. Hulot. See X.

Olive oil. Cofman-Nicoresiti. See XII.

Sulphite-cellulose in tanning extracts. Knowles. See XV.

Tannery lime liquors. Thompson and Atkin. See XV.

Methyl and ethyl alcohols. Sabalitschka. See XVIII.

Minced meats etc. Ledent. See XIXa.

Bacterin. Conn and Breed. See XIXa.

Salvarsan and neosalvarsan. Utz. See XX.

Iron filings. Hindley. See XX.

Melinite. Marquayrol and Carre. See XXII.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

- Allen and Kilby. Drying-apparatus. 6819. Mar. 5.
Anhydrous Food Products Co. 7381. See XIX.
August. Muffle furnaces. 7117. Mar. 12.
Bonsignori. Evaporator. 7468. Mar. 12.
Briscoe, Lindsay, and Stevenson. Furnaces. 7135 and 7136. Mar. 12.
Byena Steel Works, Francis, and Williams. Furnaces. 6235. Mar. 1.
Crida. Control of drying processes in drying plants. 7385. Mar. 11. (Ital., 11.3.19.)
Dorr Co. Rotating-screen filtering apparatus. 5714. Feb. 25. (U.S., 5.10.18.)
Freeman. Distilling apparatus. 5971. Feb. 27.
Fuller-Lehigh Co. Pulverising mills. 5860. Feb. 26. (U.S., 27.8.17.)
Higginbottom. Centrifugal separating machines. 7396. Mar. 12.
Hoffmann. Rotary disintegrating mills. 6789. Mar. 5. (Ger., 18.5.17.)
Hoyle. Centrifugal driers. 7265. Mar. 10.
Jagger. Grinding and mixing and agitating machines etc. 6781. Mar. 5.
Lowden. Drying materials. 7335. Mar. 11.
Lunt. Method of drying colloids. 5692. Feb. 25. (U.S., 7.1.16.)
Rennison. Gas-heated furnaces. 5520. Feb. 24.
Riba. Filter-presses. 7453. Mar. 12.
Richards. Manufacture of briquettes. 5965. Feb. 27.
Schjelderup. Drying material containing liquid. 5159. Feb. 23. (Norway, 21.2.19.)
Soc. l'Air Liquide. Protecting walls of enclosures in which reactions take place under high temperatures and pressures. 7182. Mar. 9. (Fr., 18.12.18.)
Soc. l'Air Liquide. Apparatus for carrying out exothermic chemical reactions under high temperatures and pressures. 7189. Mar. 9. (Fr., 21.12.18.)
Techno-Chemical Laboratories, Ltd., and Testrup. Evaporation, distillation, etc. 5558. Feb. 24.
Weyel and Zimmermann. Drying apparatus. 7215. Mar. 10.

COMPLETE SPECIFICATIONS ACCEPTED.

- 17,387 (1918). A.-G. Kummier und Matter. Evaporation of water from aqueous liquids. (123,716.) Mar. 10.
6096 (1919). Webb, and Ransome-ver Mehr Machinery Co. Mixing machines. (139,018.) Mar. 3.
16,210 (1919). Peck. Filter leaves. (139,390.) Mar. 10.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

- Aoki and Matsunaga. Manufacture of fuel briquettes. 6332. Mar. 8.
Chem. Fabr. Rhenania, and Projahn. 6921. See VII.
Dalley (Greenway). Distilling or cracking hydrocarbon oils. 5861. Feb. 26.
Dixon and Garton. Briquetting coal etc. 7137. Mar. 9.
Edser and Reynard. Lubricants. 6102. Feb. 28.
Elektrizitätswerk Lonza. Manufacture of a solid fuel. 7170. Mar. 9. (Switz., 16.9.19.)

- Evans. Lubricating oils. 6103. Feb. 28.
 Evans. Suction-gas producer plants. 6165. Mar. 1.
 Evans. Refining crude oil. 7180 and 7181. Mar. 9.
 Fyleman. Separating oils etc. from sand or rock. 6509. Mar. 3.
 Garrow and Nielsen. Continuous production of solid and gaseous fuel with by-product recovery. 5103. Feb. 23.
 Glover, West, and West's Gas Improvement Co. Vertical retort settings for destructive distillation of coal etc. 5563. Feb. 24.
 Goodliffe. Motor spirit. 5627. Feb. 25.
 Hurez. Coke ovens. 7432. Mar. 12. (Fr., 29,10,19.)
 Hllingworth, and South Wales and Monmouthshire School of Mines. Coking coal. 6078. Feb. 28.
 Kummel and Ternen. Manufacture of ammonia and gas by distillation of fuel. 7191. Mar. 9. (Ger., 10,3,19.)
 Lewis. Means of distilling and gasifying solid carbonaceous matter. 5926. Feb. 27.
 Perry. Apparatus for distilling carbonaceous material. 5666. Feb. 25.
 Riley. Coke ovens. 7075. Mar. 9.
 Ross and Young. Arc-lamp electrodes for producing violet and ultra-violet light. 5340. Feb. 23.
 Southcombe and Wells. Lubricating oils. 5913. Feb. 27.
 Stiansen. 7059. *See XII.*
 White. Gas-producers etc. 6451. Mar. 3.

COMPLETE SPECIFICATIONS ACCEPTED.

- 9114 (1918). Dunstan. Refining liquid hydrocarbons. (139,233.) Mar. 10.
 3503 (1919). Fornas. Gas-producers. (139,578.) Mar. 17.
 5286 (1919). Rogerson. Coal-washing plant. (139,612.) Mar. 17.
 9970 (1919). Heuser. Retort ovens for distilling fuels and bituminous ores. (125,980.) Mar. 17.
 16,523 (1919). Lane and Williams. Artificial fuel. (139,091.) Mar. 3.
 19,477 (1919). Irazusta. Agglutinant for conglomerating coal. (139,106.) Mar. 3.
 20,800 (1919). Seccombe and Sandiford. Artificial fuel. (139,115.) Mar. 3.

III.—TAR AND TAR PRODUCTS.

COMPLETE SPECIFICATIONS ACCEPTED.

- 9256 (1918). Morris and Co., and Morris. Apparatus for sulphonating, nitrating, and chlorinating organic substances. (139,231.) Mar. 10.
 2073 (1919). Edwards. Fractional distillation plant for coal tar hydrocarbons etc. (139,263.) Mar. 10.
 28,279 (1919). South Metropolitan Gas Co., and Kirby. Purification of carbazole. (139,111.) Mar. 10.

IV.—COLOURING MATTERS AND DYES.

APPLICATION.

- Kane. Production of dyes. 7028. Mar. 8.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

- Ballonhüllen Ges. Balloon envelope materials. 6519, 6520, 6613. Mar. 3 and 4. (Ger., 24,12,14, 3,3,16, 22,9,17.)
 Goldreich. Manufacture of paper. 5682. Feb. 25.
 Le Tall (Soc. Franç. des Crins Artificiels). Production of moulded articles of pure cellulose. 6382. Mar. 2.
 Roberts. Manufacture of articles from cellulose or its compounds. 5970. Feb. 27.

- Verein. Glanzstoff-Fabriken A.-G. Manufacture of viscose silk. 5556. Feb. 24. (Ger., 25,7,18.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 5952 and 15,163 (1918). Zdanowich. Manufacture of cellulose acetates. (139,232.) Mar. 10.
 15,551 (1919). Soc. Anon. des Etabl. Hutchinson. Adhesive composition especially for waterproofing fabrics and rendering them impermeable to gases. (129,630.) Mar. 3.
 19,297 (1919). Clayton. Apparatus used in the manufacture of artificial silk. (139,104.) Mar. 3.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

- Kereszty and Wolff. Production of bleaching and disinfecting agents. 6541. Mar. 3. (Hungary, 12,7,19.)
 Sutcliffe. Treatment of fabrics etc. to render them less inflammable. 6933. Mar. 8.

COMPLETE SPECIFICATIONS ACCEPTED.

- 7239 (1919). Gibson. Finishing composition for yarns and fabrics. (139,634.) Mar. 17.
 13,083 (1919). Calico Printers' Assoc., Schofield, and Farnworth. Producing certain colour effects on textile and other fabrics and materials. (139,373.) Mar. 10.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

- Basset. 7260. *See X.*
 Chance and Hunt, Gidden, and Ragg. Manufacture of oxide and carbonate of zinc. 5973. Feb. 27.
 Chem. Construction Co. 6539. *See XVI.*
 Chem. Fabr. Rhenania, and Projahn. Extraction of sulphur from gases containing sulphuretted hydrogen or sulphurous acid. 6921. Mar. 6.
 Comm. Apparatus for producing cyanogen compounds. 6559. Mar. 3.
 Dutt and Dutt. Production of potash salts, ammonia, and alumina. 5651. Feb. 25.
 Ellis (Foundation Oven Corporation). Purification of ammonium salts. 6206. Mar. 1.
 Fabr. de Prod. Chim. de Thann et de Mulhouse. Manufacture of potassium sulphate and hydrochloric acid. 6009. Feb. 27. (Fr., 4,12,19.)
 Kummel and Ternen. 7191. *See II.*
 Lamm. Manufacture of potassium carbonate and sodium carbonate. 6366. Mar. 2.

COMPLETE SPECIFICATIONS ACCEPTED.

- 8535 and 18,832 (1918). Field, and Metals Extraction Corporation. Purification of zinc solutions. (138,946 and 138,954.) Mar. 3.
 8831 and 8832 (1918). Sulman, Field, and Metals Extraction Corporation. Purification of zinc solutions. (138,947 and 138,948.) Mar. 3.
 10,708 (1918). Field, and Metals Extraction Corporation. Purification of metallic solutions. (138,950.) Mar. 3.
 4827 (1919). Orton and Robinson. Manufacture of alumina. (139,005.) Mar. 3.
 11,913 (1919). Rossi. Production of potassium compounds and nitrogen-containing fertilisers. (130,963.) Mar. 17.
 28,471 (1919). Comp. des Prod. Chim. d'Alais et de la Camargue. Purification of solutions of zinc sulphate. (139,443.) Mar. 10.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

- Grace. Drying china-clay etc. 5698. Feb. 25.
 Hill. Fullers' earth etc. 7042. Mar. 8.
 Norton Co. Abrasive stone. 5760. Feb. 25. (U.S., 26,2,19.)

Wallace. Manufacture of clayey materials. 7389. Mar. 11.

COMPLETE SPECIFICATIONS ACCEPTED.

2470 (1919.) Ionides. *See* IX.
6303 (1919.) British Thomson-Houston Co. (General Electric Co.). Porcelain. (139,315.) Mar. 10.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Boyd. Manufacture of impervious building-blocks. 6126. Mar. 1.
Collins. Kiln for burning bricks etc. 6153. Mar. 1.
Fraser. Manufacture of bituminous emulsions. 5705. Feb. 25. (U.S., 24.10.17.)
Fraser. Cementitious bituminous emulsions. 5706. Feb. 25. (U.S., 25.10.17.)
Fraser and Peters. Manufacture of bricks, building blocks, etc. 6472. Mar. 3.
McLay. Heat non-conducting compositions. 6057. Feb. 28.
Sanders. Production of artificial coloured stone. 7424. Mar. 12.
Tibbenham. Staining or fuming wood. 6375. Mar. 2.
White. Manufacture of paving and building blocks. 7439. Mar. 12.

COMPLETE SPECIFICATIONS ACCEPTED.

8305 (1918.) Hartner. Manufacture of a mortar-forming material from anhydrite. (117,605.) Mar. 10.
2470 (1919.) Ionides. Heat treatment of bricks, pottery, etc. (139,267.) Mar. 10.
6522 (1919.) Forrester (Internat. Isolations Komp.). Manufacture of thermal insulating bodies. (139,318.) Mar. 10.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

August. 7417. *See* I.
Ball and Hurst. Aluminium alloys. 6733. Mar. 5.
Ballantine. Refining metals and alloys. 5811. Feb. 26.
Basset. Production of metals or metalloids. 7260. Mar. 10.
Beck. Producing spelter from zinc dust. 6247. Mar. 2.
Chambers. Solders for aluminium. 7155. Mar. 9.
Coles. Process for sherardising. 6851. Mar. 6.
Coles. Removing scale or oxide and preparing surface for receiving coating of another metal. 7588. Mar. 13.
Diehl. Smelting zinc-bearing products. 7446. Mar. 12.
Evans. Cupolas. 6864. Mar. 6.
Field and Petersson. Treatment of sulphide ores and minerals. 6545. Mar. 3.
Goskar and Hitch. Case-hardening compositions. 6754. Mar. 5.
Hall, and Rolls-Royce, Ltd. Preparation of metallic alloys. 5722. Feb. 25.
Hall, and Rolls-Royce, Ltd. Aluminium alloys. 5724 and 5725. Feb. 25.
Homer and Homer. Surface-hardening steel parts. 6964. Mar. 8.
Irons. Manufacture of steel. 7139. Mar. 9.
Law. Solder for aluminium. 6609. Mar. 4.
Lemmon, and Minerals Separation, Ltd. Recovery of metals from their ores. 6919. Mar. 6.
Meakin. Removing oxide from sheet metal. 7301. Mar. 11.

Metallind. Schiele u. Bruchsalser. Increasing resistance of aluminium to acid and alkaline liquids. 6912. Mar. 6. (Ger., 8.3.19.)

Minerals Separation, Ltd. Ore concentration. 7190. Mar. 9. (Fr., 29.11.19.)

Naylor. Manufacture of magnet steel. 6401. Mar. 2.

Raworth (Dean). Soldering, tinning, or coating aluminium or its alloys. 5532. Feb. 24.

Rondelli, Sestini, and Sestron Colour Oxidizing Co. Colouring and oxidising metal surfaces. 6390 and 6391. Mar. 2.

Rondelli, Sestini, and Sestron Colour Oxidizing Co. Preparing ferrous surfaces for enamelling, varnishing, etc. 6392. Mar. 2.

Watkin. Chlorine-oven process of melting etc. gold. 7327. Mar. 11.

COMPLETE SPECIFICATIONS ACCEPTED.

3369 (1918.) Giesecke. Solidifying conglomerates of fine ores, iron filings, purple ore, etc. (139,229.) Mar. 10.

8535, 8831, 8832, 10,708, 18,832 (1918.) Metals Extraction Corporation and others. *See* VII.

18,030 (1918.) Marks (Luckenbach Processes). Ore concentration. (139,535.) Mar. 17.

212 (1919.) British Thomson-Houston Co. (General Electric Co.). Manufacture of metals and alloys by the aluminio-thermic method. (139,247.) Mar. 10.

1271 (1919.) Bolton. Electro-plating. (138,967.) Mar. 3.

2737 (1919.) Brighten and Peakman. Furnaces for heat treatment of metals. (138,981.) Mar. 3.
10,169 (1919.) Hisamoto and others. Process of drawing refractory metal. (139,065.) Mar. 3.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Burgess Battery Co. Dry batteries. 6222. Mar. 1. (U.S., 13.3.19.)

Holland and Skinner. Storage batteries. 7023 and 7024. Mar. 8. (U.S., 5.9.19 and 17.5.19.)

Leggett and Niece. Apparatus for subjecting molecular matter etc. to electrostatic stress of high-tension electric discharge. 6101. Feb. 28.

Le Sueur. Electrolytic cells. 6267. Mar. 2.

Oldham, and Oldham and Son. Galvanic batteries. 6654. Mar. 4.

Saunders, and Saunders Electrical Co. Electric accumulators. 5807. Feb. 26.

Sutton. Electro-thermal absorber generator for separation processes. 6780. Mar. 5.

COMPLETE SPECIFICATIONS ACCEPTED.

1271 (1919.) Bolton. *See* X.

2203 (1919.) Hossack. Electric dry batteries. (139,265.) Mar. 10.

4386 (1919.) London Battery Co., and Clarke. Electric storage batteries. 139,001. Mar. 3.

4472 (1919.) Hepburn. Mono-polar electrode electrolyzers. (139,296.) Mar. 10.

8550 (1919.) Ivey and Salisbury. Electric storage batteries. (139,652.) Mar. 17.

22,068 (1919.) Norske Aktieselskab for Elektrokemisk Industri. Electrodes for electric furnaces. (137,811.) Mar. 17.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Boehringer Sohn. Manufacture of soaps etc. 6239. Mar. 1. (Ger., 21.11.18.)

Bollman. Removal of fatty acids, resins etc. from fats and oils. 6226. Mar. 1.

Bredlik and Whiton. Extraction of fatty matter from garbage etc. 6297. Mar. 1. (U.S., 15.5.19.)

- Hill. 7042. *See* VIII.
 Snape. Soap. 5584. Feb. 24.
 Stiansen. Process of refining oils. 7059. Mar. 9.
 (Norway, 21.3.19.)
 Williams. Soap. 5488. Feb. 24.

COMPLETE SPECIFICATION ACCEPTED.

- 19,567 (1918). Martin. Hydrogenating oils.
 (139,239.) Mar. 10.

XIII.—PAINTS; PIGMENTS; VARNISHES;
 RESINS.

APPLICATIONS.

- Chance and Hunt, and others. 5973. *See* VII.
 Coles. Manufacture of white lead. 7548.
 Mar. 13.
 Ivinson and Roberts. Anti-corrosive paint etc.
 7564. Mar. 13.
 Pooley. 7488 and 7489. *See* XVII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

- Bruni. Vulcanisation of rubber. 6670 and 6774.
 Mar. 4 and 5. (Ital., 15 and 19.3.19.)
 Crozier. Black rubber compound. 7294. Mar. 11.

COMPLETE SPECIFICATION ACCEPTED.

- 227 (1919). Raap. Regenerating vulcanised
 rubber. (122,188.) Mar. 3.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

- Humphries. Treatment of quebracho tanning extract. 5787. Feb. 26.
 Melamid. Manufacture of artificial tanning substances. 5439, 5690, 5691. Feb. 23 and 25.
 (Ger., 19, 15, and 21.7.19.)
 Tannage Rationnel Meurant Soc. Anon. Tanning leather. 7215. Mar. 10. (Belg., 12.3.19.)

COMPLETE SPECIFICATION ACCEPTED.

- 21,280 (1919). Marks (Nitritfabrik A.-G.). Preparation of gallic acid. (139,419.) Mar. 10.

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

- Chemical Construction Co. Manufacture of acid phosphate. 6539. Mar. 3. (U.S., 4.3.19.)

COMPLETE SPECIFICATION ACCEPTED.

- 11,913 (1919). Rossi. *See* VII.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

- Colour Photography, Ltd., and Thomson. Adhesives. 6817. Mar. 5.
 Haddan. 5707. *See* XIX.
 Pooley. Extraction of gum from grass trees. 7488 and 7489. Mar. 12.

COMPLETE SPECIFICATION ACCEPTED.

- 13,418 (1917). Daniel. Process of obtaining inulin from plants. (109,813.) Mar. 10.

XVIII.—FERMENTATION INDUSTRIES.

COMPLETE SPECIFICATION ACCEPTED.

- 18,041 (1919). Wade (Schneible). Manufacture of ethyl alcohol and de-alcoholised beverages from fermented liquids. (139,099.) Mar. 3.

XIX.—FOODS; WATER PURIFICATION;
 SANITATION.

APPLICATIONS.

- Anhydrous Food Products Co. Drying fruits, meats, etc. 7381. Mar. 11. (U.S., 11.5.15.)
 Baker, and Fluorescent Materials, Ltd. Preparation of radium emanation water. 6926. Mar. 6.
 Bredlik and Whiton. 6207. *See* XII.
 Haddan (Naaml. Vemoots. Algem. Norit Maatsch.). Continuously treating large quantities of liquids with purifying agents. 5707. Feb. 25.
 Jones, Watson, and Woodlands, Ltd. Manufacture of flour and bread. 6624 and 7576. Mar. 4 and 13.
 Kereszty and Wolf. 6541. *See* VI.
 Langham and Pennington. 6184. *See* XX.
 Manganzone Soc. Anon. Sterilising and clarifying water. 7141. Mar. 9. (Fr., 12.12.13.)
 Werner. Treatment of flour. 6192. Mar. 1.

COMPLETE SPECIFICATIONS ACCEPTED.

- 1816 (1918). Hildebrant. Manufacture of meat-extract-like preparations. (139,525.) Mar. 17.
 18,140 (1919). Pape. Preserving materials subject to putrefaction or decay. (139,100.) Mar. 3.
 19,425 (1919). Mulertz. Sterilising or pasteurising liquids. (132,237.) Mar. 10.

XX.—ORGANIC PRODUCTS; MEDICINAL
 SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

- Datta. Treatment of tea plant and manufacture of caffeine. 6209. Mar. 1.
 Langham and Pennington. Antiseptic and prophylactic. 6184. Mar. 1.
 Napp (Hoffmann-La Roche & Co.). Manufacture of colloiddally soluble metal pyrophosphate casein compounds. 6081. Feb. 28.
 Soc. Chim. des Usines du Rhône. Manufacture of β -alkyl-amino-ethylaminobenzoic alkyl esters. 6329. Mar. 2. (Fr., 15.11.19.)
 Soc. Chim. des Usines du Rhône. Manufacture of saccharine. 7018. Mar. 8. (Fr., 24.1.20.)
 Tcherniac. Production of saturated halogenated ethers and their decomposition products. 5839 and 5840. Feb. 26.

COMPLETE SPECIFICATIONS ACCEPTED.

- 9256 (1918). Morris, and Morris and Co. *See* III.
 17,172 and 17,173 (1918). Rockefeller Institute for Medical Research. Aromatic arsenical compounds. (122,819 and 128,181.) Mar. 3.
 4163 (1919). Appelbaum. Manufacture of aldehydes. (138,999.) Mar. 3.
 8330 (1919). Christiansen. Manufacture of methyl alcohol. (125,946.) Mar. 10.
 9006 (1919). Chem. Fabr. vorm. Sandoz. Manufacture of a highly active preparation of ergot. (125,396.) Mar. 17.
 20,863 (1919). Chem. Fabr. Flora. Substituting halogens for the amino groups in aliphatic amines. (132,245.) Mar. 17.

XXI.—PHOTOGRAPHIC MATERIALS AND
 PROCESSES.

APPLICATIONS.

- Clark. Photography. 6194. Mar. 1. (U.S., 25.2.19.)
 Kent. Photographic transfer processes. 6377. Mar. 2.
 Mimosa A.-G. Toning photographic negatives, films, and positives. 5948. Feb. 27. (Ger., 3.7.18.)
 Trist. Colour photography. 6898. Mar. 6.
 Welsh and Wise. Powders for artificial lights for photography. 7404. Mar. 12.
 Wolfe. Photography. 6168. Mar. 1.

I.—GENERAL; PLANT; MACHINERY.**PATENTS.**

Grinding, crushing, or pulverising machine. O. Wauthier, Brussels. Eng. Pat. 13,878, 8.6.14.

A CYLINDRICAL casing with horizontal axis is split longitudinally for access to the interior. Concentric with the casing is a shaft carrying, say, four discs in which are cut radial slots to form the bearings for the axles of rolls, which are thus supported in the spaces between the discs. Grinding takes place between the cylindrical surfaces of the rolls and outer casing, the rolls being rotated by the friction and pressed outwards by centrifugal force.

—B. M. V.

Grinding and pulverising materials. W. C. Kirby, Harbury Station, and E. L. Lakin, Leamington Spa. Eng. Pat. 138,411, 21.1.19. (Appl. 2213/19.)

GRINDING bodies for use in tube and similar mills are made of hard material, such as cast steel, in the form of discs with their thickness less than their diameter. They are perforated with one or more holes in the axial direction and may be cylindrical or with the angles between the ends and the periphery rounded off; their end faces may also be flat or corrugated.—B. M. V.

Liquefying gases or gaseous mixtures; Process and apparatus for cooling or —. M. Zack, Zürich, Switzerland. Eng. Pat. 129,292, 4.7.19. (Appl. 16,774/19.) Int. Conv., 30.5.18.

THE apparatus consists of a two-cylinder compressor with the cranks at 180° and the cylinders connected by two sets of pipe-coils controlled by valves, the second set of intercommunication pipes being concentric with and outside the first set; the cylinders are also surrounded by a common jacket or casing. The cycle of operation is as follows:—Gas that has been purified, pre-compressed, and pre-cooled, is admitted to a heat interchanger, where it gives up more heat to the final remainder of non-liquefied gas issuing from the apparatus. From the heat interchanger the gas is admitted to one end of one cylinder (say the top of the left-hand one) and on the down-stroke of the piston expands while doing external work; on reaching the dead centre the gas is allowed to pass through the inner set of pipe coils to the underside of the right-hand piston (which has on its up-stroke been producing a vacuum), again expanding but without doing external work. Upon the commencement of the next stroke the intercommunication is shut off and the gas in both cylinders compressed while the cylinders are kept cool by means of their jackets, which are preferably filled with the non-liquefied remainder of the gas; at a certain point in the stroke, determined by the setting of the valves, the outer communicating pipe-coils are opened and the compressed gas passes from the bottom of the right-hand cylinder to the top of the left-hand cylinder and *vice versa*, again without doing external work, this movement continuing until the other dead centre is reached. Liquefied gas is removed by suitable means from both sets of cross connecting pipes and the non-liquefied gas finally allowed to exhaust into the jacket surrounding the cylinders and thence to the first-mentioned heat interchanger. The gas is expanded with external work at a comparatively high, and without external work at a comparatively low temperature.

—B. M. V.

Mixing apparatus. R. B. Grey, London. Eng. Pat. 138,286, 5.9.19. (Appl. 21,830/19.)

A CIRCULAR tank is provided at the bottom with a stirrer which produces a vortex, and, at various

distances above the stirrer with baffles which can be adjusted individually as to height by means of rods brought through the cover of the tank.

—B. M. V.

Filter apparatus. A. R. Peck, Los Angeles, Cal., U.S.A. Eng. Pat. 138,657, 22.10.19. (Appl. 17,256/18.)

FILTER-LEAVES project from fixed cover-plates which carry valves and pipes for mud to be filtered, wash water, and, if desired, compressed air. Removable chambers, mounted on wheels rolling on the channel iron framework of the apparatus, can be bolted to the fixed cover-plates. During the filtering and washing period the chambers are bolted to the cover-plates and the mud or wash water admitted through valves in the cover-plates to the interior of the chambers, whence the liquor passes through the filter-leaves and out through other valves in the cover-plates. When the cakes are built up and washed, the enclosing chambers are moved out of the way without disturbing the cakes and the cakes finally caused to drop off by means of compressed air.—B. M. V.

Filter-press plate. Filter-press device. G. F. Miller, New York. U.S. Pats. (A) 1,330,331 and (B) 1,330,332, 10.2.20. Appl., 22.1 and 14.1.19.

(A) A SUPPORTING frame contains field plates, and a longitudinal recess is made in the upper portion of each field plate to form a conduit in conjunction with the frame. (B) In order to be able to vary the thickness of the filter-cake, alternate field plates are divided, the two halves being made to move outwards from each other within the frames by means of cams or other devices operated by rods rotatable from the outside of the press.—B. M. V.

Filter. O. Baumgartner, Vienna, Ger. Pat. 315,317, 14.3.16. Int. Conv., 6.11.15.

A VESSEL is fitted with a sieve, against the underside of which cork dust or similar light, floatable, granular material is pressed and kept in position by the passage of the liquid through the filter from below. The filtering material may be cleaned by stirring it by hand or mechanically while passing a current of water under pressure through the sieve, or the liquid in the apparatus may simply be withdrawn until the level is below the sieve when the filter mass breaks up, thus allowing the impurities to sink to the bottom.—A. R. P.

Vacua; Methods of and apparatus for producing —. Western Electric Co., Ltd., London. From Western Electric Co., Inc., New York. Eng. Pat. 138,745, 20.3.19. (Appl. 6951/19.)

IN apparatus using the vapour of boiling mercury (or other liquid) to withdraw the last traces of air (or other gas) from a vessel, the walls of the conduit conveying the stream of mercury vapour are cooled at a point in front of the place where the air being exhausted is admitted to the stream, with the object of removing, by condensation, those molecules of mercury vapour which are not moving substantially in the direction of the stream, and which if not removed would heat back molecules of issuing air and prevent proper exhaustion.—B. M. V.

Raising and forcing water and other liquids; Means for —. R. A. Swainson, Newcastle-upon-Tyne. Eng. Pat. 138,758, 22.5.19. (Appl. 8328/19.)

THE liquid is raised and expelled from a pair of pumping chambers by means of the alternate exhaustion and compression of air above the liquid, float valves being provided so that no liquid can be drawn into the air pipes and no unnecessary work is done in producing too much vacuum or too much compression on varying suction and delivery heads.

—B. M. V.

Furnaces for steam boilers, kilns, and the like. J. Nash, Westcliff-on-Sea. Eng. Pat. 138,762, 9.4.19. (Appl. 9017/19.)

THE fire-box of the furnace is provided with two internal arches extending from the back towards, but not as far as, the front, the upper arch being shorter than the lower. A fire is built on the front of the lower arch as well as on the usual fire-bars or bottom hearth; the fires may be stoked alternately so that there is always incandescent fuel exposed, which promotes consumption of smoke.—B. M. V.

Tunnel-kiln. G. H. Benjamin, New York. U.S. Pat. 1,329,745, 3.2.20. Appl., 23.5.19.

A TUNNEL oven comprises a main chamber, a number of independent heating chambers arranged longitudinally in line on each side of the main chamber, and air flues situated behind and in front of each chamber. A screen is arranged in front of each chamber, with means for moving the screens to and from the chamber in front of which they are individually placed.—B. N.

Tunnel kiln combustion chamber. Tunnel kiln. P. d'H. Dressler, Zanesville, Ohio, Assignor to American Dressler Tunnel Kilns, Inc., New York. U.S. Pats. (A) 1,330,432 and (B) 1,330,433, 10.2.20. Appl., 28.5.19.

(A) A COMBUSTION chamber has a hollow wall to provide an air-circulating passage open at the ends; a window opens from the passage to the outer wall surface. (B) A tunnel kiln is provided with a preliminary heating zone between the entrance and the high-temperature zone. A combustion chamber in the high temperature zone is separated from the heating chamber by conducting walls, and is provided with an "extension space" extending through the preliminary heating chamber. A cooling fluid is supplied to the "extension space." —W. F. F.

Ring-furnace. B. E. Broadwell, Badin, N.C., Assignor to Aluminium Co. of America, Pittsburgh, Pa. U.S. Pat. 1,330,164, 10.2.20. Appl., 22.1.18. Renewed 5.9.19.

THE bottom, side walls, and end walls of the heating chamber are hollow. Air or gas enters a hollow end wall at the bottom and passes out at the top to a transverse conduit, and thence to the upper part of the hollow side wall of the furnace. The air or gas is then delivered into the upper part of the furnace chamber, which is provided with longitudinal partitions forming baffles, and the gas passes out at the bottom into the hollow bottom of the chamber. The gas passes from the flues in the hollow bottom to the second end wall and upwards through the hollow wall to the outlet at the top. —W. F. F.

Ring furnace. V. C. Doerschuk, Massena, N.Y., Assignor to Aluminium Co. of America, Pittsburgh, Pa. U.S. Pat. 1,330,175, 10.2.20. Appl., 1.5.19.

THE heating chamber is divided into compartments by longitudinal vertical partitions provided with internal flues. The end walls of the chamber are also hollow and are provided with vertical flues and with burner ports above the flues. The burner ports open directly into the flues in the vertical partitions and also into similar flues in the side walls, and means are provided for delivering fuel gas to any desired series of burner ports. The ports open into their respective flues in the direction of the flow of air.—W. F. F.

Rotary furnaces; Sectional lining for —. W. S. Rockwell, Assignor to W. S. Rockwell Co., New York. U.S. Pat. 1,330,219, 10.2.20. Appl., 8.9.19.

A ROTARY furnace having a refractory lining is pro-

vided with a cylindrical inner metallic lining in sections, each section being provided with a portion of one or more helical ribs so that when the sections are all in place the ribs are continuous and form a spiral channel with entirely metallic walls.

—B. M. V.

Furnace. S. S. Amdursky, Rochester, N.Y. U.S. Pat. 1,330,227, 10.2.20. Appl., 14.5.18.

A TWIN furnace contains two upright crucible chambers heated by separate burners. A connecting conduit tangential to both chambers allows the heated products to pass from either chamber to the other.—T. St.

Retort; Rotary —. C. Francke, Berlin. Ger. Pat. 314,546, 28.3.18.

A CYLINDRICAL retort rests horizontally on two rollers at one end. To the other end is fixed a grooved collar which can turn inside a ring with an internal rib which engages with the groove of the collar. The ring is supported on rollers by which it is caused to revolve.—H. J. H.

Shaft furnace for burning lime, dolomite, etc., with built-in gas producer. E. Skuballa, Berlin. Ger. Pat. 314,586, 12.7.17.

THE gas producer is annular in shape and surrounds the shaft of the kiln, being separated from the latter only by the gas and air passages. The top of the producer is at the level of and forms the charging floor of the kiln. The bottom of the producer is a ring-shaped mechanically driven revolving grate, which delivers ash into hoppers placed below. Regularity in the heating of the charge is claimed.—H. J. H.

Rabbles or other agitators or stirrers. W. H. Sayre, Glen Ridge, N.J., U.S.A. Eng. Pat. 138,802, 11.6.19. (Appl. 14,787/19.)

IRREGULAR lumps of hard material, such as alundum, are embedded in the arms of rabbles or blades of pug-mills etc. in such a manner that the projecting lumps take most of the wear.—B. M. V.

Drying material; Method of and apparatus for —. O. D. Rice, Winthrop, Mass. U.S. Pat. 1,328,897, 27.1.20. Appl., 15.1.19.

A VACUUM drying chamber is provided inside with a fan which maintains a heating and drying medium in continuous circulation over the material to be dried within the chamber. Means are provided for heating the drying medium and for withdrawing moist vapours from the chamber by suction.—J. H. L.

Drying solids; Process of —. R. W. G. Stutzke, Assignor to The G. A. Buhl Co., Chicago, Ill. U.S. Pat. 1,329,813, 3.2.20. Appl., 28.3.17.

SOLIDS are continuously introduced into a closed system through which there is set up a circulation of vapours which are superheated during their circulation, and the system is provided with a vent to limit the rise of pressure.—B. N.

Dryer. B. M. Kuhn, Bloomington, Ill. U.S. Pat. 1,330,195, 10.2.20. Appl., 25.2.18.

MATERIAL to be dried is supported on a horizontal reticulated partition dividing a drying chamber into two parts. Hot air is supplied to the lower compartment and passes upwards through a reticulated flue which rests on and is movable over the surface of the reticulated partition.—W. F. F.

Desiccating apparatus. A. D. Robinson, Logan, W. Va. U.S. Pats. (A) 1,330,746 and (B) 1,330,747, 10.2.20. Appl., 17.10 and 11.11.18.

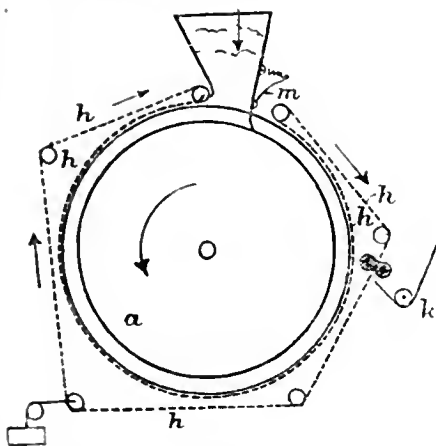
(A) The material to be dried, e.g., eggs or the like, is supplied to a belt passing over a drum, by means

of a number of discharge heads arranged transversely to the belt. A pivoted rocker arm projects inwards from the periphery over the end face of the drum, and a pin carried by the drum comes into contact with the arm at every revolution. The arm is adapted to open the discharge openings of all the supply heads simultaneously, and the material is spread on the belt by a revolving brush. (n) The drying belt passes horizontally through a hot air chamber just below a horizontal partition. This partition is perforated with elongated openings arranged transversely, their length being equal to the distance between them. The transverse rows are arranged so that the perforations in adjacent rows are out of line. Air is admitted into the chamber and deflecting strips are provided to equalise the flow of air through the perforations.

—W. F. F.

Dryer; Cylindrical —. W. Greding, Cronberg. Ger. Pat. 313,148, 23.6.16.

A NUMBER of grooves in planes normal to the axis are provided on the periphery of a cylindrical dryer, *a*, constructed of sheet-metal. The moist material is conveyed to the grooves, and the cylinder is sur-



rounded by an endless perforated band, *h*, which rests against the projections between the grooves and moves with the same velocity as the drum. The dried material is removed by a scraper, *m*, and is carried by the band, *h*, to the discharge trough, *k*.

—L. A. C.

Drying apparatus; Tubular —. K. Wagner, Dresden. Ger. Pat. 315,862, 13.3.18.

THE apparatus comprises a network of tubes through which air circulates, and a series of heaters placed in the meshes of the network. The air current flows in an opposite direction to the material and serves to cool the latter when dry.

—W. J. W.

[Electrically] precipitating suspended material from furnace-gases; Method of —. Precipitating suspended material from gases. Method of collecting suspended material from furnace-gases. E. R. Wolcott, Assignor to International Precipitation Co., Los Angeles, Cal. U.S. Pats. (a) 1,329,737, (n) 1,329,817, and (c) 1,329,818, 3.2.20. Appl., (a) 19.3.19, (n) 5.7.17, and (c) 29.4.18.

(a) Water is supplied to the gases to humidify them to a condition of partial saturation, and partially to cool them; the gases are then further cooled and the suspended particles precipitated by electrical action. The temperature of the precipitated material and the relative humidity of the gases are

controlled in such a manner as to provide sufficient moisture in the precipitated material to prevent accumulation of electric charge thereon, but not sufficient to make the deposit corrosive or adherent to the surfaces on which it is precipitated. (n) The dielectric strength of the precipitating electrical field is maintained, in cases where the precipitated material is non-conducting and tends to accumulate a charge by the action of the electrical field, by applying to the deposit an agent capable of increasing the conductivity thereof sufficiently to discharge the same without rendering the deposit fluid. (c) Suspended material is separated from hot furnace gases by injecting water into the gases to humidify and cool them, the gases being then passed through an electrical field. The surfaces of the collecting electrodes are maintained sufficiently cool to condense water on the precipitated material, the temperature of the gases and the collecting electrodes being so controlled as to cause sufficient condensation of water to form a fluid deposit with the material on the collecting electrodes.—B. N.

Electrical treatment of gases; Apparatus for —. W. A. Schmidt, Los Angeles, Cal., and L. Bradley, East Orange, N.Y., Assignors to Research Corporation, New York. U.S. Pat. 1,329,859, 3.2.20. Appl., 24.6.16.

THE apparatus is provided with inlet and outlet manifold chambers, with a number of pipes extending between them, through which the gases are passed. Discharge electrodes consisting of wires are suspended in the pipes and kept taut by weights, and are insulated from each other. Water is sprayed into the hot gases to cool them before subjecting them to the action of the electrical field.

—B. N.

Electrical purification of gases; Process and apparatus for —. H. Püning, Münster. Ger. Pat. 315,262, 22.1.18.

THE insulators are enclosed in downwardly-directed containers filled with heavy gases or with cooled gases; alternatively, upwardly-directed containers are used for light gases or the gases are heated during the operation. The filling of the containers with gases may be brought about by the introduction of small quantities of liquids which are converted into vapour. In this way the insulators are protected from dust and impurities. The loss by diffusion of the gas from the containers during purification is made up by the addition of more gas from time to time.—A. R. P.

Hot gases; Process for removing dust from —. R. Vetterlein, Schöningen. Ger. Pat. 303,831, 14.3.16.

THE hot gases are led through a spray of a salt solution which is thus evaporated, the dry salt falling to the bottom of the vessel together with any dust present in the gas, whilst the steam, together with any gaseous compound formed by interaction of the gases and the salt, passes into the original gas mixture. For example, the hot gases obtained by roasting pyrites are treated with a solution of a nitrate or nitrite whereby the latter is converted into a sulphate which falls to the floor together with any dust, and the gases are charged with steam and oxides of nitrogen.—A. R. P.

Leaching tank. W. E. Giesecke, Assignor to Ray Bros. Corp., New York. U.S. Pat. 1,330,463, 10.2.20. Appl., 31.3.17.

THE lower part of the wall of a tank, which may be shaped as a frustum of a cone, is perforated and covered with filtering material. An outer wall or a series of spaced shells are placed over the filtering area and are provided with a pipe and valve. There may also be another outlet in the bottom of the tank.—B. M. V.

Dissolved or suspended particles; Process for separating — from liquids. I. Ebers, Ahrensburg. Ger. Pat. 306,481, 22.6.15.

THE liquid is vaporised by heating to a temperature above its boiling point, and the vapours are carried forward with solids in suspension by a current of warm air and caused to pass through a filter medium which retains solid particles. The process is adapted to the evaporation of sea water, soap solutions for the production of soap powder, and to the preparation of dried milk.—H. J. H.

Volatile substances; Process for removing — from aqueous solutions. H. Wehner, Frankfurt. Ger. Pat. 314,518, 3.3.18. Int. Conv., 13.2.15.

By means of suction applied at the top, finely divided air is drawn through the liquid in vertical tubes formed with a series of alternate constricted and enlarged portions. The froth rises to the top of the tubes, and overflows to a horizontal main in which the air and vapour separate from the liquid, which flows away. If it is desired to effect a chemical reaction at the same time, means are provided for drawing an appropriate reagent into the bottom of the tubes.—H. J. H.

Chimney gases; Apparatus for cooling —. E. Eckmann, Harleshausen. Ger. Pat. 314,739, 25.5.17.

THE cooler consists of a transverse arrangement of air channels and a central air shaft, with water distributors between successive channels, the water being kept cool by introduction of fresh air and evaporation thereby reduced to a minimum.—W. J. W.

Fine-grained material; Process and apparatus for charging — into chambers with exclusion of air. K. Eltze, Mannheim-Käfertal, and R. Blume, Sürth. Ger. Pat. 315,091, 16.8.18.

THE process applies to the charging of silicon into caustic soda for the preparation of hydrogen. Water is forced through the mass of fine-grained silicon, thus driving it through a tube or channel into the gas-generator containing hot caustic soda solution and from which air is excluded. The pressure used in the operation is equal to the highest pressure in the generating vessel, so that regulation of the gas supply is possible.—A. R. P.

Decolorising material or filter-charcoal. Westfälische Mineralölwerke W. H. Schnitz und G. von der Heyde, Dortmund. Ger. Pat. 315,647, 23.3.16.

DECOLORISING materials of the nature of fullers' earth and filter-charcoal, are rendered more porous by addition of granulated slag produced in the blowing of basic pig-iron or other metallurgical product, which in itself possesses bleaching properties.—A. R. P.

Crushing stone, ore, and the like materials; Machines for breaking or —. E. Helme, Leeds. Eng. Pat. 138,387, 12.11.18. (Appl. 18,475/18.)

Grinding or pulverising ores, clinker, or the like; Process and apparatus for —. R. Forsyth, Hancock, Mich., U.S.A. Eng. Pat. 138,466, 13.5.19. (Appl. 6329/19.)

SEE U.S. Pats. 1,308,007—8 of 1919; this J., 1919, 612 A.

Electric precipitator. U.S. Pat. 1,329,237. See XI.

Heat-resisting packing. U.S. Pat. 1,330,148. See XIV.

Drying and thickening liquids. Ger. Pat. 310,192. See XIXa.

II A.—FUEL; GAS; MINERAL OILS AND WAXES.

Low temperature carbonisation; Investigation of the suitability of German coals for —. I. Coals from the German Upper Silesian coalfield. F. Fischer and W. Glud. Ges. Abhandl. Kenntn. Kohle, 1918, 3, 1—38. Chem. Zentr., 1919, 90, IV., 1064—1066.

PROXIMATE analyses of 25 coals from the area Czernitz-Birkental and Zabrze-Laurahütte are given, and the specific gravities at 25° and 50° C. of the low-temperature tars, together with their phenol content. The resulting semi-cokes are described and the yields of gas given. The revolving cylindrical retort previously described (this J., 1919, 563 A) was used, modified by the addition of a centrifugal tar extractor analogous to the Theisen washer. The caking coals gave a semi-coke in the form of balls which resisted crushing in the hand, but broke up when dropped on a stone floor. Non-caking coals gave a coke retaining the structure of the original coal, even when carbonised throughout, but varying much in hardness. Carbonisation in presence of steam frequently led to increased yield of tar. The yield and quality of the tars showed the Upper Silesian coals to be very well adapted to low-temperature carbonisation. The geologically youngest seam gave the highest yield of tar. Most of the Upper Silesian coals are gas coals resembling the younger formations of the Ruhr coal field, although some of the older formations are of the same geological epoch as the Ruhr lean coals. The great difference in tar yield, i.e., in the bitumen content of the two coals is to be explained by modification occurring since the strata were deposited. This may be a sort of condensation of the bitumen similar to that observed with low-temperature tar in an autoclave (cf. this J., 1920, 223 A). The variation in caking power may be connected with such a transformation. In order to get good compact coke from gas coal it is desirable to imitate, if possible, the natural transformation process or by working at a suitable temperature to transform the bitumen artificially. Possibly the behaviour on low-temperature coking may afford a new method of classifying coals.—H. J. H.

Low-temperature carbonisation; Investigation of suitability of German coals for — II. Coals from the Lower Rhineland and Westphalian district and Ibbenbüren. F. Fischer and W. Glud. Ges. Abhandl. Kenntn. Kohle, 1919, 3, 248—269. Chem. Zentr., 1919, 90, IV., 1066—1067. (See also preceding abstract.)

TWENTY-SIX samples of coal were examined covering all grades and including two cannels. The cannel gave the greatest yield of tar (37% on the ash and moisture-free coal). As the age of the coal increases the yield decreases, becoming zero with the less bituminous varieties. The tar from cannel was exceptionally low in phenol content. It consisted almost exclusively of hydrocarbons with an exceptional proportion of solid paraffins, being thus specially suited for lamp oil, lubricating oil, and paraffin production. The residual semi-coke was too friable to be used. The yield of tar from the long-flaming gas coals ranged from 6% to 16%, with a mean of 12% on the ash and moisture-free coal. The yield of phenol from these tars is the highest of all, reaching 40—45%. Contrary to the behaviour of the Upper Silesian coals which are otherwise very similar, they yield in most cases a poorly caking semi-coke. The coal from the upper seams, however, gave a fairly hard coke which might serve as a fuel without further treatment. These coals seem adapted for the production of low-temperature tar in gas producers and possibly even of a semi-coke. The more typical gas coals are characterised by the appearance of greater abnor-

malinity in the behaviour of individual coals not in accord with the geological stratification. The yield of tar from the gas coals varied in most cases from 5% to 10.7%, with an average of 8.2% on the ash and moisture-free coal. The phenol content was about 35%. The semi-coke obtained showed the beginning of marked caking power. The tar from the bituminous coals with lower volatile matter was 1.4%–5.3% with a mean of 3.8% on the ash and moisture-free coal. The phenol content of the tar was only 25%. The specific gravity of the tar was abnormal (1.09). The semi-coke was thoroughly caked. The non-bituminous coals give a considerable yield of volatile products and a semi-coke which resembled in external appearance the original coal. The Hbbenbürer coals resembled the older bituminous coals, and indeed were a transition between them and the non-bituminous kinds. They gave an average yield of 2–2.5% of tar and were unsuitable for low-temperature coking. The specific gravity and phenol content of the tar were unusually high.

—H. J. H.

Low-temperature carbonisation; Investigation of the suitability of German coals for —. III. Coals from the Saar district. F. Fischer and W. Glund. Ges. Abhandl. Kemtn. Kohle, 1919, 3, 270–286. Chem. Zentr., 1919, 90, IV., 1067–1068 (cf. preceding abstracts).

The Saar coals differ from those of the Ruhr and Upper Silesian coalfields in that oceanic influences have been absent during deposition and that the non-bituminous varieties do not occur in this region. They yielded from 6% to 15.6% of low-temperature tar. In specific gravity and phenol content the tars were as normally found with such rich coals. The semi-coke produced was very fragile and mostly fell to powder. That from the Wahlschieder seam was remarkable for its resemblance to wood charcoal.—H. J. H.

Low-temperature carbonisation. F. Fischer and W. Glund. Ber., 1920, 53, 250–251.

The authors maintain their scientific and technical priority against Tern (this J., 1920, 54A).—H. W.

Nitrogen of coal; Distribution of the — on low-temperature carbonisation. W. Glund and P. K. Breuer. Ges. Abhandl. Kemtn. Kohle, 1919, 3, 227–237. Chem. Zentr., 1919, 90, IV., 1066.

The distribution of nitrogen in Lohberg gas coal containing 1.86% N (maximum figure by Kjeldahl method) after distilling 10 kilos. in the revolving cylindrical retort as previously used (this J., 1919, 563A) was as follows: In the semi-coke 66.1%, as ammonia 1.8%, as pyridine 0.4%, in the tar 4.4%, as free nitrogen 10.8%. The sum of these is 83.5%, leaving 16.5% unaccounted for, which must be ascribed to the inexactitude of the Kjeldahl method (cf. Terres, this J., 1919, 399A). With coke the error may reach 0.5%, which suffices to account for the difference recorded. On simply heating the semi-coke it gives up as ammonia a further 16% of the nitrogen, reckoned on the original coal, i.e., in all 17.8%. It is concluded that in low-temperature carbonisation a satisfactory yield of ammonia can be obtained only by subjecting the coke to a further heating.—H. J. H.

Bitumen of Bohemian lignite. W. Schneider. Ges. Abhandl. Kemtn. Kohle, 1913, 3, 150–163. Chem. Zentr., 1919, 90, IV., 1072.

Its composition and calorific value, this lignite comes between the German lignites and coal, and it was examined with a view to discovering if other properties, e.g., the quality of the extract obtained under pressure, showed a similar approximation to those of coal. Two Bohemian lignites extracted

with benzene under pressure at 250°–260° C. gave an extract—bitumen B—which approximated to those obtained from German lignites. The portion of this extract soluble in light petroleum spirit was not a viscous oil similar to the corresponding extract from a bituminous coal, but a resinous sticky substance. The results obtained with the bituminous coal are not general, however, for the extract from a long-flaming gas coal was scarcely liquid at room temperature and the portion of it soluble in light petroleum spirit was a syrupy substance which acquired a resinous character.—H. J. H.

Lignite soluble in alkali from the Niederausitz district. W. Schneider and O. Jantsch. Ges. Abhandl. Kemtn. Kohle, 1918, 3, 164–190. Chem. Zentr., 1919, 90, IV., 1072–1073.

A TERTIARY lignite worked near Guben in Eastern Lausitz for use as a pigment and fertiliser was examined and 93–94% of the material dried at 105° C. was found to be soluble in alkali. It consisted mainly of substances resembling humic acid. A portion of the lignite is comparatively rich in bitumen. Soxhlet extraction with benzene yields about 15% of crude montan wax (bitumen A); further extraction under pressure gives 9% of bitumen B. On distilling the lignite in a retort about 16% of tar is obtained. With respect to the production and yield of tar by distillation it resembles the lignites of Saxon-Thüringen, and it is recommended that such treatment should precede the utilisation of the material for colours and as a fertiliser. The humic acids dissolved by 5% ammonia out of the material extracted by benzene under pressure, on dry distillation gave very little tar. The solubility of the humic acids of this lignite was almost the same in solutions of sodium hydroxide, sodium carbonate, and ammonia, whether cold or warm. This does not hold good generally with lignites. The humic acids when heated to 260° C., as in the pressure extraction, are converted into products which are insoluble in cold alkalis, although almost completely dissolved on warming. Where it is desired to retain the humic acids unaltered it is therefore desirable to extract with aqueous ammonia before extraction under pressure. The ammonia solution, however, takes up a portion of both the bitumen A and bitumen B. By a preliminary steaming at 100° C. this can be minimised by rendering the bitumen insoluble in ammonia. Pure montanic acid was practically insoluble in 10 times its weight of 5% aqueous ammonia. From crude montan wax under the same treatment only 0.5% passed into solution.—H. J. H.

Formolite reaction, and interaction of methylal with unsaturated cyclic hydrocarbons. L. G. Radcliffe. Perf. Ess. Oil Rec., 1920, 11, 48–50.

Using the modification devised by Herr (this J., 1910, 1094), with methylal in place of formaldehyde, the author has attempted to correlate formolite numbers (Nastjukoff, this J., 1904, 1082), iodine values, and yields of solid oxidation products (prolonged action of air in presence of metallic catalysts) of mineral oils, but no very characteristic relationships were observed. Qualitatively, the methylal reaction enables 1% of benzol to be detected when mixed with light petroleum spirit. The terpenes and cyclohexane were non-reactive, whilst the xylenes, *p*-cymene, and naphthalene gave the test, which thus appears to be discriminatory for benzenoid hydrocarbons.—A. E. D.

Pyridine from coke-ovens. Dodge and Rhodes. See III.

Gas-fired blast-furnace. Torkar. See X.

PATENTS.

Fuel briquettes; Manufacture of —. H. G. Hills, Bramhall. Eng. Pat. 138,414, 31.1.19. (Appl. 2441/19.) (See also Eng. Pats. 3284 of 1913 and 18,576 of 1914; this J., 1914, 245; 1915, 949.)

ANTHRACITE dust (say 4 parts) is mixed cold with crude coal tar (1 part). The mixture is placed in specially constructed open shallow trays and distilled in an oven, heated externally to a temperature not exceeding 600° C. The briquettes are improved by placing loose weights upon them during the heating.—W. P.

Pulverised fuel; Combustion of solid —. A. Assereto, Savona, Italy. Eng. Pat. 138,502, 23.4.19. (Appl. 10,175/19.)

THE fuel falls directly from the pulveriser into a mixing chamber, where it is mixed with a current of air, which keeps it in suspension. It is withdrawn from here and introduced into the furnace. The feed hopper is thus eliminated and the liability of obstruction lessened.—W. P.

Coke oven with alternative heating arrangement for rich or poor gas; Horizontal regenerative —. H. Engbert, Heerde. Ger. Pat. 312,181, 5.6.18.

THE velocity of the gas and air mixture is increased by reducing the cross-sectional area of the heating flue and lengthening the passage, thereby attaining a rapid and complete combustion and a more favourable heat transmission owing to the higher velocity. The rich or poor gas burners are distributed uniformly over the whole heating surface, whilst air, poor gas, or products of combustion flow through the combustion chambers in a horizontal diagonal direction.—J. F. B.

Distillation residues [coke]; Process for cooling —. E. Zbinden, Zurich. Ger. Pat. 314,661, 2.12.17.

THE hot coke from the carbonising plant is transferred to closed vessels fitted with suitable pipe connections and jackets. Air is drawn through the coke, and thence by an opening to the annular space round the vessel. The air, now hot and containing more or less carbon monoxide and dioxide, is passed on through a jacket surrounding a steam boiler or other waste heat appliance. The air thereby cooled is then returned to the hot coke chambers, circulation being maintained by means of a pump.—H. J. H.

Gas producers. L. Fornas, Paris. Eng. Pat. 123,323, 12.2.19. (Appl. 3431/19.) Int. Conv., 13.2.18.

IN a gas producer of the single or multiple type the feeding tubes are in the form of truncated cones. As combustion progresses the fuel descends from the tubes into a dryer, and thence into the producer chamber. Air is forced downward through the producer from the chamber surrounding the dryer.—W. P.

Gas producer. G. H. Benjamin, New York. U.S. Pat. 1,329,744, 3.2.20. Appl., 19.6.18.

A GAS producer is provided with a tunnel-kiln, and a series of cars for holding the material to be decomposed and progressively moving it through the kiln. A series of grate-bars is carried by each car, with means to cause them to revolve and discharge ash. Gas and air burners, separately controlled, are located on opposite sides of the kiln. The cars are provided with means for supplementing the heat set free from the gas and air burners, and the gaseous body produced is exhausted from the kiln.—B. N.

Gas producer. O. A. Winter, Buxtehude. Ger. Pat. 313,032, 22.6.17.

THE producer, provided with an outlet for producer gas, is fitted with an inner retort through which distillation gases are withdrawn separately. The retort is provided with holes in the walls and is rotated. Within the retort is a core for distributing the bituminous fuel. By the rotation of the retort the distillation gases are forced continuously to find new paths through the pasty mass of bituminous fuel.—J. F. B.

Gases; Apparatus for cooling and scrubbing —. A. M. Hunt, Berkeley, and D. E. Fogg, Oakland, Cal. U.S. Pat. 1,327,599, 6.1.20. Appl., 25.5.14.

A TOWER has a gas inlet at one side above the bottom and a gas outlet at the top; banks of cooling coils are situated in the tower above and below the gas inlet; means are provided for spraying a liquid on the pipes in a counter direction to the currents of gases; the cooling pipes are connected with a header in the tower, and means are provided for maintaining a constant circulation, with intermittent cooling, of a second liquid, through the header and pipes.—J. F. B.

Hydrocarbon and other compounds; Apparatus for the electro-chemical treatment of liquid —. L. B. Cherry, Kansas City, Mo. U.S. Pat. 1,327,023, 6.1.20. Appl., 26.2.16.

A LIQUID hydrocarbon compound is treated by introducing a gas carrying hydrogen and vaporising the liquid. Means are provided for treating the mixture of vapour and gas electrically, so as to produce a compound of different gravity from that of the original liquid, and the mixture is heated whilst being treated.—B. N.

Gasoline from natural gases; Apparatus for recovering —. A. B. Cross, Denver, Colo. U.S. Pat. 1,327,906, 13.1.20. Appl., 5.3.18.

A CYLINDRICAL receiver is surrounded by a jacket, caps being provided on the ends of the jacket, and having packing boxes through which the ends of the receiver pass; a pipe on one end of the receiver is connected to a gas supply. The opposite end of the receiver is connected by a pipe to the inlet of a pressure regulator, and the outlet of the pressure regulator is connected to one end of the jacket. An outlet pipe extends from the opposite end portion of the jacket, a drain pipe connects the jacket with a tank, and another drain pipe connects the receiver near its outlet end with the tank.—J. F. B.

Hydrocarbons; Process of treating —. B. Andrews, Houston, Tex., and W. C. Averill, jun., Meraux, La. U.S. Pat. 1,329,739, 3.2.20. Appl., 19.9.18.

HYDROCARBON oil is injected into tubes containing melted metal, and the resulting vapours and melted metal are circulated through the tubes, the hydrocarbon vapours being then withdrawn.—B. N.

Sulphonic acids; Process of recovering — from mineral-oil sludge. R. E. Divine, Assignor to The Twitchell Process Co., Cincinnati, Ohio. U.S. Pat. 1,330,624, 10.2.20. Appl., 24.6.18.

A NEARLY colourless alkaline-earth sulphonate is prepared from the sludge formed by sulphonating a portion of a mineral oil distillate by mixing the sludge with water, separating free oil, neutralising the solution with an alkaline earth, and salting out the sulphonates from the solution with a soluble salt of the same alkaline earth.—L. A. C.

Petroleum, tar, and the like; Preheater for use in the distillation of —. L. Steinschneider, Brünn, Austria. U.S. Pat. 1,302,988, 6.5.19. Appl., 17.9.13.

SEE Ger. Pat. 308,768 of 1917; this J., 1919, 167 A.

Tar oils for Diesel engines. Ger. Pat. 315,030. See III.

Nitrogen, hydrogen, etc. Ger. Pat. 303,881. See VII.

Refuse-destroyer furnaces. Eng. Pat. 133,297. See XIX u.

Furnace-gases. Eng. Pat. 138,156. See XXIII.

Gas constituents. U.S. Pat. 1,316,602. See XXIII.

IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Arc lamps; New cadmium vapour —. F. Bates. Phil. Mag., 1920, 39, 353—358.

AN inverted U-tube of 10 c.c. capacity is fitted at its lower ends with two long quartz capillaries by means of which tungsten wire electrodes are admitted. The electrodes are fastened into the capillaries by lead seals. The lamp is filled by distilling (at 0.001 mm. pressure), from a quartz bulb attached at the bend of the U-tube, a gallium-cadmium alloy containing 2—3% of gallium. The presence of the gallium renders the cadmium soft and so prevents the breakage of the lamp when the metal solidifies. When sufficient metal has been distilled over the lamp is sealed off. The lamp will burn with 3 amps. at 110 volts with a drop of 14 volts across the terminals, but is more efficient with a current of 7 amps. and a drop of 25 volts. To start the lamp one limb must be heated with a Bunsen flame. The light is that of almost pure cadmium vapour, and the lamp furnishes a source of monochromatic red light of wave length $\lambda 6439 \text{ A.}$ —J. F. S.

Acetyl content of wood. Pringsheim and Magnus. See V.

PATENTS.

Destructive distillation of wood; Process for recovering oils through the —. D. L. Hanson, Wilmington, N.C., Assignor to Pine Nene Products Co., Jacksonville, N.C. U.S. Pat. 1,330,632, 10.2.20. Appl., 7.2.19.

To prevent decomposition of the gases and vapours from the destructive distillation of wood they are continuously removed by the combined action of a vacuum pull towards the retort outlet and the pressure of an oxidising agent introduced at a point remote from the outlet.—W. P.

Arc lamp carbons, especially for searchlights. Allgem. Elektrizitäts-Ges., Berlin. Ger. Pat. 307,073, 6.11.17.

THE carbons consists of an inner cylindrical part over which is drawn an outer tubular part, either or both of which are provided with longitudinal grooves, which are partly or wholly coppered. The molten copper, as long as it is not vaporized by the arc, flows along the grooves.—A. R. P.

Graphite electrode, specially suitable for searchlights. C. Conradty, Nürnberg. Ger. Pat. 310,603, 25.9.18.

THE electrode consists of flake graphite bound with tar or other suitable organic bond which, on heating in the electric furnace, changes to graphite.

The flakes are so arranged that they lie in the direction of the current.—A. R. P.

Decolorising carbon. U.S. Pat. 1,327,222. See XVII.

III.—TAR AND TAR PRODUCTS.

Phenols; Hydrolysis of phenoxycetic acid with alkali and preparation of pure —. F. Fischer and W. Glund. Ges. Abhandl. Kenntn. Kohle, 1918, 3, 75—81. Chem. Zentr., 1919, 90, IV., 1052.

IN order to identify the individual phenols of low-temperature tar separated as phenoxycetic acids (cf. Glund and Breuer, this J., 1920, 151 A), it was necessary to recover therefrom the free phenols in satisfactory yields. Acids proved ineffective. By the action of 4 mols. of potassium hydroxide on 1 mol. of the phenol derivative at about 275° C. almost pure phenol was obtained with a 50—75% yield. For each phenol the optimum reaction temperature must be determined by trial. A tightly closed, and continuously shaken, electrically heated Mannesmann tube was used as autoclave. The results are tabulated to show the yield of pure phenol set free, and the proportion remaining as acetyl derivative; the difference between the sum of these and 100 gives the loss on working:—

Acid.	Temp.	Mols. KOH.	Free phenol.	Combined phenol.
Phenoxycetic acid ..	250° ..	4 ..	52.1 ..	14.5 ..
..	300° ..	3 ..	57.0 ..	— ..
o-Cresoxycetic acid ..	275° ..	4 ..	73.2 ..	2.5 ..
p-Cresoxycetic acid ..	275° ..	4 ..	72.2 ..	9.0 ..
Thymoxycetic acid ..	275° ..	4 ..	48.5 ..	46.2 ..

—H. J. II.

Phenol; Determination of small quantities of — in mixtures of phenols. F. Fischer and P. K. Breuer. Ges. Abhandl. Kenntn. Kohle, 1918, 3, 82—88. Chem. Zentr., 1919, 90, IV., 1032.

THE method is based on a suggestion of Müller (Lunge-Köhler, 1912, p. 762) that when homologous phenols are fractionally precipitated by a restricted amount of hydrochloric acid, ordinary phenol is precipitated last in a relatively pure condition. Thus 10 grms. of pure phenol was dissolved in 90 grms. of mixed cresols (b. pt. 200°—202° C.) from a low-temperature tar and 192 c.c. of 5N sodium hydroxide was added. This solution was extracted twelve times with 157 c.c. of 5N hydrochloric acid, followed by vigorous shaking with 150 c.c. of ether for 15 minutes and, after separating the ether layer, with a further 100 c.c. of ether, the last extraction being made after the addition of salt. The united ether extracts were evaporated at the ordinary temperature to avoid loss of phenol. The eleventh acid extract gave 7 grms. of crystallisable phenol, and from the sixth and succeeding extracts was recovered altogether 92% of the phenol taken, in the form of a mixture with cresol melting at 26° C.—H. J. II.

Phenol content of low-temperature tar and over-heated tars. F. Fischer and P. K. Breuer. Ges. Abhandl. Kenntn. Kohle, 1918, 3, 89—97. Chem. Zentr., 1919, 90, IV., 1070—1071.

THE low-temperature tar from coal contains only about 0.06% of phenol. Normal coal tar results from the cracking of low-temperature tar at 700°—750° C. and contains about 1% of phenol. It is possible that a considerably higher percentage than this could be realised. By the improved analytical methods described (Fischer and Breuer, preceding) a producer low-temperature tar was shown to contain 0.21% of phenol. The total phenol fraction from a large quantity of this tar was cracked at 700°—750° C., and the phenol content was thereby

doubled, presumably by degradation of the higher phenols. As the quantity of tar was thereby halved, the concentration of the phenol was increased fourfold, i.e., to 0.97%. The original producer tar from a long-flame gas coal from the Saar field contained more phenol than coke-oven tar from the Ruhr field. Lohberg coal distilled in the revolving retort (this J., 1919, 563A) gave a tar containing only 0.06% phenol.—H. J. H.

Low-temperature coal tar; Suitability of — for the production of coumarone-resin. W. Gluud and P. K. Breuer. Ges. Abhandl. Kennt. Kohle, 1919, 3, 238—242. Chem. Zentr., 1919, 90, IV., 1071.

If the fraction 155°—185° C. of low-temperature tar is shaken with strong sulphuric acid, a dark brown soft product is obtained amounting to 0.05% of the tar. Thus the ordinary process does not lead to the formation of coumarone-resins. If, however, the phenols are cracked by heating to a suitable temperature and the appropriate fraction is collected, a typical yellow hard coumarone-resin is subsequently obtained. Apparently the coumarone-resins are produced by substances which originate by the cracking of the phenols in low-temperature tar.—H. J. H.

Distillation; Brown's formula for [fractional] — [applied to benzene-toluene mixtures]. S. Young. Sci. Proc. Roy. Dublin Soc., 1920, 15, 667—672.

BROWN'S formula $M'_A/M'_B = C M_A/M_B$ (where M'_A , M'_B and M_A and M_B are the relative numbers of molecules of A and B in the vapour and liquid respectively, and C is a constant depending on the relative vapour pressures of the pure substances at the boiling point of the mixture; Chem. Soc. Trans., 1879, 547; 1880, 49; 1881, 304, 517) is applicable without serious error to mixtures of benzene and toluene, of which the vapour pressure P approximately equals $MP_A + (1-M)P_B$, P_A and P_B being the vapour pressures of the two pure substances at the same temperature, and M the molar fraction of the substance A. Further, for benzene and toluene the best value of the constant C differs but slightly from the ratio P_A/P_B , viz., 2.591.

—G. F. M.

Pyridine; Recovery of — from by-product coke-ovens. F. E. Dodge and F. H. Rhodes. Amer. Inst. Chem. Eng., Dec., 1919. Chem. and Met. Eng., 1920, 22, 274—275.

THE quantity of pyridine bases passing the tar separators amounts to about 1 lb. per ton of coal coked, and these bases are absorbed by the acid solution in the ammonia saturation plant. No pyridine sulphate was found in the ammonium sulphate crystals when the mother liquor contained 2.5% pyridine. If the mother liquor is neutralised by bubbling through it a gas rich in ammonia, the heat generated by the reaction is sufficient to raise the solution to its boiling point and to distil off the liberated pyridine bases. In a plant designed for the recovery of pyridine, the acid mother liquor, saturated with pyridine salts, is treated with ammonia from an ammonia still. Vaporised pyridine passes through a condenser system to receivers, and the residual liquor in the still is acidified before returning it to the ammonium sulphate plant. The condensed pyridine bases are separated from the accompanying water by the addition of solid ammonium sulphate, the dense solution of ammonium sulphate then being drawn off.—C. A. K.

Formolite reaction. Radcliffe. See IIA.

Cresols and cresol-soap substitutes. Hailer. See XIXb.

PATENTS.

Tar oils for Diesel engines; Process for improving —. F. Raschig, Ludwigshafen. Ger. Pat. 315,030, 27.4.15.

THE tar oil is shaken with a suitable quantity of water and the aqueous layer is separated. The latter removes mainly substances containing chlorine, which owing to corrosive action on the oil-inlet valves render the tar oil unsuitable for Diesel engines.—H. J. H.

Chlorobenzol; Method of nitrating —. R. Heyder, Forest Hills, N.Y., Assignor to Williamsburg Chemical Co., Inc., Brooklyn, N.Y. U.S. Pat. 1,330,074, 10.2.20. Appl., 26.4.18.

CHLOROBENZOL is treated with a nitrating agent, and the mixture is maintained below about 65° C. during the greater part of the time necessary to complete the reaction.—L. A. C.

Farnish from indene. Ger. Pat. 310,783. See XIII.

Aromatic amines. Eng. Pat. 138,372. See XX.

IV.—COLOURING MATTERS AND DYES.

Butyl alcohol; Use of — as a solvent for anthocyanins. O. Rosenheim. Biochem. J., 1920, 14, 73—74.

BUTYL alcohol dissolves anthocyanins much more readily than does amyl alcohol. It is therefore the best solvent from which to isolate these substances in the pure condition. (See also J. Chem. Soc., April, 1920.)—J. F. S.

Glyoxal from acetylene. Wohl and Bräunig. See XX.

PATENT.

Azo-dyestuffs; Manufacture of — and intermediate products therefor. O. Imray, London. From Soc. of Chem. Ind. in Basle, Switzerland. Eng. Pat. 137,733, 12.5.19. (Appl. 11,891/19.)

α -HYDROXYNAPHTHALENESULPHOCARBOXYLIC acids are obtained from naphthalenedisulphocarboxylic acids by partial fusion with 40% sodium hydroxide for 3—4 hours at 195° C. under pressure in an autoclave, and precipitation from the reaction mixture with hydrochloric acid. Mordant-dyeing azo dyestuffs are produced from these acids by coupling with aromatic o-hydroxydiazocompounds, or preferably their o-carboxylic acids, i.e., o-diazo derivatives of salicylic acid. Coupling is effected by mixing equimolecular proportions of a solution of the diazonium compound with a concentrated solution of the mono-sodium salt of the α -hydroxynaphthalenesulphocarboxylic acid containing 2 mols. of sodium hydroxide and about 4 mols. of anhydrous sodium carbonate. The precipitation of the dye is complete in about 12 hours. Examples are given of the coupling of 4-nitro-2-diazo-1-hydroxybenzene-6-carboxylic acid and 1-hydroxynaphthalene-3-sulpho-6-carboxylic acid to an azo-dye giving on wool in an acid bath red shades which when after-chromed change to a bordeaux fast to fulling and potting. In chrome printing on cotton it yields a bordeaux fast to soap, chlorine, and light. 4-Sulpho-2-diazo-1-hydroxybenzene-6-carboxylic acid couples with either 1-hydroxynaphthalene-3-sulpho-5-carboxylic acid or -6-carboxylic acid to give a red wool dye which on after-chroming becomes red-violet and gives a fast red-violet in chrome printing on cotton. The analogous 4-chlorodiazohydroxybenzenecarboxylic acid gives on coupling a red dye becoming a fast violet on after-chroming or in chrome printing on cotton.—G. F. M.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cotton; Deterioration of — on wet storage. N. Fleming and A. C. Thaysen. *Biochem. J.*, 1920, 14, 25—28.

DETERIORATION of damp cotton is brought about by a *streptothrix* and by a *schizomycete*, both of which cause a breaking down of the cuticle. Deterioration is retarded if the moisture content of the cotton falls below 9%. The amount of deteriorated cotton may be estimated microscopically by counting the fibres after the sample has been treated by Ball's viscose process.—J. F. S.

Broom; Treatment of — with alkali for the production of fibres suitable for spinning. H. Kämpf. *Mitt. Deuts. Forschungsinst. Textilstoffe*, 1918, 255—300. *Chem. Zentr.*, 1919, 90, IV., 1062.

DIGESTION with alkali alone does not give a satisfactory product while retting alone does not suffice for the isolation of the fibres. If carried too far the latter causes partial attack of the fibre itself. A combination of the two processes, retting followed by treatment with alkali, gave a pure, almost colourless, strong but soft fibre, which can be used as substitute for more costly materials. The retting is best prolonged over several days until the bast is readily loosened. Afterwards the material is treated with 0.5—1% caustic soda solution for 2—3 hours at ordinary pressure or 2 atm. pressure. The hydrochloric acid furfural reaction affords a method of control. The pentosan number is connected in certain cases with the tensile strength of the product.—H. J. H.

Wood; Acetyl content of —. H. Pringsheim and H. Magnus. *Z. angew. Chem.*, 1920, 33, 56.

THE authors reply to a criticism by Schwalbe and Becker (this J., 1920, 185 A) of their previous communication in regard to the respective amounts of acetic acid obtained from pine woods and wood from deciduous trees. Beechwood lignin contains 37.8% acetyl as against 19.85% in pine wood lignin. Boiling the wood for 3 hours with 2.5% sulphuric acid is not sufficient to extract all the acetic acid. It is necessary to treat the wood for 6 hours with eight times its quantity of 3.5% sodium hydroxide under a pressure of 6 atms.—W. J. W.

Cotton hull fibre for pulp; Cooking —. O. Kress. *Paper*, 1920, 25, 961—968, 1009—1011. (See also this J., 1919, 858 A.)

EXPERIMENTS were carried out on the large scale on the digestion of cotton hull fibre in plant employed for the manufacture of soda wood pulp. On account of the spongy nature of the material the circulation of the liquor had to be assisted by an external auxiliary pump and a somewhat high ratio of liquor to fibre (7:1) had to be employed. A maximum steam pressure of 80—85 lb. per sq. in. was used; with 45 lb. steam pressure the disintegration of the hull particles was far from satisfactory. Owing to the high liquor ratio the proportion of caustic soda (3% concentration) used was somewhat excessive, viz., 21—21% of the weight of the charge. The maximum pressure was reached in 4 hours and the digester was kept under 80—85 lb. pressure for a further 4 hours. The dry nature of the stuff caused considerable difficulty in blowing out the charge after digestion in the manner used for wood pulp. The ordinary treatment of soda wood pulp after washing in the diffusers is not suitable for cotton hull pulp owing to the length and "freeness" of the fibre. The hydration of the cellulose is developed with considerable difficulty, and the best results would probably be

obtained by starting the beating treatment before the washing of the pulp was complete, so that the roll of the washing engine would brush the fibre with considerable pressure while still in the alkaline condition. The removal of the cellular residues of the disintegrated hulls during washing is desirable for the manufacture of strong papers; it is, however, more rational to remove the hull particles themselves as completely as possible by suitable mechanical treatment of the raw fibre before digestion. The paper produced is not so strong as that produced from hard rag stock, but resembles paper from soft "seconds" or "thirds" rags; this, however, is largely a matter of the development of hydration, and the fibre calls for a heavier crushing treatment in the beater without undue reduction in length. The stock is cleaner than that prepared from munition linters, being freer from extraneous dirt; with suitable digestion no difficulty is experienced in bleaching economically to a good white colour.—J. F. B.

Turbidimeter for cellulose. Sheppard. *See* XXIII.

PATENTS.

[*Fabric*] *articles; Process and apparatus for drying —.* D. K. Tullis, Clydebank. Eng. Pat. 137,427, 12.3.19. (Appl. 6175/19.)

THE fabric articles are placed on a travelling apron which carries them round a heated roller in order to preheat them and then transfers them to a closed drying chamber through which they are led, exposed to hot air currents, in a zig-zag course by means of tapes in such a manner that the exposed surfaces are repeatedly changed by reversing the goods each time they pass from one set of tapes to another. Inside the drying chamber a number of pairs of superposed rollers are arranged in stepped relationship and tapes are wrapped around the pairs of rollers, the tapes wrapped round one pair of rollers being staggered in relation to the tapes wrapped round the pair of rollers immediately beneath.—J. F. B.

Fabric; Process of treating — [with lubricant]. W. C. Carter, Radnor, Ohio, Assignor to The Goodyear Tire and Rubber Co., Akron, Ohio. U.S. Pat. 1,327,904, 13.1.20. Appl., 19.10.16.

YARN or fabric is bathed in a hot vapour and a relatively cold body of liquid carrying the lubricant in suspension is then brought in contact with the yarn or fabric, whereby the vacuum formed by condensation of the vapour causes impregnation of the material.—J. F. B.

Vegetable proteins; Product derived from — [for manufacture of artificial silk, films, etc.]. Z. Ostenberg, San Jose, Cal. U.S. Pat. 1,316,854, 23.9.19. Appl., 12.2.17.

A NON-THERMOPLASTIC material suitable for the manufacture of artificial silk, films, etc., is composed of a mixture of a vegetable prolamine, i.e., a plant protein soluble in 70% alcohol (e.g., gliadin, zein) and a small quantity of a toughening agent (fat, wax, resin, soap, sulphonated oil, sugar, glycerin, phenols, terpenes, etc.). The product may be rendered resistant to water by treatment with formaldehyde or the like.

Yarn windings, particularly paper yarn; Apparatus for drying —. E. Jagenberg, Düsseldorf, Germany. U.S. Pat. 1,330,025, 3.2.20. Appl., 9.5.17.

YARN windings, such as paper yarn on cops, crossed reels, and the like, are dried in an apparatus comprising an air-tight casing, means for producing a current of air through the casing, nozzles on the casing which lead the air current through the cops or cross reels, and means for heating the

air current. Valves are fitted over the nozzles, and are opened by means of stems when reels are fitted on to the nozzles.—J. F. B.

Pulp; Method of preparing —. C. Bache-Wüg, Portland, Me. U.S. Pat. 1,327,221, 6.1.20. Appl., 20.2.19.

CELLULOSIC material is treated with sodium chloride and the mass is subsequently cooked with bisulphite liquor until the ligneous matter has been separated from the fibre.—J. F. B.

Rotary digesters and like boilers. K. Hellner, Falun, Sweden. Eng. Pat. 137,742, 28.5.19. (Appl. 13,436/19.)

IN rotary digesters for the manufacture of cellulose and the like the liquor is heated with indirect steam in order to avoid dilution by condensed water. Heating elements are provided outside the digester in such a manner that they take part in its rotation and are connected with the inside of the digester in such a manner that the liquor can pass constantly through the heating elements and be heated by means of steam pipes located therein, the supply of steam being passed through one trunnion of the digester, while the condensed water is discharged through the other.—J. F. B.

Fireproof [pyroxylin] composition. J. C. Emhardt, Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,329,386, 3.2.20. Appl., 25.2.18.

A COMPOSITION containing pyroxylin and ammonium magnesium phosphate.—J. F. B.

Cellulose esters; Process for softening —. F. Redlich, Zehlendorf-Mitte. Ger. Pat. 304,224, 14.2.17.

CELLULOSE esters are incorporated with the neutral ester of a di- or poly-basic organic acid of the aliphatic or aromatic series with the aid of a volatile solvent. Resins, fats, and oil may also be incorporated by dissolving them in the neutral ester or in the volatile solvent. For example, 12 kilos. of cellulose acetate is mixed with 10 kilos. of diethyl tartrate.—A. R. P.

Paper; Process of re-pulping old or waste —. J. M. Burby, Webster, Mass. U.S. Pat. 1,327,590, 6.1.20. Appl., 14.4.16.

OLD paper made from mechanical wood pulp is converted into chemical wood pulp by boiling the paper in a solution of chemicals adapted to convert the woody fibre into cellulose.—J. F. B.

Carbon-paper and ink composition therefor. H. Ohashi, New York, N.Y.; M. V. Ohashi, administratrix. U.S. Pat. 1,328,188, 13.1.20. Appl., 5.12.16.

PAPER is treated with ink prepared with the reaction products of an aniline dye soluble in water, calcium chloride, an emulsifying agent, and an emulsifiable substance.—J. F. B.

Paper; Apparatus for making ornamental —. E. Mahler, Assignor to Kimberly Clark Co., Neenah, Wis. U.S. Pat. 1,329,130, 27.1.20. Appl., 7.9.17.

A FOURDRINIER wire is arranged to move in a straight line free from lateral shake while the paper is being formed on the wire, thereby causing the formation of depressions and elevations in the paper web. Liquid colour is sprayed by means of a gas under pressure on to the upper surface of the wet plastic web of paper moving with the wire, the spray impinging on the paper at an acute angle to the horizontal.—J. F. B.

Paper and pasteboard pulp; Process for engine-sizing of —. Zellkoll Ges. m. b. H., Cologne-Rodenkirchen. Ger. Pat. 303,828, 11.8.16.

THE sizing agent is an easily dissociated organic salt of aluminium such as the acetate or formate, which may be obtained by the interaction of aluminium sulphate or alum and an easily obtainable salt of the organic acid, such as the barium salt. The use of soap or resins in addition is not necessary.—B. V. S.

Safety paper. E. Haussmann, Berlin. Ger. Pat. 303,989, 6.3.17.

A COMPOSITE paper has a middle sheet uniformly coloured with an easily reacting colouring matter, and on either side of this one or more covering films of a thin absorbent paper, only slightly coloured if at all. The colouring matter of the inner sheet is destroyed if the outer surface is attacked by chemical means, and the inner sheet is soon exposed to view by abrasion.—B. V. S.

Size for paper; Production of —. A. Mitscherlich, Freiburg. Ger. Pat. 314,652, 22.9.15.

TANNERY glue is treated with waterglass and the dark-coloured substance which is deposited after some time is separated. The purified clear liquid is added to the paper pulp in the hollander.—H. J. H.

Paper-making machines; Press-roll arrangement for —. J. K. Darby, Chillicothe, Ohio. U.S. Pat. 1,327,225, 6.1.20. Appl., 7.6.16.

Paper-making machine. N. J. Nicks, Kalamazoo, Mich. U.S. Pat. 1,327,289, 6.1.20. Appl., 3.9.18.

Lanolin. U.S. Pat. 1,330,210. See XII.

Tanning material. Ger. Pat. 304,349. See XV.

Films or filaments. Eng. Pat. 138,379. See XXI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Amylolytic action. Waksman. See XVIII.

PATENTS.

Finishing and waterproofing textile fabrics; Process for —. E. Naefe, Berlin-Britz. Ger. Pat. 314,969, 9.12.15.

THE fabric is treated in a solution of the sodium ammonium salt of alginic acid, and is then passed through a bath of zinc sulphate or similar heavy metal salt. The insoluble salt of alginic acid produced separates in a colloidal form giving a waterproof finish which resists a boiling neutral dye-bath.—H. J. H.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Ammonia; Catalytic oxidation of —. B. Neumann and H. Rose. Z. angew. Chem., 1920, 33, 41—44, 45—48, 51—55.

THE authors give an extensive survey of the numerous processes and researches in connection with the catalytic oxidation of ammonia and contribute some results of their own investigations. Special attention has been directed to the influence of the ammonia concentration, the velocity of the gas stream, the temperature, and of various catalysts including platinum, metallic oxides, and mixtures of the latter. The highest yields are obtainable with platinum at 500° C. (96%); ferric

oxide at 670° C. (90%); and iron/bismuth oxide at 600° C. (95%). From curves plotted to show the gas composition at approximately 300°–700° C. with the above catalysts, it is apparent that, under fixed conditions, the oxidation will proceed continuously without extraneous heat. With platinum gauze of 3000 meshes per sq. cm. as catalyst, the best yield (96%) was obtained with a velocity of gas current of 20 litres per hour and the yield was not greatly affected by increase of the ammonia concentration from about 3 to 9% by vol. With ferric oxide the best velocity was about 22 litres per hour (about 90% yield).—W. J. W.

Acid industry; [Use of] Raschig's rings in —.

H. Schellhaass. Chem.-Zeit., 1920, 44, 122–123.

ATTENTION is directed to the advantages attained by the use of Raschig's rings as a filling material for absorption towers in the manufacture of sulphuric acid, nitric acid, hydrochloric acid, etc. The rings are made of earthenware in various sizes, usually 120 mm. in height and 100 mm. in diameter (external), the thickness of the wall being about 10 mm.—W. P. S.

Sulphur; Production of — [from gypsum] in Germany. O. F. Kaselitz. Z. angew. Chem., 1920, 33, 49–51.

ONE of the processes developed in Germany during the war to produce the sulphur required for sulphuric and sulphurous acid manufacture consisted in reducing gypsum with coke, and converting the resultant sulphur dioxide into trioxide by a contact process. Cement is manufactured from the by-products. For the production of sulphur gypsum is heated with coke or coal in a rotary furnace to 1100° C., and the calcium sulphide is treated with steam to form hydrogen sulphide, which is finally oxidised in a contact furnace. The purity of the product is 99.95%.—W. J. W.

Ammonia-ammonium thiocyanate; Equilibrium in the system —. H. W. Foote and M. A. Hunter. J. Amer. Chem. Soc., 1920, 42, 69–78.

SOLUTIONS of ammonia saturated with ammonium thiocyanate (prepared from dry ammonia gas and solid ammonium thiocyanate) have the following vapour pressures: –78° C., 1 mm.; –65°, 4 mm.; –50°, 9 mm.; –34°, 21 mm.; –23°, 34 mm.; –20°, 41 mm.; 0°, 107 mm., and +20° C., 225 mm. Saturated solutions of ammonium thiocyanate in ammonia contain the following amounts of ammonia; at 0° C., 23.3; 10°, 22.65; 20°, 21.6; 30°, 20.15; 40°, 18.4; and at 50° C., 16.65%. The practical application of ammonium thiocyanate as a commercial absorbent for ammonia is discussed. It could be used in cases where it is necessary to eliminate water. For example, from the equilibrium mixture of nitrogen, hydrogen, and ammonia, produced in the synthesis of ammonia, the use of ammonium thiocyanate would permit of the recovery of 98.1% of the ammonia at –20° C., 98.9% at –30°, and 99.6% at –50° C. (See also J. Chem. Soc., Apr., 1920).—J. F. S.

Equilibria in solutions containing mixtures of salts. II. The system water and the chlorides and sulphates of sodium and magnesium at 25° C. W. C. Blasdale. J. Ind. Eng. Chem., 1920, 12, 164–167.

PREVIOUS work on this subject (*cf.* Hildebrand, this J., 1918, 180 A) indicates that the solid phases to be expected at 25° C. are $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$, $\text{MgSO}_4 \cdot \text{H}_2\text{O}$, $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$, Na_2SO_4 , $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, NaCl , and $\text{MgSO}_4 \cdot \text{Na}_2\text{SO}_4 \cdot 4\text{H}_2\text{O}$ (astrakanite). Experiments were made to ascertain the limits of the fields representing the composition of all the solutions which can be in equilibrium with each of the eight solid phases. The results are shown in a curve in which the horizontal axis is used to represent the number of mols. of MgSO_4

and NaCl , and the vertical axis the number of mols. of MgCl_2 and Na_2SO_4 , per 1000 mols. of water. The compositions of the solutions which represent critical points are given in the following table:—

Composition (grms. per 100 grms. of water) of solutions saturated at 25° C.

Saturated with:	MgSO_4	Na_2SO_4	NaCl	MgCl_2
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	36.39	—	—	—
$\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$	—	27.94	—	—
NaCl	—	—	35.63	—
$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	—	—	—	54.97
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ and astrakanite	32.07	19.68	—	—
$\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ & astrakanite	25.36	27.09	—	—
$\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ and Na_2SO_4	—	21.68	18.82	—
Na_2SO_4 and NaCl	—	9.81	32.16	—
MgCl_2 and $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	—	—	—	54.87
$\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ & $\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$	3.61	—	—	54.01
$\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$ & $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	6.37	—	—	49.74
$\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$, Na_2SO_4 , and astrakanite	7.71	18.67	15.50	—
Na_2SO_4 , NaCl , & astrakanite	12.59	1.01	29.67	—
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$, and astrakanite	12.03	—	9.79	20.47
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$, and NaCl	6.53	—	2.20	39.94
$\text{MgSO}_4 \cdot 6\text{H}_2\text{O}$, NaCl , and $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	2.53	—	0.18	53.88

—S. S. A.

Cyanide; A new —. [Calcium cyanide, from calcium cyanamide.] W. S. Landis. Amer. Electrochem. Soc., Jan., 1920. Chem. and Met. Eng., 1920, 22, 265–268.

THE conversion of calcium cyanamide into calcium cyanide by fusion with sodium chloride was first attempted in a resistance furnace of the tilting type. Calcium cyanamide was fed by hand into a bath of molten sodium chloride, and the molten charge was tapped off and cooled. Considerable difficulty was experienced by the increase in conductivity of the magnesite brick lining of the furnace due to the reduction of the iron content to the metallic state by the cyanide. The excessive foaming produced on the addition of calcium cyanamide was controlled by adding a small quantity of calcium carbide. It was not found necessary to keep the reaction temperature below 960° C. as in previous German practice. Later developments included the use of a single-phase furnace with conducting hearth and a single suspended electrode, together with a considerable reduction in the quantity of salt used. A large furnace has been successfully operated with a charge consisting of two parts of calcium cyanamide to somewhat less than one part of salt. A product containing as high as 50% equivalent sodium cyanide has been obtained, and the cyanogen content has been proved in practice relatively as efficient in the extraction of precious metals as that of 98% sodium cyanide, the cost of production of which is considerably higher. Analysis indicates that the product is a mixture of calcium cyanide, sodium chloride, and free lime.

—C. A. K.

Sodium fluoride; Case of poisoning by —. O. Vallée. J. Pharm. Chim., 1920, 21, 5–8.

SEVERAL persons suffered severe illness after eating pancakes in the preparation of which, as was discovered subsequently, sodium fluoride had been used accidentally in place of sodium bicarbonate. The amount of sodium fluoride taken was, in most cases, about 0.228 gm.—W. P. S.

Hydrogen and oxygen mixtures; Catalysis of — at the ordinary temperature by moistened contact substances.—II. Platinum metals as hydrogen carriers. K. A. Hofmann and L. Zipfel. Ber., 1920, 35, 298–314. (Compare this J., 1917, 136.)

MIXTURES of oxygen and hydrogen are catalysed

at palladium, platinum, or iridium surfaces at very different rates, depending on the condition of the metal with regard to its gas charge. Pre-treatment with oxygen is 30–50 times as effective in the case of palladium, 3 times with platinum, and 3–10 times with iridium as is pre-treatment with hydrogen. The greater activity caused by charging with oxygen is not due to the adsorbed or occluded gas serving as a source for the formation of water, since the amount of metal required for catalysis is insufficient to produce this effect, but depends on the production of a "fresh" hydrogen-metal combination, which, in the cases of palladium and platinum, is far more active than an "aged" preparation. The catalyst shows greater activity in proportion as this "fresh" condition is more rapidly and completely developed. In the case of iridium another factor is involved, since the metal, in presence of gases containing excess of oxygen, can function as an active oxygen electrode. The close parallelism between catalytic and electromotive activity indicates that both effects are to be ascribed to the same cause. This probably depends on the presence of free or metallically-combined hydrogen atoms, which cause the hydrogen potential and ultimately combine with the oxygen of the gas mixture to form water. Under the experimental conditions adopted an active oxygen electrode is only formed with finely-divided iridium when the oxygen concentration is high; platinum and palladium behave entirely as hydrogen electrodes. The maximum velocity of formation of water is not invariably observed when the gases are used in the proportion of 2 vols. of hydrogen to 1 vol. of oxygen, but, according to conditions, may occur at a greater or smaller oxygen concentration according as the catalyst becomes more or less readily saturated with hydrogen. If the catalyst can also function as an oxygen electrode, as in the case of finely-divided iridium, a second maximum may occur at a higher oxygen-content of the gas. It may be stated generally that the surface does not show its greatest catalytic activity when hydrogen and oxygen are absorbed according to the measure of their combination at an electrically-neutral electrode, but that the maximum possible hydrogen or oxygen potential must be "freshly" developed in order to react with the gaseous mixture at the maximum rate.—H. W.

PATENTS.

Sulphuric acid; Process for the preparation of — from naturally occurring sulphates. B. Dirks, Hemelingen. Ger. Pat. 301,791, 24.3.17.

NATIVE sulphates, such as gypsum or anhydrite, are transformed by known methods into ammonium sulphate, which is mixed with metallic oxides, especially iron oxide or hydroxide (*e.g.*, limonite), and the mixture slowly heated to 250° C. until the ammonia is expelled. On further heating to redness, sulphur trioxide is evolved in almost quantitative yield. The residue of iron oxide is moistened with water and used again in the process. Substitution of alumina or chromic oxide for iron oxide does not give such good results; manganese, nickel, and cobalt oxides give quantitative yields, but their sulphates are more difficult to decompose.—A. R. P.

Synthetic ammonia; Transformation of — into a transportable product directly utilisable for agriculture in conjunction with the production of sodium carbonate. L'Air Liquide Soc. Anon. pour L'Etude et L'Exploit. des. Proc. G. Claude, Paris. Eng. Pat. 131,870, 14.3.19. (Appl. 6448/19.) Int. Conv., 24.8.18.

The process described in Eng. Pat. 130,365 (this J., 1919, 690 A) is improved by adding to the liquid

from which sodium bicarbonate has been precipitated, sea salt, ammonia, and carbon dioxide, the sea salt being added in such limited quantities that it remains dissolved throughout the operations, and so does not mix with the ammonium chloride. The solution is cooled to 0°–5° C.; the ammonium chloride precipitated is separated, centrifuged, and heated to about 70° C. to destroy alkalinity. The remaining liquor is treated with sea salt and carbon dioxide, thereby precipitating sodium bicarbonate and forming a fresh ammonium chloride liquor. For the production of the low temperature required, liquid ammonia is evaporated in a coil in direct contact with the ammonium chloride to be cooled, and the resulting gaseous ammonia is passed into cooled brine contained in the dissolving and carbonating tank. The expansion of the ammonia with external work may be utilised for the production of additional cooling effect, and the evaporation of liquid or solid carbon dioxide may also be utilised for cooling purposes.—S. S. A.

Ammonium nitrate; Manufacture of —. J. R. Partington, Lostock Gralam, and G. J. Jones, London. Eng. Pat. 134,562, 1.2.18. (Appl. 1874/13.)

AMMONIUM nitrate, produced in the form of a mist by the interaction of ammonia gas and nitric acid or by other means, is caused to pass through a dehydrating agent, such as sulphuric acid, and can then be readily condensed and collected. Very little mist is absorbed by the sulphuric acid.

—W. J. W.

Ammonium nitrate; Production of —. J. R. Partington, G. J. Jones, and T. K. Brownson, London. Eng. Pat. 136,190, 4.3.18. (Appl. 3792, 18.)

AMMONIUM nitrate is manufactured by bringing together oxides of nitrogen, oxygen, water, and ammonia in such proportions that the oxygen is in excess of the theoretical quantity required to convert the oxides of nitrogen into nitrogen peroxide, the ammonia is insufficient to react with the whole of the oxides of nitrogen, and the water does not exceed the amount necessary to convert all the ammonia into ammonium nitrate by interaction with the oxides of nitrogen. Where gases containing about 10% of oxides of nitrogen are utilised, the amount of oxygen should be approximately three to four times the theoretical quantity, and time should be allowed for the reaction to take place before admixture with the ammonia. If the gases after any stage of the process still contain oxides of nitrogen, a further quantity of oxygen, ammonia, and water may be added to produce more ammonium nitrate. By this process less ammonium nitrate is lost than by other methods of manufacture.—W. J. W.

Tungstates; Method of obtaining pure —. J. B. Ekeley and W. B. Stoddard, Boulder, Colo., U.S.A. Eng. Pat. 138,211, 20.3.19. (Appl. 6999/19.)

TUNGSTATE solutions prepared as described in Eng. Pat. 122,051 (U.S. Pat. 1,255,144; this J., 1918, 206 A) are purified by the process described in Eng. Pat. 122,264 (this J., 1919, 134 A), but both the oxidising agent (sodium hypochlorite) and the ammonia are added subsequently to the addition of the alkaline-earth salt.—S. S. A.

Alkali chromates and iodine; Manufacture of —. R. L. Datta, Calcutta, India. Eng. Pat. 138,291, 23.9.19. (Appl. 23,375/19.)

ALKALI iodide in admixture with an excess of sesquioxide of chromium or of chrome iron ore is heated to redness in a current of air and steam.

The whole of the iodine volatilises and is collected in a condenser. The residue is lixiviated with water to recover alkali chromate.—S. S. A.

Barium chloride and other chlorides of the alkaline-earth metals; Preparation of —. F. B. Shroff, Gwalior, India. Eng. Pat. 138,486, 1.4.19. (Appl. 8152/19.)

For the production of barium chloride free from barium sulphate or barium hydroxide, barytes is mixed with coal or other carbonaceous material and a quantity of magnesium chloride mother liquor, and introduced into a retort from one end and heated to about 600° C., steam being injected near the exit end to complete the reaction. The gases evolved are collected in a gas holder and the sulphur is recovered by burning the hydrogen sulphide in a Claus kiln. By lixiviation of the retort charge, barium chloride is dissolved, leaving a residue of magnesium oxide. The magnesium oxide may be dissolved out with sulphuric acid and recovered as magnesium sulphate, or the barium may be precipitated as sulphate by sulphuric acid, forming hydrochloric acid, which may be used to dissolve the magnesium oxide, thus producing magnesium chloride for subsequent operations. The process is applicable to the production of the chlorides of other alkaline-earth metals.—S. S. A.

Ferric sulphate; Basic — and method of preparing same. J. MacKaye, Cambridge, Mass., Assignor to River Smelting and Refining Co., St. Louis, Mo. U.S. Pat. 1,316,909, 23.9.19. Appl., 13.9.18.

A basic ferric sulphate, soluble in dilute sulphuric acid, is prepared by heating granular ferrous sulphate in contact with air at 200°–300° C., e.g., in a rotary cylinder provided inside with radial fins.

Alkaline icaters [brines]; Process of treating —. N. T. Bacon, Peace Dale, R.I., Assignor to The Solvay Process Co., Solvay, N.Y. U.S. Pat. 1,330,573, 10.2.20. Appl., 20.6.19.

IN a process of precipitating sodium carbonates from alkaline brine, the latter is treated first with flue-gas to effect a partial conversion of the lower carbonates into bicarbonate, and then with pure carbon dioxide to complete the conversion.

—W. E. F. P.

Manganese dioxide; Process of making conducting hydrated black —. C. Ellis and A. A. Wells, Montclair, N.J., Assignors to National Carbon Co., Cleveland, Ohio. U.S. Pat. 1,330,738, 10.2.20. Appl., 26.9.16.

By the oxidising action of a reactive hypochlorite compound on a slightly acid solution of a manganese salt in a mineral acid at about 100° C., a product suitable for use as a depolarising agent is obtained.—S. S. A.

Ferrous perchlorate; Preparation of pure, stable, technical —. E. Merck, Darmstadt. Ger. Pat. 309,133, 11.6.18.

AN impure solution of ferrous perchlorate is purified by digesting it with ferrous or ferric oxide, hydroxide, or carbonate, or with finely-divided metallic iron, whereby a brown gelatinous precipitate is produced which carries down with it the impurities present. The purified solution and the crystals obtained from it are much less readily oxidised, and also show a greater stability towards moisture than the original material.—A. R. P.

Perorides; Process for the production of stable solid —. W. Trumpp, Mannheim. Ger. Pat. 310,193, 16.3.18.

A PEROXIDE is mixed with a salt, the acid radicle

of which forms a soluble compound with the base of the peroxide; for example, equivalent parts of sodium peroxide and magnesium sulphate are digested with a very small quantity of water until interaction is complete, and the resulting mixture is dried.—A. R. P.

Lime; Process and apparatus for slaking —. J. Joachim and J. Schulte, Berlin. Ger. Pat. 315,241, 2.8.18.

WATER to be used for slaking lime is previously converted into steam by passing it through pipes embedded in the lime being slaked. The process is carried out in a drum around the inner wall of which a tubular worm is fixed; one end of the latter is connected to the water supply and the other end terminates in a steam pipe. The heat of combination of the lime and water is sufficient to transform more than the necessary water into steam.

—A. R. P.

Nitrogen, hydrogen, and carbon dioxide; Process for the preparation of technically pure — from natural gas and air. E. Herman, Buda-Pesth. Ger. Pat. 303,881, 12.4.11.

METHANE (natural gas) is burnt with the calculated quantity of air to give nitrogen and carbon dioxide, while the heat evolved is utilised to split up another portion of the gas into carbon and hydrogen. Both the combustion and decomposition of the methane are carried out under pressure as well as the separation of the carbon dioxide and water from the other products. At 950° C. methane is completely decomposed into carbon and hydrogen; at lower temperatures only partly so.—A. R. P.

Chromate and bichromate of sodium and potassium; Process for the conversion of — to chromic oxide and sulphate. F. M. Mooney, Montreal. U.S. Pat. 1,330,131, 10.2.20. Appl., 21.4.19.

SEE Eng. Pat. 129,958 of 1919; this J., 1919, 681 A.

Bleaching-powder; Manufacture of —. A. Rudge, Gateshead. U.S. Pat. 1,330,495, 10.2.20. Appl., 25.3.19.

SEE Eng. Pat. 126,773 of 1918; this J., 1919, 462 A.

Hydrogen; Producing —. C. Bosch, A. Mittasch, and C. Beck, Ludwigshafen, Germany, Assignors to Chemical Foundation, Inc. U.S. Pat. 1,330,772, 10.2.20. Appl., 4.6.14.

SEE Ger. Pat. 279,582 of 1913; this J., 1915, 355.

Removing dust from gases. Ger. Pat. 303,831. See I.

Burning lime etc. Ger. Pat. 314,586. See I.

Charging fine-grained material. Ger. Pat. 315,091. See I.

Roasting furnaces. Eng. Pat. 138,472. See X.

Blast-furnace slags. Ger. Pat. 309,134. See X.

Electrolytic apparatus. Eng. Pat. 137,553. See XI.

Oxidising mercury. Eng. Pat. 137,609. See XI.

Electrolytic cell. Eng. Pat. 138,406. See XI.

VIII.—GLASS; CERAMICS.

PATENTS.

Glass furnace. Naaml. Vennoots. Glasfabriek "Leerdam" voorheen Jeekel, Mijnsen, and Co., Leerdam, Holland. Ger. Pat. 314,605, 6.12.18. Int. Conv., 11.1.18.

THE furnace is of the tank type fired with liquid

fuel. The jet of liquid fuel is driven into the furnace at the working end and under pressure so regulated that the highest temperature is developed at a distance from the burner at the melting end of the furnace where the fresh batch is fed in. In this way the jet of fuel on entering the furnace passes over the molten glass and not over the cold raw batch which might otherwise take up carbonaceous matter and yield discoloured glass.—H. J. H.

Quartz glass; Method of building up objects of — W. S. Quimby and F. W. Robinson, Assignors to Hanovia Chemical and Manufacturing Co., Newark, N.J. U.S. Pat. 1,330,611, 10.2.20. Appl., 23.11.18.

ARTICLES made of quartz glass are produced by heating a nucleus of quartz glass until it is plastic, applying a coating of powdered quartz, and reheating until the latter is fused, whilst at the same time elongating the article so as to keep its diameter constant. (See also Eng. Pat. 131,233 of 1919; this J., 1919, 769 A.)—A. B. S.

Plastic [ceramic] composition. E. J. Buckley, Longton, Staffs. Eng. Pat. 138,477, 24.3.19. (Appl. 7319/19.)

A PERMANENTLY plastic material which can, if required, be made hard by firing, is made by mixing any suitable pottery body with petroleum jelly or similar hydrocarbons. The mixture may be coloured with underglaze colours or metallic oxides.—A. B. S.

Refractory compounds; Process for producing — W. M. Handy, Spokane, Wash. U.S. Pat. 1,330,263, 10.2.20. Appl., 3.6.18.

A BASIC refractory material is made by calcining a pulverised mixture of calcined magnesite, an iron compound, and silica so as to cause the oxides of magnesium and iron to unite.—A. B. S.

Dryer for ceramic ware. A. Bühner, Constance. Ger. Pat. 312,765, 10.9.15. Addition to 311,258.

FOR use with certain classes of goods, for which moist air is preferable to hot dry air, the apparatus described in the chief patent (this J., 1919, 636 A) is modified by providing direct communication, which can be controlled by a damper, between the moist air flue and the fan chamber.—J. W. D.

IX.—BUILDING MATERIALS.

PATENTS.

Cement; Waterproof — S. Cabot, Canton. Assignor to S. Cabot, Inc., Boston, Mass. U.S. Pat. 1,305,645, 3.6.19. Appl., 26.2.16.

A COMPOSITION for use in the production of waterproof cement consists of powdered pitch (from coal tar, blast-furnace tar, coke-oven tar, or petroleum) brought into paste form in water by means of a protective colloid such as dextrin, soap, or the like. The composition may be mixed with slaked lime, clay, etc., before incorporation with the cement.

Slaking lime. Ger. Pat. 315,241. See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron in iron ores; Determination of — by permanganate. L. Brandt. Chem.-Zeit., 1920, 44, 101—103, 121—122.

A METHOD described by Schwarz and Rolfe (this J., 1919, 179 A) is further criticised (see this J., 1919,

662 A); the author maintains that the addition of colloidal silica does not hinder the oxidation of the hydrochloric acid.—W. P. S.

Blast-furnace; Gas-fired — A. Torkar. Mitteil. Inst. Kohlenvergasung, 1919, 1, 81—85. Chem. Zentr., 1920, 91, 11., 36—37.

LIGNITE cannot be used directly in a blast furnace in place of coke; the gas obtained from it is, however, of high calorific value, and may be used both as a reducing and heating agent in the furnace. A shorter, broader furnace is most suitable for this work; the hot gases are passed downwards through the column of ore, thereby reducing it with consumption of one-fifth of the gas. Another fifth of the original gas mixture is burnt by the highly heated air-blast at the tuyères, and the remainder passes out of the furnace unburnt, and may be used for the production of power. Comparison with the coke-fired blast furnace and with electric smelting shows this process to be most economical.—A. R. P.

Spiegeleisen in steel manufacture; Use of — H. D. Hibbard. Report to U.S. Bureau of Mines, Jan. 7, 1918. Chem. and Met. Eng., 1920, 22, 209—210.

FOR convenience iron-manganese alloys containing 40% Mn or less are called spiegeleisen, alloys richer in manganese being termed ferro-manganese. For low-carbon steel the high ratio of manganese to carbon (about 12 to 1) in ferro-manganese is an important advantage. Spiegeleisen containing 31.25% Mn has been used on a commercial scale in basic open-hearth and Bessemer practice, but although the product is fairly free from red-shortness the ingot is too "thin skinned," so that seams, pits, and scabs are sometimes produced during reheating and rolling. The manganese content of the finished steel is 50—70% of that added. At another works the alloy is added in a molten condition, and for steels containing more than 0.3% C the use of spiegeleisen is considered to be the most effective method of adding manganese. The successful use of spiegeleisen is not yet proved in the manufacture of steel containing less than 0.1% C, low-carbon steels requiring a fine surface, ingots for plates or better quality sheets or castings made by the converter process where plants are not provided with means for melting re-carburising agents.—C. A. K.

Tin-plate; Use of hydrogenated oils in the manufacture of — W. D. Collins and W. F. Clarke. J. Ind. Eng. Chem., 1920, 12, 149—152.

OF various suggested substitutes for palm oil (see U.S. Pat. 1,242,532 of 1917) for covering the coating bath in the manufacture of tin-plate, hydrogenated cottonseed oil gave the most satisfactory results. Experiments on the effect of heat on various oils indicated that a satisfactory hydrogenated fish oil might be obtained. Laboratory tests showed that hardened cottonseed oil might be used without the evolution of unpleasant odours or excessive loss from volatilisation or from polymerisation. A commercial test during twelve weeks showed somewhat better results with hardened cottonseed oil than with palm oil; less oil was used, but no saving of tin was effected.—S. S. A.

Iron; Lead-coated — C. Baskerville. J. Ind. Eng. Chem., 1920, 12, 152—154.

To eliminate rupture or scaling of the protective lead coating applied to iron work, antimony may be used as a binding agent. The iron is pickled in sulphuric acid (6:1), washed with water, thoroughly blackened with an antimony chloride solution, again washed with water, and while still wet dipped through zinc chloride flux into molten lead-antimony alloy (12.5% Sb), then into molten lead, and finally quenched in oil. The process gave satisfactory results in the case of "shingles," iron wire

and netting, but was impracticable in the case of barbed wire, iron pipes, and shell cases on account of the irregularity of their surfaces. By the introduction of a preliminary pickling in hydrofluoric acid the process may be applied to cast-iron for filter presses and filter frames, an alloy coating being produced on to which a thick lead or other metal coating may be cast. Processes have been devised for plating lead-coated iron with copper or nickel or both.—S. S. A.

Brasses; Heat treatment of beta —. H. M. Brayton. Chem. and Met. Eng., 1920, 22, 211–217.

A COMMERCIAL brass containing 56.17% Cu, 41.24% Zn, 2.53% Pb, and 0.06% Sn showed a progressive fall in strength as the time of heating at 800° C. was prolonged. This is due first to the elimination of the effects of cold work on the original material, followed by a reduction due to the rapid grain growth at high temperatures. Heating at 600° C. for two hours, and quenching, gives the maximum strength combined with high ductility. Slow cooling from temperatures higher than 500° C. without mechanical work on the metal reduces the tensile strength, and large alpha crystals separate from the beta alloy. This treatment will produce an alloy possessing excellent shock-resisting properties if strength is not of vital importance. Similar experiments on an alloy containing 60.5% Cu, 38.67% Zn, and 0.75% Sn showed that the reduction in area falls off when the alloy is quenched from a temperature above 600° C., but that this property is not diminished appreciably if the metal is cooled slowly from temperatures up to 800° C.—C. A. K.

Alloys of copper, zinc, and nickel. L. Guillet. Comptes rend., 1920, 170, 460–462.

BRASSES, containing nickel, in which the copper content falls to 40% and even less and in which the nickel content rises to 15% are of particular interest because of their mechanical properties. Their breaking strain varies from 45 to 75 kilos. per sq. mm., with elongations of 45–18%. They are readily malleable at high temperatures, and in colour and resistance to oxidation they resemble German silver.—W. G.

Copper, silver, and gold; Growth phenomena of —. A. Beutell. Centr. Min., 1919, 14–28.

WHEN silver foil and silver sulphide or selenide or telluride were heated together *in vacuo* in a sealed tube at temperatures between 450° and 600° C. for several days remarkable growths of "hair" or "moss" silver were observed in the cooler end of the tube, the silver appearing to wander through the sulphide, from which the growths appeared to come. To obtain the growths it was necessary for the metal and the sulphide to be actually in contact. When silver sulphide was heated alone under similar conditions a small quantity of "hair" silver was formed, due to the partial decomposition of the sulphide. Much more rapid growths of hair silver were obtained when the tubes in which the heating was carried out had access to the air. Silver is not attacked by sulphur vapour *in vacuo* at 450° C. Copper gave similar growths, but much more slowly. In the case of gold the best results were obtained by heating gold telluride containing silver with silver foil for seven days at 500° C., when growths of "moss" gold containing silver appeared at the cooler end of the tube.—E. H. R.

Lead; Attempt to determine if common — could be separated into isotopes by centrifuging in the liquid state. J. Joly and J. H. J. Poole. Phil. Mag., 1920, 39, 372–375.

CENTRIFUGING liquid lead gives no evidence that a separation of isotopes has been effected.—J. F. S.

Alloys; Effect of centrifuging certain — in the liquid state. J. Joly and J. H. J. Poole. Phil. Mag., 1920, 39, 376.

CENTRIFUGING molten silver-lead alloys at a rate of 9000 revolutions per minute effects no separation, but similar treatment of lead-tin alloys brings about a certain amount of separation. Thus, an alloy containing 82 Pb and 18 Sn gives a top layer which is 1.8 lighter than the bottom layer.

—J. F. S.

Metals; Variation of thermal conductivity during the fusion of —. S. Konno. Sci. Rep. Tôhoku Imp. Univ., 1919, 8, 169–179.

THE thermal conductivity of tin, lead, zinc, and aluminium decreases with rise of temperature from ordinary temperatures up to the melting point. On fusion it drops considerably and on further increase of temperature it decreases slightly. The thermal conductivity of bismuth decreases to a minimum (at 160° C.) and then rises until the melting point is reached, where it increases considerably and then decreases slightly with further rise in temperature. In the case of antimony the conductivity falls to a minimum (at 182° C.), then rises to the melting point, where it decreases slightly and continues to decrease with further rise of temperature.—J. F. S.

New cyanide. Landis. See VII.

PATENTS.

Steel and other metals of high melting point; Reverberatory furnace for smelting —. J. Schmid, Lucerne, Switzerland. Ger. Pat. 313,919, 19.4.18.

ONE side of a chamber for melting steel or other metals is provided with three inlets so arranged that continuations of the same would meet in the centre of the chamber. Through the lowest opening flame from the white-heat zone of a gas producer passes into the chamber; the middle opening conveys combustible gas into the chamber; and the top opening admits the air necessary for combustion. Two gas producers, separated by a perforated wall, are attached to the chamber, and the gas generated in the producer filled with fresh coal is heated by means of the other producer in which flame formation takes place.—L. A. C.

Ores; Process and apparatus for treating — in the blast furnace. L. P. Basset, Paris. Eng. Pat. 112,275, 21.12.17. (Appl. 18,974/17.) Int. Conv., 22.12.16. Addition to Eng. Pat. 109,452, 7.9.16 (this J., 1918, 771 A).

READILY burning carbon in a very finely divided state, preferably in the form of "flour," is delivered from a hopper by an endless screw, into one of the tuyères of a blast furnace in the correct amount to bring about reduction of the ore, and from a second hopper powdered anthracite is similarly supplied in amount sufficient to effect the desired carburisation of the reduced iron. (Reference is directed to Eng. Pat. 109,452; *loc. cit.*)—T. St.

Alloy, and method of making and using same. A. G. Mumford, Ltd., and A. G. Mumford, Colchester. Eng. Pat. 138,228, 14.1.19. (Appl. 9160/19.)

A WHITE-METAL alloy consisting of Sn 61, Cu 29.7, and Zn 9.3, is alloyed with copper in the proportion Cu 80.36, white-metal alloy 19.64%. The resulting alloy is cast in iron chills to produce floats for automatic feed water regulators and the like, which are subjected to very high and varying temperatures and pressures.—T. St.

Electrodes of electric [smelting] furnaces; Manufacture of —. O. R. Olsen, Christiania, Norway. Eng. Pat. 138,272, 6.8.19. (Appl. 19,383/19.)

ELECTRODES for use in smelting copper, iron, and

other ores are prepared by adding to the usual constituents about 25% of the concentrate of the ore under treatment.—W. E. F. P.

Furnace; Mechanical — for roasting ores. Huntington, Heberlein, and Co., Ltd., and H. C. Bingham, London. Eng. Pat. 138,443, 22.2.19. (Appl. 4474/19.)

IN an ore-roasting furnace of the type having superposed hearths on which operate rabbles carried by a central shaft, the space beneath the muffle hearth is divided into two annular concentric flues, and the heating gases from the firing chamber are made to pass first through the inner flue and then, in the opposite direction, through the outer flue. The temperature thus produced on the muffle hearth is sufficient to ensure the decomposition of sulphates before the roasted ore is discharged from the furnace. By dividing the flues, by means of tiles, into upper and lower passages, the lower concentric flues thus formed serve for the supply of preheated air, which may be used either in the combustion chamber or in the muffle as desired.—T. St.

Roasting furnaces for sulphurous ores. Soc. Anon. de Vedrin, and J. Marcotty, Vedrin, Belgium. Eng. Pat. 138,472, 18.3.19. (Appl. 6739/19.)

AN apparatus for the continuous and intensive roasting of sulphurous ores, particularly blende, comprises an inclined, cylindrical, rotary furnace, the upper part of which is divided into several radial compartments which are subdivided lengthwise into sections which are offset or placed in alternation with one another, the lower part being provided with vanes which raise the material and drop it through the current of gas. The ore is thus subjected to a creeping motion and to successive overturnings in the upper part of the furnace and then spread through the current of oxidising gases in the lower part, whereby a maximum oxidation with a reduced amount of dust is obtained.—S. S. A.

Reverberatory furnace, for melting metals and alloys of all kinds. F. Wüst, Aix-la-Chapelle. Ger. Pat. 315,264, 4.1.17.

THE furnace is provided with a shaft situated at the opposite end to that at which the fuel and air are admitted. The metal is charged into the shaft and the hot flue gases leaving the furnace play around and through the lower part of the column of the charge, thereby melting it. The shaft is so constructed that the burning gases are concentrated into a pointed flame.—A. R. P.

Sherardising apparatus. C. J. Kirk, New Castle, Pa. U.S. Pat. 1,327,102, 6.1.20. Appl., 1.3.19.

THE apparatus comprises a rotary retort with external and internal electric heating units.—B. N.

Ores; Separation of —. A. A. Lockwood, Merton Park. U.S. Pat. 1,329,127, 27.1.20. Appl., 24.11.17.

A LAYER of pulp containing the material to be separated is caused to flow over a supporting surface; the pulp is agitated and baffled during its passage over the surface, and electric currents are passed through the pulp between baffling elements, thereby evolving gas which adheres to and buoys up certain of the components of the pulp.—J. F. B.

Gold; Production of white —. D. Belais, New York. U.S. Pat. 1,330,231, 10.2.20. Appl., 5.10.18.

A WHITE gold alloy is composed of a major part of gold and lesser proportions of nickel and zinc.—T. St.

Zinc-retort residues; Process for working up —. K. Eichhorn, Hildesheim. Ger. Pat. 309,162, 29.3.18.

THE residues are smelted in the producers which supply the gaseous fuel for the distillation furnace. The gases are freed from zinc oxide and then used for heating the retorts. The heat generated in the producers is more than sufficient to melt the metals as well as the gangue, which are tapped off as usual.—A. R. P.

Copper alloys containing valuable metals; Process for treatment of —. Hirsch Kupfer- und Messingwerke A.-G., Halberstadt. Ger. Pat. 312,394, 13.3.18. Addition to 301,265 (this J., 1920, 161A).

TO obtain the copper sulphate which must necessarily be added to maintain the concentration of the bath during the electrolysis of blister copper, the slag from the furnace in which the copper has been melted is treated with a portion of the electrolyte drawn from the bath. Copper present in the slag, as metal, or as cuprous and cupric oxides, silicates, and ferrites, is rapidly extracted, especially if the slag is first crushed and calcined. The silica then separates out more rapidly, and the copper, being oxidised to cupric compounds, is more rapidly extracted.—H. J. H.

Ductile metal in wire or tube form; Production of —. L. Weiss, Barmen. Ger. Pat. 314,791, 7.1.14.

THE metal is precipitated upon a core of the same or another metal which under the conditions is inert, by heating a volatile salt of the metal, which either dissociates at a high temperature or is reduced by an admixed gas, e.g., hydrogen. The resulting rod can be either wire drawn or rolled out. Two or more metals, either together or separately in layers, can be deposited by suitably choosing the materials and conditions, so that alloys which are otherwise unobtainable can be prepared. Volatile chlorides or metallo-organic compounds, such as the metal alkyls and the acetoacetates, are suitable materials. Thus a wire of platinum or tungsten heated electrically in a mixture of tungsten hexachloride and hydrogen can be coated with a bright, dense, adherent layer of tungsten, the thickness of which depends on the temperature and time of the heating.—H. J. H.

Annealing and melting metals and alloys without oxidation; Process for —. A. Barth, Frankfurt. Ger. Pat. 314,990, 22.5.18.

THE metal is heated after applying a layer of suitable protective material, e.g., phosphoric acid or fusible phosphates. The process may be applied to the fusion of turnings, to casting, and enamelling processes.—H. J. H.

Aluminium alloys containing copper, tin, antimony, and magnesium; Production of —. A. Manhart, Vienna. Ger. Pat. 314,999, 23.5.18. Int. Conv., 10.4.14.

IN aluminium alloys a portion of the copper—say one-third—can with advantage be replaced by manganese-copper alloy. The resulting alloys are hard without being brittle.—H. J. H.

Alloy suitable for protective coatings for iron and steel. Akt.-Ges. der Dillingen Hüttenwerke, Dillingen. Ger. Pat. 315,074, 20.10.18.

THE alloy contains 0.5 part of aluminium, 1 part of antimony, 1 part of tin, and 97.5 parts of soft lead. Instead of antimony, an antimonial lead with 8–10% Sb may be used. The aluminium is first warmed, and the fused hard lead added, then the tin, and lastly the soft lead.—H. J. H.

Metal matrix; Preparation of a — to obtain easily detachable electro-deposits. M. Schlöter, Berlin-Wilmersdorf. Ger. Pat. 315,711, 1.12.17.

THE matrix is treated with an aqueous solution of gelatin, glue, or similar material which, by treatment with reagents or light, is rendered insoluble. The coating should be so thin that it offers no noticeable resistance to the current; it withstands a bath temperature of more than 50° C.—A. R. P.

Blast-furnace slags; Treatment of — [for production of sulphur dioxide]. H. Petersen, Berlin-Steglitz. Ger. Pat. 309,134, 26.5.18.

THE slag is tapped from the blast-furnace into an insulated ladle, from which portions are removed to smaller vessels in which they are treated with a current of air to convert the contained sulphur into sulphur dioxide, which is used for making sulphuric acid. Two or more vessels may be used simultaneously for the operation in such a manner that the first half of the treatment in the second vessel corresponds with the second half of the treatment in the first vessel, and *vice-versa*. In this way a regular supply of sulphur dioxide is obtained.

—A. R. P.

Furnaces for heating [metal] blooms [; Slide tracks for —]. M. Mathy, Flemalle-Grande, Belgium. Eng. Pat. 138,712, 21.2.19. (Appl. 4413/19.)

XI.—ELECTRO-CHEMISTRY.

Electrode; Simple hydrogen —. C. H. Bailey. J. Amer. Chem. Soc., 1920, 42, 45—48.

A SIMPLE hydrogen electrode may be constructed as follows:—A piece of 7 mm. bore tube is blown out at one end to form a bulb of 15 mm. diam.; the tube is then bent to form an angle of about 45° at a distance of 50 mm. from the top of the bulb. The other limb of the tube is about 80 mm. long and is fitted with a ground glass stopper. The metal electrode is a thin platinised gold disc of 5 mm. diam. welded to a piece of thin platinum wire and fused into the shorter limb of the tube about 5 mm. above the bend. The tube is filled with the liquid under investigation and then by means of a fine tube the bulb is filled with hydrogen, thus expelling some of the liquid. The tube is stoppered, care being taken to exclude air, and vigorously shaken until the solution is saturated with hydrogen. Connection is made with the standard electrode by means of a siphon tube filled with saturated potassium chloride solution and inserted well down the longer limb. The electrode is rapid in action and gives reliable results.—J. F. S.

PATENTS.

Electrolytic cell [for oxidising mercury]. H. W. Matheson, Shawinigan Falls, Canada. Eng. Pat. 138,406, 23.1.19. (Appl. 1731/19.)

AN electrolytic cell for oxidising mercury consists of a shallow, insulated metal pan or anode, covered by a hood, and containing a layer of mercury above which a metal cathode is suspended. The pan is provided with annular ribs by which the mercury is prevented from coming in contact with the sides of the pan, so that electrolysis occurs only between the mercury and the cathode, the pan serving as a conductor. Means are provided for supplying mercury and the electrolyte to the pan and for gauging the amount of each; and a stirring device having a variable speed drive located above the hood is disposed just above the level of the mercury. The metal cathode is provided with openings through which the gases produced pass into the hood, whence the lighter and heavier constituents escape by upper and lower outlets.—W. E. F. P.

Electrolytic cells [for oxidising mercury]. H. W. Matheson and F. T. Kaelin, Shawinigan Falls, Canada. Eng. Pat. 137,609, 23.1.19. (Appl. 1732/19.)

THE cell consists of a metal receptacle lined with concrete or the like and provided with an anode buried in the lining, and a cathode supported by the lining out of contact with the anode. The cathode is disposed horizontally, and comprises a hub, with radial supports projecting therefrom, and inclined plates secured between the supports in spaced overlapping relation. Means are provided for raising and lowering the cathode. The receptacle is provided with an aperture in the bottom through which the lining and anode project, and an electric conductor is clamped on to the anode out of contact with the shell. A stirring device is provided between the anode and cathode, with means for rotating it at different speeds. The stirring device may pass through the cathode, and a centre pin on the anode engages the stirrer and holds it out of contact with the cathode.

—B. N.

Electrolytic apparatus [for production of oxygen and hydrogen]. E. O. Benjamin, Newark, N.J., U.S.A. Eng. Pat. 137,553, 1.8.18. (Appl. 12,567/18.) Int. Conv., 1.8.17.

A THIN cast-iron frame constitutes the top, bottom, and sides of the cell, and two plates of conducting material applied to two opposite faces of the frame constitute the end walls, which are connected to a source of current to form the working electrodes. The frame is insulated from the walls, and the cell is divided into compartments by a porous diaphragm. Passages for separately collecting and leading off oxygen and hydrogen from the separate compartments, and passages for supplying electrolyte to each compartment, are formed in or upon the insulated frame, and may be moulded therein. The frame is provided at the top with a cavity or cavities into which the electrolyte extends, so that the level thereof is maintained above the uppermost portion of the diaphragm, and above the uppermost portion of the inner surfaces of the walls constituting the electrodes. Water-sealed exits are provided for the gases collected within the chambers, and a gas vent is connected with the exits below the level of the electrolyte in the water-seals.—B. N.

[Electric] battery. R. J. Crowley, Assignor to A. Kramer, T. R. and H. B. Tennant, and M. Rothman, Dallas, Tex. U.S. Pat. 1,306,214, 10.6.19. Appl., 1.5.17.

A PASTE composed of a salt of aluminium, *e.g.*, the sulphate, an oxide of lead, *e.g.*, litharge, and sulphuric acid, is used as the active material on the positive and negative plates of storage batteries. The paste is applied to grids of lead or aluminium or an aluminium alloy, and the plates are then "formed" by the action of an electric current, using sulphuric acid of sp. gr. 1.2 as electrolyte.

Electrolytic cell. A. M. Griffin, Kansas City, Mo. U.S. Pat. 1,327,091, 6.1.20. Appl., 21.8.18.

THE cell comprises a pair of flanged "pan-members," secured together in gas- and water-tight relation, and insulated from each other. A diaphragm permeable to water and electricity divides the cell into two compartments. A plate is arranged within each compartment, and is secured to the adjacent side wall of the cell, each plate being corrugated and in contact at the crests of the corrugations with the adjacent wall of the pan. Openings are provided between the corrugations, with walls projecting inwards flanking the openings.—B. N.

Electrolytic apparatus. E. O. Benjamin, Newark, N.J. U.S. Pat. 1,328,981, 27.1.20. Appl., 24.6.18.

AN electrode for electrolytic apparatus comprises a plate of conducting material having its surface formed or provided with triangular pyramid frustums vertically alined, so as to form upward channels between them and oblique channels between the upward channels.—B. N.

Electrolytic apparatus. V. M. Weaver, Harrisburg, Pa., Assignor to Weaver Co. U.S. Pat. 1,329,315, 27.1.20. Appl., 9.7.15.

AN apparatus, for treating a chloride, comprises a closed chamber adapted to contain an electrolyte, with a conducting interior bottom as one electrode and a conducting block depending from a sealed cover as the opposite electrode. Means are provided for mechanically feeding non-gaseous material into the electrolyte at a definite and controlled rate.

—B. N.

[Aluminium] plates for electrolytic cells; Preparation of —. J. Coulson, Wilkinsburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,330,581, 10.2.20. Appl., 7.10.15.

ALUMINIUM for use in electrolytic cells is immersed in hydrofluoric, hydrochloric, and nitric acids in succession, then cleansed, heated for several hours at a temperature not exceeding 600° C., and cooled.

—W. E. F. P.

Electric furnace; Three phase —. F. T. Snyder, Oak Park, Ill., Assignor to Industrial Electric Furnace Co., Chicago, Ill. U.S. Pat. 1,327,174, 6.1.20. Appl., 11.3.18.

AN electric furnace, having two upper electrodes and a bottom contact, is combined with three transformers, the secondaries of which are so connected as to produce a phase angle, between the voltages existing between the bottom contact and the upper electrodes, greater than 60° and less than 90°.

—B. N.

Electric precipitator. H. I. Frisbie, Anaconda, Mont. U.S. Pat. 1,329,237, 27.1.20. Appl., 6.1.19.

A PRECIPITATING chamber is provided with an inlet and outlet and with electrodes, suitably disposed across the chamber, forming collectively zones or fields of opposite polarity. The electrodes forming the zones of one polarity extend substantially the width of the precipitating chamber, whilst the electrodes forming the zones of opposite polarity are spaced a suitable distance from the walls of the chamber, and from the first-mentioned electrodes to prevent short-circuiting therewith, whereby a circuitous course is ensured for the gases with maximum precipitation.—B. N.

Electric insulating moulded articles; Process of manufacturing —. F. Grünwald, Pozsony, Hungary. U.S. Pat. 1,330,444, 10.2.20. Appl., 3.7.17.

A MIXTURE of inert substances, phenol-formaldehyde condensation products, and a liquid in which the condensation products are insoluble and which has a boiling point and vapour tension near to those of water, is moulded, and the moulded articles are hardened by rapid heating under atmospheric pressure.—W. E. F. P.

Manganese dioxide cells; Process for regeneration of depolarisation masses from exhausted —. G. Klopstsch, Berlin. Ger. Pat. 315,335, 8.7.17.

THE material is powdered and the salts contained therein are extracted by boiling with a suitable solvent, e.g., ammonium chloride. The washed

residue is re-oxidised by heating it under pressure with strong oxidising agents, such as hypochlorous acid or its salts or chloric acid. Oxidation may also be effected by electrolysis in the presence of any of the above compounds or of substances which, by electrolysis, give rise to hypochlorites (e.g., magnesium chloride) or to strongly oxidising compounds (e.g., bichromate, permanganate).—A. R. P.

Suspended material from gases. U.S. Pats. 1,329,737 and 1,329,817—8. See I.

Electrical treatment of gases. U.S. Pat. 1,329,859. See I.

Electrical purification of gases. Ger. Pat. 315,262. See I.

Electrochemical treatment of hydrocarbons. U.S. Pat. 1,327,023. See IIa.

Manganese dioxide. U.S. Pat. 1,330,738. See VII.

Electrodes. Eng. Pat. 138,272. See X.

Separation of ores. U.S. Pat. 1,329,127. See X.

XII.—FATS; OILS; WAXES.

Arachis oil; [Composition of the] fatty acids of —. A. Heiduschka and S. Felser. Z. Unters. Nahr. Genussm., 1919, 38, 241—265.

THE composition of the mixed fatty acids obtained from a sample of arachis oil (iodine value 86.1) was: —Arachidic acid, 2.3; lignoceric acid, 1.9; stearic acid, 4.5; palmitic acid, 4.0; oleic acid, 79.9; linolic acid, 7.4%. The oleic acid may have included a small quantity of hypogaeic acid.—W. P. S.

Electrolytes; Colloidal —. Soap solutions and their constitution. J. W. McBain and C. S. Salmon. Proc. Royal Soc., 1920, 97, A, 44—65. (See also this J., 1918, 249 t.)

N/1 SOLUTIONS of sodium and potassium soaps all contain colloid material, in the case of caproate 15% of colloid and in that of stearate 99%. The amount of colloid decreases rapidly with decrease in concentration and is inappreciable in N/5 laurate solutions. (See also J. Chem. Soc., Apr., 1920.)

—J. F. S.

Hydrogenated oils in manufacture of tin-plate. Collins and Clarke. See X.

PATENTS.

Oils and other liquids; Process and apparatus for deodorising, purifying, distilling, and vaporising —. J. T. Bateman, London. Eng. Pat. 138,388, 14.11.18. (Appl. 18,691/18.)

OIL, after passage through a preheater of the type described in Eng. Pat. 24,605 of 1914 (this J., 1915, 367), is led through a coil submerged in a heated metal bath, and then into an enclosed or sealed chamber in which it is brought in contact with superheated steam. The steam is led into the chamber by means of perforated tubes arranged on the bottom of the chamber, and the chamber may be divided into compartments by partitions, the oil flowing over and under alternate partitions, or the oil may be led through two or more separate chambers. The vapours from the chamber are led to the preliminary heating system, and the oil, after treatment, is conveyed away through cooling coils.—L. A. C.

Lanoline; Separation of — from crude wool-grease and the like. F. J. Metzger, New York. U.S. Pat. 1,330,210, 10.2.20. Appl., 7.6.16. Renewed 16.6.19.

CRUDE wool-grease is subjected to the action of a

solution of an alkali and an alkali salt, the alkali being present in sufficient quantity to saponify all the free fatty acid present.—B. V. S.

Oil and soap solution and process of making same. J. A. De Cew, Montreal, Canada. U.S. Pat. 1,330,173, 10.2.20. Appl., 25.2.18.

A STABLE emulsion of oil with about 25% of its weight of soap is prepared by mixing the ingredients while hot, discharging the hot mixture into hot water under pressure, and then discharging the product into cold water.—L. A. C.

Emulsifying oil solutions and the like; Method and apparatus for —. J. A. De Cew, Montreal, Canada. U.S. Pat. 1,330,174, 10.2.20. Appl., 17.5.18.

OIL or the like and water are heated in separate reservoirs and forced by pressure through pipes into a common chamber. The oil and water inlets into the chamber consist of unequally perforated plates, whereby the liquids are atomised, and, as they mix, form an emulsion, which is led away through another perforated plate having as many perforations as both the inlet plates, into a reservoir containing cold water.—L. A. C.

Sulphonic acids. U.S. Pat. 1,330,624. See IIa.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Coumarone resins and formolites. M. Ragg. *Farben-Zeit.*, 1919, 25, 16—18, 61—62, 105—106, 145—146. *Chem. Zentr.*, 1920, 91, II., 15.

COUMARONE resin should contain neither free nor combined sulphuric acid, as all sulphuric acid compounds rust iron. The resin is employed as varnish for lithographs, as well as in the manufacture of lacquers and paints; it also finds employment as a substitute for colophony in the manufacture of explosives. When used for sizing paper, it must be emulsified with animal glue or resin soap. The presence of added impurities is detected by the formation of a precipitate on the addition of sulphuric acid to the resin in benzol. Formolites (phenol-formaldehyde resins) may be divided into four classes: Insoluble in the usual solvents and infusible, *e.g.*, bakelites; soluble in certain solvents, but giving a lacquer which must be heated to be durable, *e.g.*, bakelite A and B, resinite; soluble in alcohol, and giving a usable varnish without heating, *e.g.*, issolin; soluble also in fatty oils and other solvents, *e.g.*, albertole. The necessary heating of the varnishes prepared from these condensation products somewhat limits their application; they can be employed as stove lacquers or for insulation, but their price prohibits their use on a larger scale. Issolin is recommended as a substitute for spirit-soluble hard resin, shellac, or Manila copal, and in alcoholic solution is employed for insulating armatures, electro-magnets, and coils.—L. A. C.

Coumarone-resin from low-temperature tar. Gluud and Breuer. See III.

Turbidimeter for varnishes. Sheppard. See XXIII.

PATENTS.

Antimony colours [for rubber]. E. F. Morris, Roby. Eng. Pat. 138,137, 8.10.18. (Appl. 16,335/18.)

FOR the preparation of antimony colours which do not blacken during the normal vulcanisation process, solutions of antimony are treated first with alkali-waste liquor (and/or a similar solution

obtained by boiling a mixture of lime, sulphur, and water), to which a soluble thiosulphate may be added, and then with a suitable excess of sulphuric acid (or nitre-cake) or hydrochloric acid, whereby sulphur is precipitated with the antimony sulphide.—W. E. F. P.

Resinous condensation products of phenol and formaldehyde; Production of —. Chem. Fabr. K. Albert, and L. Berend, Amöneburg. Ger. Pat. 301,381, 15.10.13. Addition to 301,374.

FORMALDEHYDE or a substance which liberates it is treated with mixtures of *m*-cresol or its homologues with other phenols in presence or absence of condensing agents, but only for such time and in such proportions that practically only the meta-substituted phenols react with the formaldehyde. The other phenols are then removed by distillation. The process can be carried out with a cresol mixture which has been specially freed from *o*-cresol, *e.g.*, by fractional distillation, the quantity of formaldehyde taken corresponding with the proportion of *m*-cresol in the mixture. Further purification of the still fusible resin is effected by distillation *in vacuo*. Alternatively, the condensation may be effected with a proportion of formaldehyde adjusted to the quantity of *m*-cresol and the removal of *o*- and *p*-cresol effected subsequently, *e.g.*, by sulphonation of the *o*-compound and separation of the *p*-compound by known methods. To avoid too rapid condensation, which leads to the immediate production of infusible products, the mixtures condensed should not be too rich in *m*-cresol, and slightly less formaldehyde than that theoretically required can be taken. A suitable cresol mixture is one containing 50—60% of the *m*-compound.—H. J. H.

Drier. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 305,692, 2.9.17.

THE drier consists of metallic compounds of 1,3-diketones or 1,3-ketocarboxylic acids; for example, manganese acetoacetate, cobalt acetylpinacolin, lead acetoacetanilide, or similar metallic compounds of acetylacetone, oxaloacetic acid, oxaloacetic esters, di- or tri-benzoylmethane. The products can be used alone or mixed with each other or with other known driers.—A. R. P.

Resin or varnish; Process for making a —. Vereinigte Chem. Werke A.-G., K. Lüdecke, und L. Mamlock, Charlottenburg. Ger. Pat. 307,622, 30.9.17.

FURFURAL is treated with a mineral acid and a metal (preferably zinc, but not an alkali metal) that evolves hydrogen from acids. The product differs from furfuryl alcohol made by reduction of furfural with sodium amalgam and acetic acid; it is insoluble in water, but soluble in alcohol and chloroform, and can be used in the varnish industry mixed with other substances in a suitable solvent.—A. R. P.

Varnish; Preparation of a — from indene. M. Claasz, Munich. Ger. Pat. 310,783, 19.2.18.

INDENE is condensed with formaldehyde in the presence of mineral acids, whereby a colourless balsam is obtained which is liquid when warm and gummy when cold, and which dries on exposure to the air.—A. R. P.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber obtained by coagulation of latex; Resistance of —. V. Henri, Caoutchouc et Gutta-Percha, 1920, 17, 10196—10202.

By coagulating latex with sulphuric acid in the presence of phenol it is possible to obtain sheets of

raw rubber of surprising strength and elasticity, the breaking strength being 190 grms. per sq. mm. and the permanent set small. After heating for 20 minutes at 100° C. the extensibility and permanent set are greater but the breaking strength is unaffected; heating for 4 hours at 110° C., however, causes the rubber to become very weak and plastic. If strips of the rubber are vulcanised in a sulphur bath at 135°–140° C., a very elastic product is obtained with a breaking strength of 666 grms. per sq. mm.—D. F. T.

PATENTS.

Heat-resisting [rubber] packing. J. Stromeyer, Philadelphia, Pa. U.S. Pat. 1,330,148, 10.2.20. Appl., 15.3.17.

THE material consists of finely-divided magnesite incorporated in a vulcanised rubber compound.
—B. M. V.

Antimony colours. Eng. Pat. 138,137. See XIII.

XV.—LEATHER; BONE; HORN; GLUE.

Tannins. III. Chlorogenic acid, the tannin-like constituent of coffee. K. Freudenberg. Ber., 1920, 53, 232–239.

ACCORDING to Gorter (see this J., 1908, 242, 464; 1910, 587; 1911, 235) chlorogenic acid is formed by the loss of a molecule of water from two molecules of hemichlorogenic acid. This view is not readily reconciled with its fission by penicillium and mucor varieties or, as in now shown, by tannase into caffeic and quinic acids. Further, chlorogenic acid, which Gorter considered to be anhydrous, contains 0.5 H₂O, thus accounting for the difference between it and the hypothetical hemichlorogenic acid. Chlorogenic acid is simply a depside of caffeic and quinic acids, the carboxy group of the former being united to one of the hydroxy groups of the latter as indicated by the formula,

$C_6H_3(OH)_2 \cdot CH : CH \cdot CO \cdot O \cdot C_6H_3(OH)_2 \cdot CO_2H$.
(See further J. Chem. Soc., Apr., 1920.)—H. W.

Casein in glue making; Influence of the method of manufacture on the use of —. S. Buttermann. J. Ind. Eng. Chem., 1920, 12, 141–144.

To obtain good results in the manufacture of water-resistant glue from casein (see this J., 1919, 732A), variations in the proportions of the ingredients are necessary in using caseins of different origin. Analyses of a large number of samples of casein manufactured in various ways showed that the ash content was the chief variant, and experiments were carried out to determine the effect of the ash content on the amount of water required to make casein glues of similar consistency. 100–200 grms. of dry, powdered casein was mixed with x parts of water, and after soaking for a few minutes a quantity of a suspension of lime (one part of hydrated lime to six of water) was stirred in to give 15% of lime on the weight of casein. A quantity of sodium silicate of sp. gr. 1.4, equal to 0.7 of the weight of casein, was then added and the mixture stirred vigorously. It was found that the ash content plotted against the ratio of the total water used to the casein gave a graph represented by an equation of the type, $y = mx + c$. It is thus possible to plot a graph for any casein glue formula from which the necessary variation in water content can be determined according to the ash content of the casein to be used. The author describes briefly various methods used in the manufacture of casein.—S. S. A.

Gelatin; Liquefaction of — by salts. T. R. Briggs and E. M. C. Hieber. J. Phys. Chem., 1920, 24, 74–75.

SOLUTIONS of 5% gelatin in 33% potassium chloride

solution set in 30 mins. at 19° C.; in similar solutions of magnesium chloride or ammonium chloride the solutions set in 24 hours, but in solutions of zinc chloride, potassium thiocyanate, potassium nitrate, or ammonium nitrate they do not set in 36 hours. The salt may be removed from the last group by dialysis and the gelatin then sets. The process of liquefaction and gelatinisation by salts is strictly reversible.—J. F. S.

Electrolytes; Penetration of — into gels. I. Penetration of sodium chloride into gels of agar-agar containing silver nitrate. W. Stiles. Biochem. J., 1920, 14, 58–72.

THE rate of penetration of sodium chloride into agar gels containing silver nitrate is proportional to the square root of the time; it is dependent on the initial concentration of the sodium chloride, and may be expressed by the formula $P/\sqrt{t} = k \log c + k'$, where P is the penetration, t the time, c the initial concentration of the sodium chloride, k a constant depending on the nature of the penetrating salt, and k' a constant depending on the concentration of the silver nitrate in the gel. (See also J. Chem. Soc., Apr., 1920.)—J. F. S.

Turbidimeter for gelatin. Sheppard. See XXIII.

PATENTS.

Tanning material [from sulphite-cellulose waste liquors]. W. Mensing, Freiberg. Ger. Pat. 304,349, 15.2.16.

A TANNING bath consists of sulphite-cellulose waste liquor which has been heated with solid or liquid oxidising agents, such as chlorates, nitrates, permanganates, or chromates.—L. A. C.

Tanning animal skins; Process for —. Deutsche Koloniale Gerh- und Farbstoff-Ges.m.b.H., Karlsruhe. Ger. Pat. 305,516, 25.12.15.

ANIMAL hides are steeped several times alternately in separate baths of a 2–5% formaldehyde solution and a solution of an aromatic compound which is either soluble in water or forms soluble salts and forms a compound with formaldehyde insoluble in water, e.g., a 1–2% solution of α -naphthylamine hydrochloride, or a 2% solution of resorcinol. The resorcinol bath yields a pliable leather of good tensile strength.—L. A. C.

XVI.—SOILS; FERTILISERS.

Plants; Action of chlorine and different vapours on —. P. Guérin and C. Lormand. Comptes rend., 1920, 170, 401–403.

MOST plants maintained for one hour, sometimes two hours, in an atmosphere containing 1 part in 2000 of chlorine, palite (chloromethyl chloroformate), bromoacetone, chloropicrin, or mustard gas (yperite, dichlorethyl sulphide) resist the action of these gases. The plants lose their leaves, but new ones appear after a more or less prolonged interval, and in the end the plant resumes its normal vegetation. The leaves die by reason of plasmolysis, the phenomenon being most rapid with chlorine and very slow with mustard gas under the experimental conditions.—W. G.

Calcium; Absorption of — by the roots of plants and its antitoxic properties with respect to copper. L. Maquenne and E. Demoussy. Comptes rend., 1920, 170, 420–425.

THE antitoxic action of calcium with respect to copper is not due to its preventing the penetration of this latter metal, since, even when present in excess, calcium does not hinder the absorption of copper by the roots of plants, and its diffusion into their aerial organs. Similarly the presence of copper

does not prevent the assimilation of calcium, so that the poisonous effect of copper is not due to its depriving the young plant of one of its essential nutrients. The antitoxic action of calcium is rather physiological in favouring the growth of the plant, thus increasing the volume into which the copper diffuses and hence, within certain limits, preventing any dangerous accumulation of this latter metal.—W. G.

Calcium cyanamide; Improvement of crude — [for use as fertiliser]. J. Baumann. Chem.-Zeit., 1920, 44, 158–159.

CRUDE calcium cyanamide has a number of disadvantages as a nitrogenous fertiliser, and to obtain the nitrogen in a more suitable form the author suggests a combination of the cyanamide and ammonia-soda processes. It is proposed that the ammonia for the process should be obtained from calcium cyanamide, and that the liquors from the sodium bicarbonate, which contain, after boiling, 135 grms. of ammonium chloride and 124 grms. of sodium chloride per litre, should be evaporated to crystallise out ammonium chloride. The ammonia is thus recovered as chloride for use as a fertiliser, and the lime which would have been used for regeneration of the ammonia is used for the manufacture of cyanamide. Alternatively, the ammonia could be regenerated in the usual way with lime, and the calcium chloride-sodium chloride liquors then treated successively with ammonia from calcium cyanamide and with carbon dioxide, whereby calcium carbonate would be precipitated and ammonium chloride could be crystallised from the solution. It is calculated that by the adoption of the proposed combination of the two processes considerable economies would be effected. Ammonium chloride has the advantage that it can be used for mixed fertilisers. Mixed with superphosphate, it does not affect the solubility of the latter nor cause it to become lumpy. Ammonium bicarbonate, which is quite as good as ammonium sulphate as a fertiliser, could also be prepared simply and cheaply from calcium cyanamide, but it cannot be mixed with superphosphate.—E. H. R.

PATENTS.

Fertiliser. L. T. Buck, Weems, Va. U.S. Pat. 1,330,369, 10.2.20. Appl., 30.9.19.

A FERTILISER is prepared by adding to fish sufficient quicklime to cook and desiccate it thoroughly.

—W. E. F. P.

Product from ammonia. Eng. Pat. 131,870. See VII.

XVII.—SUGARS; STARCHES; GUMS.

Sugars; Oxidation of — by mercuric acetate in presence of ammonia. T. Ingvaldsen and L. Bauman. J. Biol. Chem., 1920, 41, 147–148.

DEXTROSE and galactose are oxidised to gluconic and galactonic acids respectively by this method, the ammonium salts being isolated in 50% yield. Maltose and lactose are also oxidised, but the corresponding acids have not been isolated.—J. C. D.

PATENTS.

Decolorising-carbons; Process for the regeneration of —. G. Blardone, New Orleans, La. U.S. Pat. 1,327,222, 6.1.20. Appl., 9.7.17.

DECOLORISING and deodorising carbon is revived by washing with sodium bisulphite.—J. F. B.

Adhesive paste; Preparation of an —. Veredelungsges. für Nahrungs- und Futtermittel m.b.H., Bremen. Ger. Pat. 315,536, 28.3.18.

STRAW is boiled with caustic soda, with or without

pressure, and the solution neutralised with a mineral or organic acid, especially formic or acetic acid, or with carbon dioxide in excess. The resulting precipitate is preserved in a half-dried state and is suitable for use as an adhesive with fabrics or paper. Salts that produce an insoluble base with the soda, such as aluminium sulphate or formate, calcium or magnesium chloride, may be used for neutralising, in which case the precipitated hydroxide remains mixed with the precipitated paste.—A. R. P.

XVIII.—FERMENTATION INDUSTRIES.

[Barley;] **Further experiments on titration in stages** [applied to —]. A. Reichard. Z. ges. Brauw., 1919, 42, 223–227. (See also this J., 1917, 399; 1918, 523 A.)

A SAMPLE of barley of pure strain (originally propagated from a single plant) was divided by screening into two fractions containing the small and large corns respectively, and the acidities of the two fractions were compared by titration in stages (*loc. cit.*), followed by formol titration (*cp. this J.*, 1920, 202 A). The titrations were carried out on extracts made directly from the ground material, and also on extracts made after destruction of the enzymes (*cp. this J.*, 1918, 523 A). All the acidity values found, referred to a given weight of dry substance, were higher for the small than for the large corns. The difference was most marked for the acidity representing substances acid to phenolphthalein but not to litmus. As these substances consist largely of amino-acids, and the latter are required by the barley embryo for synthetic processes during germination, the relatively large amounts of them present in small corns must tend to accelerate the early stages of germination. This would account for the rapid heating which has been observed in the case of small-corned barleys on the malting floors (*cp. Delbrück, this J.*, 1902, 556).—J. H. L.

Amolytic action of the diastase of *Aspergillus oryzae*; Method of testing the —. S. A. Waksman. J. Amer. Chem. Soc., 1920, 42, 293–299.

IN connection with the removal of size from textile goods, the following is a convenient method of measuring the starch-liquefying power of enzymes. A suitable amount of the enzyme solution is added to 10 c.c. of a 2% paste of raw potato starch which is maintained at 40° C., and the time required (from 1 to 15 mins., according to the quantity of enzyme) for the liquid to become clear is observed. The determination of the end-point may be facilitated by staining the starch, preferably with Neutral Red. When the stage of hydrolysis corresponding with this end-point is tested with iodine, it is found to be identical with that at which no intense blue colour is obtained, but only a deep-brown to faintly violet-brown colour or faint blue-violet colour, showing that all the starch paste has been transformed into dextrins. Comparing the starch-liquefying powers of malt enzymes and those obtained from *Aspergillus oryzae* (water extract known commercially as Polyzyme), the first are found to give a higher Lintner value, while the second give a higher liquefying value. The ratio of the latter to the former in the case of malt preparations is 1:4 to 1:5, while in the case of *Aspergillus oryzae* enzyme it is 1:1 to 1:5:1.—H. W.

Water; Analysis of brewing —. H. Lüers. Z. ges. Brauw., 1919, 42, 209–210, 219–221, 227–228, 210–211, 252–254, 263–265.

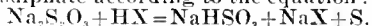
THE author gives working details of a set of known

volumetric methods by which the analysis of waters may be carried out rapidly and accurately. Since the salts present in waters are usually ionised to the extent of 70–80%, he recommends that analytical results should be stated in terms of ions, expressed as millival per litre, the millival being 1/1000 of the grm.-equivalent weight of an ion. This method has been adopted officially in Germany in returning the composition of mineral and potable waters.

—J. H. L.

Fermentation; Chemistry of alcoholic —. E. Zerner. Ber., 1920, 53, 325–334.

An account is given of experiments made in Austria-Hungary on the production of glycerol by the Connstein-Lüdecke process (this J., 1919, 691 A); some of the conclusions are at variance with those arrived at by Neuberg and Reinfurth (this J., 1920, 76 A). Equivalent amounts of acetaldehyde and glycerol are produced in the process, and, in addition, alcohol and carbon dioxide are formed, these being the only substances obtained in appreciable amount. Increase in the amount of sodium sulphite increases the yield of glycerol and aldehyde, but more than 38% of glycerol cannot be obtained. Sodium sulphite may be replaced by sodium hydro-sulphite. Failure to produce glycerol in quantitative amount by sufficiently increasing the relative proportion of sulphite has been attributed by Neuberg to dissociation of the aldehyde-bisulphite compound; this conclusion appears to be unfounded, since with moderate addition of sulphite the equivalent amount of glycerol is almost obtained, whilst with very large additions (which should have the effect of suppressing the dissociation of the aldehyde-bisulphite compound) the yield of glycerol sinks to about two-thirds of that theoretically possible. The effect seems rather to be attributable to the fact that union between aldehyde and bisulphite is not an instantaneous process, and that a certain amount of opportunity is thus offered for further degradation of the aldehyde and glycerol to alcohol and carbon dioxide. Neuberg has shown that pyruvic acid can be fermented in the presence of sulphite, but in his experiments he only used one-eighth to one-quarter of the equivalent amount of the sulphite; the use of larger proportions shows that the bisulphite compound of pyruvic acid is not fermentable, and that neither pyruvic nor glyceric acid can constitute the intermediate stage in the production of aldehyde during alcoholic fermentation. When sugar is fermented in the presence of sodium thiosulphate, sulphur is soon precipitated, hydrogen sulphide is evolved and, after a time, fermentation ceases; the yield of glycerol, reckoned on the sugar actually fermented, is very considerable. When the experiments are repeated with the addition of sodium sulphite or sodium bicarbonate, reaction proceeds as if the thiosulphate were not present. It appears, therefore, that an acid is produced during fermentation which decomposes the thiosulphate according to the equation:—



The sulphur is transformed into hydrogen sulphide by the reducing action of the yeast, whilst the sodium bisulphite combines with the aldehyde and thus provides the possibility of a good yield of glycerol. Fermentation is stopped owing to the production of organic sulphur derivatives which poison the yeast. In the presence of sulphite or carbonate the acid is neutralised by these, and the thiosulphate remains undecomposed. Generally it may be stated that glycerol and acetaldehyde are intermediate products of alcoholic fermentation which are simultaneously formed and, under normal circumstances, simultaneously disappear; as a preliminary phase an acid must be formed which, however, is not identical with pyruvic acid. During normal fermentation (without sulphite) the maximum amount of aldehyde appears to be present in the middle period of action.—H. W.

Acetaldehyde; Detection of — in alcoholic fermentation. C. Neuberg. Woch. Brau., 1919, 36, 292. J. Inst. Brew., 1920, 26, 99–100.

For the detection of acetaldehyde in fermenting liquids, 3 c.c. of the liquid is treated, without filtration, with 0.5 c.c. of 4% sodium nitroprusside solution and 2–3 c.c. of 3% piperidine solution. In presence of acetaldehyde, e.g., when fermentation is conducted in presence of calcium sulphite (see Neuberg and Reinfurth, this J., 1920, 76 A) a deep-blue coloration is produced, whilst no coloration appears in liquids fermented under normal conditions, i.e., in absence of sulphites.—J. H. L.

Sugar-beet; Fermentation experiments with —. W. Hartmann. Z. Unters. Nahr. Genussm., 1919, 38, 287–290.

BETROOT slices were heated at 70° C. with water, or treated with steam for some hours; the liquid portion was then drawn off and submitted to fermentation. A beverage was obtained which resembled beer but had a distinct, though not unpleasant, taste of beetroot. This taste was less in the beverage from the steamed beetroot and was masked to a great extent by the addition of hop extract; it is probable that treatment with steam at a high temperature would remove the beetroot taste entirely.—W. P. S.

Oxalic acid; Formation of — by *Aspergillus niger*. H. Raistrick and A. B. Clark. Biochem. J., 1919, 13, 329–344.

The production of oxalic acid by *Aspergillus niger* when grown in artificial culture media containing the salts of various organic acids as the sole source of carbon has been studied. Good growth of the fungus and good yields of oxalic acid were obtained with salts of succinic, fumaric, malic, tartaric, and acetic acids. With monobasic C_4 acids, C_5 acids, and glycollic and glyoxylic acids there was practically no production of oxalic acid, although in some cases growth was satisfactory. These results are discussed, and it is considered that the first step in the breakdown of sugar to oxalic acid is β - δ -diketoadipic acid. This undergoes hydrolysis into acetic and oxalacetic acids, the latter on further hydrolysis giving oxalic acid and acetic acid. Both molecules of acetic acid are then oxidised to oxalic acid.—J. C. D.

PATENTS.

Beer and low alcoholic malt beverages; Process of brewing —. C. A. Nowak, St. Louis, Mo. U.S. Pat. 1,328,888, 27.1.20. Appl., 13.2.18.

ORTHOPHOSPHORIC acid is added to the wort, in presence of yeast, to provide nutriment for the latter and to decompose carbonates present in the wort, and after fermentation metaphosphoric acid is added to precipitate proteins, which are subsequently removed.—J. H. L.

Acetone and butyl alcohol; Fermentation process for the production of —. C. Weizmann and G. A. Hamlyn, London. U.S. Pat. 1,329,214, 27.1.20. Appl., 27.3.18.

A STERILISED mash of amylaceous material is subjected to the action of moulds possessing strong proteolytic power, and fermented by bacteria of the *Amylobacter* group.—J. H. L.

XIXA.—FOODS.

Gluten; Determination of wet — in low-grade flours. J. Bouyer. Bull. Soc. Pharm. Bordeaux, 1919, No. 2. Ann. Chim. Analyt., 1920, 2, 52–54.

In the analysis of low-grade flours and mixed flours it is sometimes impossible to obtain a coherent cake of gluten; disintegration during the washing process may, however, be prevented by immersing the

cake for a few seconds in ether. The washing and kneading are then continued, with further treatment with ether, if necessary.—W. P. S.

Caseinogen; Composition of —, and method for preparing esters of amino-acids. F. W. Foreman. *Biochem. J.*, 1919, 13, 378—387.

THE amino-acids obtained by the acid hydrolysis of a protein may be converted into the ethyl esters by the action of gaseous hydrogen chloride and alcohol on the dry lead salts. The process is superior to that usually employed in that there is much less loss during the manipulations which the mixture of esters undergo prior to fractional distillation. The amino-acid content of caseinogen is considered, and it is shown that some 97.36% of the molecule is now accounted for. (See further *J. Chem. Soc., Apr.*, 1920.)—J. C. D.

Cocoa [cacao] butter; Digestibility of —. J. A. Gardner and F. W. Fox. *Biochem J.*, 1919, 13, 368—377.

CACAO butter is rather less digestible than butter, and beyond a slight laxative action no undesirable physiological effects were observed to follow the administration of considerable quantities of the fat. —J. C. D.

Beans; Hydrocyanic acid content of Phaseolus lunatus —. H. Lüthrig. *Chem.-Zeit.*, 1920, 44, 166—167.

FOR the determination of hydrocyanic acid in cyanogenetic beans it is recommended that 50 grms. of the beans be steeped in water for about 18 hrs., then ground to a thin paste, acidified with tartaric acid, and steam distilled in an atmosphere of carbon dioxide; the distillate, amounting to about 400 c.c., is collected in a receiver containing dilute silver nitrate solution. The precipitate formed consists of silver cyanide, silver sulphide, etc. Ammonia is added and the mixture warmed until the silver cyanide has dissolved, the solution filtered, and the filtrate acidified slightly with nitric acid. After 18 hrs. the silver cyanide is collected, washed, dried, ignited, and the resulting metallic silver weighed. Nine samples of *Phaseolus lunatus* beans examined yielded from 5.1 to 23.4 mgrms. of hydrocyanic acid per 100 grms. Treatment with emulsin previous to distillation did not appreciably increase the yield of hydrocyanic acid. When the beans had been washed and boiled (cooked) the consumption of three quantities of 200 grms. each did not produce any poisonous symptoms. (See also Guignard, this J., 1916, 1174.)—W. P. S.

Cyanogenesis in Sudan grass. Modification of the Francis-Connell method of determining hydrocyanic acid. P. Menaul and C. T. Dowell. *J. Agric. Res.*, 1920, 18, 447—450.

THE amount of hydrocyanic acid in Sudan grass has been determined. The colorimetric method of Francis and Connell (*J. Amer. Chem. Soc.*, 1913, 35, 1624) for the determination of hydrocyanic acid has been improved, as follows: The distillate containing the hydrocyanic acid is evaporated with 1 c.c. of concentrated yellow ammonium sulphide at 170° C. The residue is heated to 130° C., dissolved in 10 c.c. of warm water acidified with hydrochloric acid, and calcium chloride solution added, drop by drop, till the sulphide ceases to form; then 10% ferric chloride solution is added until the red colour produced is permanent. The solution is filtered and 5 c.c. of ferric chloride solution added to the filtrate. By heating at 130° C. colloidal sulphur is prevented from passing into solution again, and the second addition of ferric chloride produces the maximum intensity of colour. A standard solution

of hydrocyanic acid is prepared from a solution of potassium cyanide in a similar manner, and the two solutions are compared in a Bock-Benedict colorimeter (this J., 1918, 608 A).—J. H. J.

Vitamines; Fat-soluble and water-soluble — present in green plant tissues. H. Steenbock and E. G. Gross. *J. Biol. Chem.*, 1920, 41, 149—162.

CLOVER, spinach, alfalfa (lucerne), lettuce, cabbage, and chard contain fat-soluble A. Lettuce and cabbage are not as rich in this factor as the other plants, which is in accordance with the theory that this accessory factor is associated with certain yellow plant pigments. The water-soluble vitamin is present in alfalfa, clover, and cabbage in approximately the same amount.—J. C. D.

Fat-soluble vitamin; Thermostability of — in plant materials. H. Steenbock and P. W. Boutwell. *J. Biol. Chem.*, 1920, 41, 163—171.

HEATING yellow maize, chard, carrots, sweet potatoes, or squash for three hours in an autoclave at 15 lb. pressure caused no appreciable destruction of the fat-soluble factor. The process of ensilage was also without deleterious action. It is concluded that in plant tissues the factor is comparatively thermostable.—J. C. D.

Maize silage; Role of pentose-fermenting bacteria in production of —. W. H. Peterson and E. B. Fred. *J. Biol. Chem.*, 1920, 41, 181—186.

PENTOSE-FERMENTING bacteria are capable of bringing about decided changes in raw or in sterilised maize tissue. In sterilised silage these organisms develop rapidly and produce acetic acid, lactic acid, ethyl alcohol, and carbon dioxide.—J. C. D.

Chlorine; Estimation of — in feeds, faeces, and urine. J. O. Halverson and E. B. Wells. *J. Biol. Chem.*, 1920, 41, 205—208.

THE official method of the Association of Official Agricultural Chemists (U.S.A.) is modified in that the complete washing out of the silver chloride precipitate is eliminated. Instead, an aliquot portion of the direct filtrate is titrated with ammonium thiocyanate. The solutions employed are 0.05N in strength.—J. C. D.

Chlorogenic acid. Freudenberg. See XV.

PATENTS.

Drying and thickening liquids [e.g. milk] by means of compressed air and heated air or gases. L. Galland, Berlin-Wilmersdorf. Ger. Pat. 310,192, 21.10.17.

THE liquid is sprayed in either a vertical, horizontal, or sloping direction towards a vertical current of compressed air, care being taken to prevent it entering the nozzle of the air pipe. The process is specially applicable to the drying of milk, blood, emulsions, etc.—W. J. W.

Milk and cream; Method of ascertaining the quantity of fat in —. H. M. Høyberg, Frederiksborg, Denmark. U.S. Pat. 1,329,183, 27.1.20. Appl., 2.1.19.

A MIXTURE of 97 parts by volume of the milk, 3.4 parts of an aqueous solution of potassium sodium tartrate and sodium hydroxide, and 0.6 part of isobutyl alcohol, contained in a butyrometer glass, is kept for 15—20 mins. in a water bath at 60°—70° C., and the quantity of fat is then read off directly.—J. H. L.

XIXB.—WATER PURIFICATION; SANITATION.

Water-softening process: Lime-soda —. M. R. Herrle and F. M. Gleeson. Chem. and Met. Eng., 1920, 22, 269—272.

THE effect of insufficient, correct, and excessive additions of lime and soda ash have been experimentally determined. A water of 20° hardness (c.c. standard soap solution) was reduced to 7° hardness by the addition of lime alone, and to 11° hardness by soda alone. Correct additions of lime and soda together reduced the hardness to a minimum value of 0.5°.—C. A. K.

Rivers and streams; Self-purification of —. A. E. Cooper, E. A. Cooper, and J. A. Heward. Biochem. J., 1919, 13, 345—367.

FULLY aerated sewage effluents and polluted river waters may contain considerably less dissolved oxygen than distilled water under the same experimental conditions. The low results appear to be due to various factors, and cannot be prevented by any method so far employed. As, however, the oxygen content is still the same after five days' incubation and subsequent aeration, there seems to be no appreciable error in Winkler's dissolved oxygen absorption test as modified by Rideal and Stewart (this J., 1901, 841). The oxidation of the constituents of sewage proceeds much more slowly at 10° C. than at the standard temperature of 18.3°, and it is important to adhere to a standard temperature for the test. Dilution appears to have comparatively little influence on the rate of oxidation of constituents of sewage, but the rate may be very different in waters of different geological sources. Mud from unpolluted rivers contains a considerable amount of oxidisable matter, and may have a de-oxygenating effect upon the water. In the standardisation of sewage effluents the river-water into which the effluent is actually discharged must be used for dilution in Winkler's dissolved oxygen absorption test. The oxidation by permanganate, or Tidy test, does not appear to be affected by the presence of water from different geological sources.—J. C. D.

Cresols and cresol soap substitutes; Disinfectant properties of —. E. Hailer. Arbb. Reichs-Gesundh.-Amt, 1919, 51, 556—576. Chem. Zentr., 1920, 91, II., 111.

THE disinfectant properties of cresol are diminished in alkaline solution. Such solutions are ineffective with *Staphylococcus*, but on the other hand they are active to typhus bacilli, and with *B. coli* they are not inferior to cresol soap solution. The alkaline cresol solutions are therefore suitable for use in hospitals etc.; they also have the advantage of cheapness. A mixture of equal parts of 25% sodium hydroxide and cresol diluted with 20 times its volume of water gives the best results.—W. J. W.

Poisoning by sodium fluoride. Vallée. See VII.

Brewing water. Lüers. See XVIII.

Dichloroethyl sulphide. Yablick and others. See XX.

PATENT.

Refuse destructor furnaces. A. Brechot, Paris. Eng. Pat. 133,297, 10.9.19. (Appl. 22,274/19.) Int. Conv., 30.9.18.

MATERIAL to be treated is delivered into the combustion chamber on to a bridge, whereon it is dried by hot gases from the furnace which pass above and below. It is delivered from the bridge on to the grate. The walls of the furnace immediately above the grate are inclined outwards from above down-

wards to facilitate discharge of the clinker and slag, and a conduit is formed in the walls through which the air supply for the furnace is passed to preheat it. The air passes from this conduit into the hollow grate, which is formed in two sections adapted to rock on horizontal trunnions. The air is admitted to the charge from the hollow grate through perforations, which are made tapering in cross-section. When the burning of the charge is complete the two halves of the grate are opened downwards simultaneously by means of a lever. The ash-pit is connected by a pipe to the combustion space of the furnace so that hot gas from the clinker and slag passes into the furnace. The furnace may be used for the combustion of low-grade fuel such as turf and the like.—W. F. F.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Digitalis substances. H. Kiliani. Ber., 1920, 53, 240—250. (See also this J., 1919, 477 A.)

AN account is given of some of the properties and reactions of digitaligenin, digitogenic acid, and digitoxigenin. (See J. Chem. Soc., Apr., 1920.) —H. W.

Glycogen; Colorimetric determination of —. R. Thieulin. J. Pharm. Chim., 1920, 21, 91—93.

THE method depends on the red-brown coloration produced when glycogen is treated with iodine; the intensity of the coloration is proportional to the amount of glycogen present. Two c.c. of the neutralised glycogen solution, containing from 0.05 to 0.2% of the substance, is treated with 2 drops of iodine solution (iodine, 1, potassium iodide, 2 grms., water, 20 c.c.), and the coloration is compared with that of standards containing known amounts of glycogen. It is important that 1 drop of iodine solution be used per c.c. of test solution or standard solution.—W. P. S.

Caryacrol; Fusion of sodium p-cymenesulphonate with sodium hydroxide for the production of —. H. D. Gibbs and M. Phillips. J. Ind. Eng. Chem., 1920, 12, 145—149.

BY fusing a mixture of sodium p-cymenesulphonate and sodium hydroxide in the theoretical proportions (1:2 mols.) at various temperatures for different periods, it was found that the best temperature and time are 350° C. and 6 hours respectively, a yield of 74.1% of the theoretical being obtained under these conditions. Trials with varying amounts of sodium hydroxide showed a maximum yield with a proportion of 2.2 mols. per 1 mol. of the sulphonate. When water is added to a melt to obtain a more fluid fusion the time must be increased, and higher temperatures may be employed without deleterious effect. The results demonstrate the advantages of conducting the fusion in an autoclave as compared with the use of an open pot.—S. S. A.

p-Dimethylaminobenzaldehyde; Preparation of —. T. Ingvaldsen and L. Bauman. J. Biol. Chem., 1920, 41, 145—146.

SLIGHT modifications of the process described by Ullmann and Frey (Ber., 1904, 37, 855) are used. —J. C. D.

Urea; Schiff's reaction for the detection of —. D. Ganassini. Boll. Chim. Farm., 1920, 59, 3—5.

SCHIFF'S colour reaction for the detection of urea by means of furfural (Gazz. Chim. Ital., 1877, 7, 348) is given only if the latter contains acetone as impurity. The reaction is given also by allantoin, biuret, and urethane, the colour tending to violet

with the last compound. It is rendered very sensitive as follows: A drop of the liquid to be tested is placed on a microscope slide together with a drop of concentrated nitric acid; in presence of urea, colourless overlapping rhombic or hexagonal crystals of the nitrate separate. When exposed to the vapours from a mixture of equal parts of furiurid and acetone the crystalline residue, if it is urea nitrate, becomes coloured more or less intense reddish-violet, while under the microscope the crystals are seen to be undergoing solution and to be surrounded by a pink halo.—T. H. P.

Glyoxal; Preparation of — by the action of ozone on acetylene. A. Wohl and K. Bräunig. *Chem.-Zeit.*, 1920, 44, 157.

INTERACTION of ozone and acetylene proceeds smoothly when the gases are suitably diluted with air and a certain amount of water is present, a mixture of glyoxal and formic acid being formed. By spraying water into the reaction vessel an aqueous solution is obtained containing 1.5 to 2.0 of glyoxal, and stronger concentrations can be obtained by using this solution for spraying instead of water. By converting the glyoxal into its bisulphite compound and acting on this with aniline, the anilide of anilidoacetic acid is obtained which can be used for the preparation of indigo, avoiding the acetic and chloroacetic acid stages. Alternatively, the glyoxal may be converted into glycollic acid or glycol. The original glyoxal solution contains nitric acid, formed by the action of ozone on the air, and this must be neutralised with calcium carbonate before the solution is concentrated by evaporation.—E. H. R.

Dichloroethyl sulphide [mustard gas]; Determination of — in air. M. Yablick, G. St. J. Perrott, and N. H. Furman. *J. Amer. Chem. Soc.*, 1920, 42, 266—274.

A SOLUTION of selenious acid in sulphuric acid (1:1) is reduced by traces of dichloroethyl sulphide giving an orange-red suspension of selenium; the presence of about 0.005 mgrm. of dichloroethyl sulphide can be detected in this manner. The estimation of the vapour in air is effected by bubbling the mixture through a suitable absorbent, such as a 1% solution of selenium dioxide in water, and adding 10 c.c. of a solution of selenious acid in sulphuric acid of such concentration that a 1% solution of selenious acid in 1:1 sulphuric acid is finally obtained. The solution is then heated for 10 minutes at 85° C. ($\pm 5^\circ$ C.), cooled, and the strength estimated by nephelometric comparison with standard solutions prepared from weighed quantities of the reagents. Amounts of substance between 0.1 mgrm. and 0.01 mgrm. can be determined with a maximum error of 0.005 mgrm. The selenious reagent is not specific for mustard gas; arsine and the substituted arsines and several other toxic gases react with it vigorously. (See further, *J. Chem. Soc.*, Apr., 1920.)

—H. W.

Hexamethylenetetramine; Process for making —. W. Herzog. *Z. angew. Chem.*, 1920, 33, 48.

COMMERCIAL ammonium carbonate is dissolved in 40% formaldehyde solution, the reaction taking place readily without application of heat and with brisk evolution of carbon dioxide. The solution is evaporated to dryness on a water bath under reduced pressure, and the residue is sublimed *in vacuo* or recrystallised from absolute alcohol. To compensate for its variable composition the amount of ammonium carbonate taken should be about 10% in excess of the theoretical quantity. The yield of hexamethylenetetramine is about 66% of the theoretical amount.—W. J. W.

Acetoacetic acid and its esters; Formation of β -methylumbelliferone as a reaction of —. V. Arreguine and E. D. Garcia. *Ann. Chim. Analyt.*, 1920, 2, 36—41.

WHEN 2 c.c. of concentrated hydrochloric acid containing a small quantity of acetoacetic acid is boiled for a few mins. with the addition of 0.1 grm. of resorcinol, condensation occurs with the formation of β -methylumbelliferone; the latter, after the mixture has been cooled and diluted, yields an intense blue fluorescence on the addition of a slight excess of ammonia. The reaction is not given by acetone or by β -hydroxybutyric acid; it will detect as little as 0.00001 grm. of acetoacetic acid in 5 c.c. of urine.—W. P. S.

Organic chemical reagents. V. Preparation of alkyl and alkylene bromides. O. Kamm and C. S. Marvel. *J. Amer. Chem. Soc.*, 1920, 42, 299—309.

IN the preparation of *n*-butyl bromide from the corresponding alcohol by means of hydrobromic acid, addition of sulphuric acid to the reaction mixture is of greater value than an increase in the quantity of hydrobromic acid. The Norris-Bodroux method (this *J.*, 1915, 217; 1916, 652) for preparing primary alkyl bromides has been modified in the following manner. The alcohol is treated with a 25% excess of hydrobromic acid together with some concentrated sulphuric acid. The mixture is heated under a reflux condenser, and the bromide is subsequently removed by direct distillation. The procedure is varied somewhat according to the physical and chemical properties of the alcohol used or of the bromide formed. For example, in the preparation of ethyl and allyl bromides the mixture is not heated under a reflux condenser because of the volatility of the former compound and the chemical activity of the latter; in the preparation of isoamyl bromide a somewhat smaller proportion of sulphuric acid is used in order to avoid charring; halides of high molecular weight, because of their high boiling points, are separated from the reaction mixture mechanically instead of by distillation. Detailed directions are given for the preparation of the following compounds, the corresponding yields being enclosed within brackets; *n*-butyl bromide, b.pt. 101°—104° C. (95—97%); isoamyl bromide, b.pt. 116°—120° C. (88—90%); trimethylene bromide, b.pt. 162°—165° C. (88—95%); trimethylene chlorobromide, b.pt. 142°—145° C. (89%); ethyl bromide, b.pt. 38°—40° C. (90—95%); allyl bromide, b.pt. 69°—72° C. (92—96%); *n*-octyl bromide, b.pt. 196°—200° C. (91%); lauryl bromide, b.pt. 175°—180° C. at 45 mm. (91%). The hydrobromic acid is prepared by the reduction of bromine by sulphur dioxide (Bodroux, this *J.*, 1915, 217); the mixture may be utilised directly without preliminary distillation, the necessary additional quantity of sulphuric acid being added as concentrated acid. Mixtures of constant-boiling hydrochloric and hydrobromic acids (such as are obtained when alkyl bromides are used with aluminium chloride in Friedel-Crafts syntheses) may be readily separated by fractional distillation.—H. W.

Organic chemical reagents. VI. Reagents from n-butyl alcohol. R. Adams and C. S. Marvel. *J. Amer. Chem. Soc.*, 1920, 42, 310—320.

n-BUTYL alcohol may be used for preparing *n*-amyl derivatives and *n*-caproic acid and its derivatives. Detailed instructions are given for the preparation of the following substances, the yields being placed within brackets. *n*-Butyl bromide (preceding abstract); *n*-butyl cyanide, b.pt. 138°—141° C. (75—80%); *n*-valeric acid, b.pt. 183°—186° C. (81%); ethyl *n*-valerate, b.pt. 142°—146° C. (85—90%); *n*-amylamine, b.pt. 102°—105° C. (57—68%); *n*-amyl alcohol (56—61%); ethyl *n*-butylmalonate, b.pt. 141°—145° C. at 40 mm., 235°—

240° C. at atmospheric pressure (89–92%); *n*-caproic acid, b.pt. 200°–205° C. (74%); α -bromocaproic acid, b.pt. 148°–153° C. at 30 mm. (71%); α -aminocaproic acid (65%).—H. W.

Ammonium tetroxalate, a product of the decomposition of isouanyl nitrite. H. Sandqvist and E. Mohlin. Ber., 1920, 53, 171–173.

A CRYSTALLINE deposit, needles or rods, m.pt. 129.5°–130.5° C. (decomp.), from the sides of a bottle containing commercial amyl nitrite which had become extensively decomposed, was found to consist of ammonium tetroxalate. Crystals formed under similar circumstances have been described by Böttger (Bull. Soc. Chim., 1915, [4], 17, 371), who, however, overlooked the nitrogen content and erroneously considered them to be methanetetra-carboxylic acid.—H. W.

Acetone; Analysis of — by Messinger's method. L. F. Goodwin. J. Amer. Chem. Soc., 1920, 42, 39–45.

MESSINGER's method for the determination of acetone (this J., 1889, 138) under proper conditions gives accurate and concordant results. Allowing the solutions to stand, the method of shaking and adding the iodine solution, an excess of acid, and dilution have no effect on the results. If methyl and ethyl alcohols are present they use up a certain amount of iodine.—J. F. S.

Ethylene; Nitration of —. H. Wieland and E. Sakellarios. Ber., 1920, 53, 201–210.

THE action of a nitrating acid consisting of 1 part by weight of nitric acid of sp. gr. 1.4 and 2 parts of sulphuric acid containing 20% SO_3 on ethylene at 0° C. leads to the production of glycol dinitrate, b.pt. 105.5° C. at 19 mm., sp. gr. 1.433 at 17° C., and β -nitroethyl nitrate, colourless poisonous oil, b.pt. 120°–122° C. at 17 mm., sp. gr. 1.468 at 18° C. β -Nitroethyl alcohol prepared by the action of silver nitrate on ethyleneiodhydrin is contaminated with glycol mononitrate and glycol. The purified alcohol is a colourless, odourless liquid, b. pt. 103° C. at 11.5 mm., sp. gr. 1.309 at 13.3° C. (See further, J. Chem. Soc., Apr., 1920).—H. W.

Mercury in organic combination; Method of determining — by means of zinc filings. M. François. J. Pharm. Chim., 1920, 21, 85–91.

THE method, as described previously (this J., 1913, 445, 609 A) may be applied to organic mercury compounds. The substance is dissolved by heating with alcohol, and the solution then treated with hydrochloric acid and zinc filings under the conditions given; the addition of potassium iodide is omitted. With compounds which are insoluble in alcohol the reduction with zinc may be carried out in sodium hydroxide solution to which potassium iodide is added; residual zinc is then dissolved in hydrochloric acid.—W. P. S.

Orolic acid. Raistrick and Clark. See XVIII.

Esters of amino-acids. Foreman. See XIXA.

PATENTS.

Tobacco; Treatment of inferior —. S. Hagino, Tokyo, Japan. Eng. Pat. 137,683, 24.3.19. (Appl. 7335/19.)

THE quality of inferior tobacco is improved by moistening the leaves with a mixture of the following liquids: an extract made by boiling resin with sodium chloride solution, an extract made by boiling pine bark with sodium bicarbonate solution, and a solution of an organic salt of iron (prepared, for example, by digesting iron oxide in vinegar).

—J. H. L.

Acetic anhydride; Manufacture of —. F. P. Leach, and The United Alkali Co., Ltd., Liverpool. Eng. Pat. 137,701, 4.4.19. (Appl. 8552/19.)

ACETIC anhydride is produced in a continuous process by the interaction of anhydrous sodium acetate and carbonyl chloride, according to the equation, $2\text{CH}_3\text{CO}_2\text{Na} + \text{COCl}_2 = (\text{CH}_3\text{CO})_2\text{O} + \text{CO}_2 + 2\text{NaCl}$. The reaction is started by suspending sodium acetate in acetic anhydride and adding alternately, with cooling, carbonyl chloride and sodium acetate, keeping the latter in slight excess until the desired quantities have been mixed. A portion of the mixture is then run off into a still and the acetic anhydride distilled off in a vacuum, whilst fresh quantities of sodium acetate and carbonyl chloride are added to the mixture remaining in the reaction vessel.—G. F. M.

Iodine dusting powder and process for making same. N. Sulzberger, New York. U.S. Pat. 1,329,148, 27.1.20. Appl., 14.10.15.

AN antiseptic dusting powder is made by treating boric acid with a solution of iodine in a suitable solvent and then evaporating the solvent.—J. H. L.

Aromatic amines; Production of —. E. C. R. Marks, London. From E. I. du Pont de Nemours and Co., Wilmington, Del., U.S.A. Eng. Pat. 138,372, 18.7.17. (Appl. 10,374/17.)

UNSUBSTITUTED poly-arylamines are formed by heating at about 300° C. unsubstituted monoaryl amines in the presence of 0.5–3% of a catalyst containing a halogen, such as an ammonium halide, ferrous chloride, magnesium bromide, chloro- or bromo-benzene, aniline hydrochloride, etc. The reaction is facilitated by the presence of not more than 2% of water. For example, aniline is heated with 1% of ferric chloride for 48 hours at 300° C. in an autoclave provided with a vertical column having a release valve at its apex, through which the ammonia is removed from time to time. The reaction product is fractionated, the lower fractions being returned to the autoclave for redigestion, whilst the fraction boiling above 300° C. consists mainly of diphenylamine, and a tarry residue remains behind in the still.—G. F. M.

Mono- and di- β -hydroxyethylaminobenzoic esters; Manufacture of —. Soc. Chim. des Usines du Rhône, Paris. Eng. Pat. 128,552, 30.5.19. (Appl. 13,760/19.) Int. Conv., 20.6.18.

MONO- and di- β -hydroxyethylaminobenzoic esters are obtained by heating to temperatures varying from 50° to 110° C. a mixture of ethylene oxide (1–2 mols.) and an aminobenzoic ester (1 mol.). The reaction can be accelerated by the addition of small quantities of water or ethyl alcohol. Thus ethyl β -hydroxyethyl-*p*-aminobenzoate, $\text{OH}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{NHC}_6\text{H}_4\cdot\text{CO}_2\text{C}_2\text{H}_5$, a colourless, crystalline substance, m.pt. 63° C., is produced by heating for several hours in a closed vessel at 50° C. equimolecular proportions of ethylene oxide and ethyl *p*-aminobenzoate; whilst if under similar conditions 2 mols. of ethylene oxide are used together with $\frac{1}{2}$ mol. of water and 1 mol. of ethyl alcohol, ethyl di- β -hydroxyethyl-*p*-aminobenzoate is formed. After crystallisation from benzene it forms white lamellae, m.pt. 94° C.—G. F. M.

Bromoacylized urea compounds; Manufacture of derivatives of —. Farbenfabr. vorm. F. Bayer und Co., Leverkusen, Germany. Eng. Pat., 132,795, 16.9.19. (Appl. 22,766/19.) Int. Conv., 9.8.17.

ON treating bromoacylized urea compounds with acylising agents in presence of condensing agents such as zinc chloride or sulphuric acid, acyl compounds are obtained which have a rapid but lasting sedative and soporific action. Examples are given

of the preparation of acetyl bromodiethylacetylurea, m. pt. 108° — 109° C., from bromodiethylacetylurea (174 parts), acetic anhydride (1000 parts), and zinc chloride (75 parts), the mixture being heated for one hour at 60° C. and then poured into iced water (3000 parts); of acetyl bromoisovalerylurea, m. pt. 108° — 109° C., from bromoisovalerylurea and acetic anhydride; of propionyl bromodiethylacetylurea, m. pt. 103° C., and of benzoyl bromodiethylacetylurea, m. pt. 139° — 140° C., sulphuric acid being employed as condensing agent in each of the latter three examples.—G. F. M.

Dihydroxy - diamino - arseno - benzol - dihydrochloride; Sodium compound of —. C. Suzuki, Tokyo, Japan. U.S. Pat. 1,330,288, 10.2.20. Appl., 7.6.16.

A SOLUTION of sodium alcoholate (ethoxide) is mixed with a solution of dihydroxydiaminoarsenobenzene dihydrochloride, and the resulting sodium compound precipitated by the addition of ether.

—B. V. S.

Saponin or similar substances; Production of — from vegetable extracts rich in sugar or colouring matters. Billwärders Seifen- u. Glycerinfabr. W. Krauss and W. H. Hofmann, Hamburg. Ger. Pat. 314,877, 23.5.18.

AQUEOUS or alcoholic vegetable extracts are treated at 60° C. with aqueous solutions of alkali or alkaline-earth hydroxides until the reagent is in slight excess, and then a mineral acid or an acid salt is added to cause precipitation. The precipitate can be purified by treatment with reducing bleaching agents. The effect of the preliminary treatment with alkali is to double the yield of precipitate, which is deposited as a fine flocculent mass. The precipitate contains saponin in a fairly pure form, and a higher degree of purity can be attained by repeating the solution in alkali and re-precipitation with acid. Decolorisation of the first precipitate can be effected with reducing bleaching agents, e.g., hydrosulphites, sulphurous acid, or sulphites, followed by acidification with mineral acids. The slightly soluble saponin becomes soluble if just sufficient calcined soda is added to cause the dissolved mixture to react slightly alkaline.—H. J. H.

Acetic acid; Concentration of solutions of —. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 314,943, 26.8.16.

ACETIC acid containing bisulphate in solution is extracted with ether. The equilibrium concentration of acetic acid in ether when in contact with the bisulphate solution is almost double that attained in the absence of bisulphate, while only traces of ether are dissolved in the bisulphate solution. The extraction process is also accelerated. On distilling the ether off, a residue of 70—80% acetic acid is obtained from which glacial acetic acid can be fractionated, the fore-runings being returned to the extraction process.—H. J. H.

Lanolin. U.S. Pat. 1,330,210. See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Penetration of electrolytes into gels. Stiles. See XV.

Turbidimeter for gelatin etc. Sheppard. See XXIII.

PATENTS.

Non-inflammable films or filaments for kinematographic and other industrial purposes; Manufacture of —. W. J. Stevenson, London. Eng. Pat. 138,379, 21.8.18. (Appl. 13,569/18.)

A NON-INFLAMMABLE, impermeable, durable, flexible, and transparent film is obtained from a solution

of cellulose acetate, preferably in tetrachloroethane or benzyl alcohol, containing also triacetin and triphenyl phosphate; the amounts of the two latter are about equal and may vary from 10% to 30% of the weight of the cellulose acetate.—B. V. S.

Coloured photographs or kinematograph films; Producing —. W. Finnigan, London, and R. A. Rodgers, Hove. Eng. Pat. 138,396, 7.1.19. (Appl. 120/19.)

A NEGATIVE is taken through a translucent colourless screen, such as ground glass, in contact with the sensitive film, and a print is obtained from this negative on a sensitised material provided with a coloured dotted screen film between the base and the sensitive film.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Nitrocellulose; Notes on the German stability tests and on a new hot storage test (75° C.) for —. F. Lenze and B. Pleus. Z. ges. Schiess- u. Sprengstoffw., 1919, 14, 297—299, 315—318. Chem. Zentr., 1920, 91, II., 107.

TESTS with samples of nitrocellulose from various sources and which had been subjected to different methods of purification proved that the manometric and nitric oxide methods did not give sufficient indication of the degree of stability. The use of a bath with a liquid of constant boiling point, such as carbon tetrachloride (75° C.), gives good results.—W. J. W.

Mercury fulminate; By-products in the preparation of —. A. Langhaus. Z. ges. Schiess- u. Sprengstoffw., 1919, 14, 300—304, 318—321, 334—337, 350—353. Chem. Zentr., 1920, 91, II., 106, 107.

THE CONDENSATION liquors contain approximately twenty times as much mercury as the mother liquor. The acidity varies inversely with the mercury content. Control tests of the amount of mercury are made by an electrolytic method; a platinum basin must be used. Mercury settles out from the condensation liquors after 24—48 hours' standing, and in the receivers after 1 day. In the quantitative estimation of the mercury as mercurous chloride hydrogen chloride must not be present in excess, and solution of the mercurous chloride depends on the nitric acid concentration. Mercury in the condensation liquors may also be readily titrated with sodium hypochlorite. The precipitation of chloride from the liquors and neutralisation of these may be carried out simultaneously in one vessel by using sodium chloride solution and soda instead of hydrogen chloride and lime, but the method is more expensive. With lime some lime is always precipitated, or a double compound of lime and mercurous oxide may be formed. Four-fifths of the liquors are volatile, and the residue is explosive. (See also this J., 1919, 442 A.)—W. J. W.

PATENTS.

Explosives. C. Mammelli and L. Bernardini, Rome. Eng. Pat. 138,371, 17.7.17. (Appl. 10,328/17.)

EXPLOSIVE products are obtained by melting together ammonium nitrate and guanidine nitrate or biguanide nitrate, or ammonium nitrate and dicyanodiamide, with or without the addition of nitroguanidine, in such proportions that the product melts below 130° C. Good explosive mixtures are obtained by melting, for example, ammonium nitrate 6—8, dicyanodiamide 0.6—0.8, and nitroguanidine 1.2—2.5 parts by weight. The mixture has high explosive power and great stability, is completely homogeneous, and may be safely handled.—T. St.

Colloid bodies [smokeless powders]; Preparation of —. W. O. Snelling, Allentown, Pa. U.S. Pat. 1,305,946, 3.6.19. (Appl., 14.8.17.

SMOKELESS powder is prepared by mixing nitro-starch with an agent, *e.g.*, liquid trinitrotoluene, which does not gelatinise the nitro-starch during the mixing process, and subsequently heating the mixture to 60°–90° C., to cause "colloidisation" (gelatinisation).

Explosive; Preparation of an —. R. Mewes, Berlin. Ger. Pat. 301,796, 26.2.16.

A COMPOUND of oxygen and nitrogen, with or without chlorine, and in either the liquid or solid state, is mixed with a non-combustible absorbent such as ferric chloride. This mixture is conveyed in suitable containers, in bulk or in cartridge form, to the place where it is to be employed, and a liquid combustible material is there added to it immediately before use.—W. J. W.

Hexanitrotriphenyl phosphate; Production of —. Imhansen und Co., and K. Lehmstadt, Witten. Ger. Pat. 302,501, 20.3.17.

TRIPHENYL phosphate is nitrated with mixed nitric and sulphuric acids or nitric and phosphoric acids. The former mixture at 50°–60° C. gives *p*-trinitrotriphenyl phosphate, and at 80° C. a good yield of the 2,4-hexanitrotriphenyl phosphate. The latter is not sensitive to shock and so is a serviceable explosive. More prolonged nitration at higher temperature gives picric acid sufficiently pure for use.—H. J. H.

Propellant charges containing ammonium nitrate. Verein. Köln-Rottweiler Pulverfabriken, Berlin. Ger. Pat. 303,979, 27.6.17.

To reduce the pressure necessary for forming charges of which ammonium nitrate is an ingredient, and also to avoid evolution of smoke on firing, a mixture of wood charcoal, 2 parts, and lampblack, 1 part, is added to the composition. The lampblack serves further to prevent decomposition of the pressed charges during drying and enables them to sustain a temperature of 35° C. without fracture. To prevent absorption of moisture wood tar may be added to the composition, or the charges may be encased in a protecting composition of nitroglycerin, nitrocellulose, and a eutectic solution of tri- and dinitrotoluenes.—W. J. W.

Explosives; Manufacture of — in a form in which they can be poured. Sprengstoff A.-G. Carbonit, Hamburg. Ger. Pat. 307,040, 4.8.17.

Two metallic nitrates are mixed together in non-molecular proportions and heated to a temperature well below the melting point of either of them. A halogen salt or urea may be added, as well as nitro compounds to increase the power of the explosive. If urea is added, one nitrate only may be used. Typical compositions are: (1) Ammonium nitrate, 90; sodium nitrate, 10 parts. (2) Ammonium nitrate, 90; sodium nitrate, 5; and sodium chloride, 5 parts. (3) Ammonium nitrate, 37.5; urea, 6.25; sodium chloride, 6.25 parts.—W. J. W.

Nitrocellulose; Manufacture of —. Vereinigte Köln-Rottweiler Pulverfabriken, Berlin. Ger. Pat. 307,120, 25.4.18.

INSTEAD of washing the nitrocellulose by displacing the nitration acids with water, the nitrocellulose is caused to travel slowly through the layer of water. Risk of mixing acids and water is thus eliminated.—W. J. W.

Explosives; Manufacture of — from hexanitrodiphenylamine and trinitrotoluene. O. von Schroetter, Oberlössnitz-Radebeul. Ger. Pat. 315,306, 3.2.12.

HEXANITRODIPHENYLAMINE is added to molten trini-

tritoluene to form an explosive mixture, which, on account of its greater density, is more active and less liable to disintegration at higher temperatures than the product obtained by mixing the components and subsequent compression of the mixture, and is also superior in regard to susceptibility to shock or percussion.—L. A. C.

Explosive mixtures. Atlas Powder Co., Wilmington, Del., Assignees of R. L. Hill and A. J. Strane, Tamaqua, Pa., U.S.A. Eng. Pat. 131,524, 1.9.19. (Appl. 21,139/19.) Int. Conv., 1.11.18.

SEE U.S. Pat. 1,299,942 of 1919; this J., 1919, 480 A.

XXIII.—ANALYSIS.

Condensers [; Laboratory —]. F. Friedrichs. Z. angew. Chem., 1920, 33, 29–32.

THE various forms of laboratory condensers devised since the time of Liebig are discussed, and forty-five references to literature are given. The most efficient condenser appears to be the screw-form tube with five threads and internal cooling; this form has also advantages over the spiral form condenser when used for reflux purposes.—W. P. S.

Viscosity measurements. O. Faust. Z. physik. Chem., 1919, 93, 758–761.

VISCOSITY measurements, by the method of noting the time taken by an air bubble in rising through a measured vertical column of liquid, are best made with tubes 24–18 mm. diameter. In these tubes the time is independent of the size of the bubble and the diameter of the tube. (See also J. Chem. Soc., 1920, ii., 166.)—J. F. S.

Analysis; Method of stating the results of —. A. Thiel. Chem.-Zeit., 1920, 44, 31–32.

It is suggested that the results of analysis be expressed in equivalent-units per 100 grms. or 100 c.c. of the original substance.—W. P. S.

Turbidimeter for solutions of gelatin, cellulose, and varnishes. S. E. Sheppard. J. Ind. Eng. Chem., 1920, 12, 167–169.

FOR dealing with limited quantities of liquids an apparatus is described which operates with small constant thicknesses of liquid and comprises two superposed opaque line gratings arranged to rotate relatively to each other (Ives, Elect. World, 1910, 939; J. Opt. Soc. Amer., 1917, 100). Viewed by transmitted light, at such a distance that the grating lines are below the limit of resolution, parallel dark bands are seen. If a scattering, turbid medium be placed in front of the crossed gratings, the visibility limit can be measured by the angle through which the gratings must be turned to make the bands just disappear. Readings with an instrument constructed on this principle can be made as rapidly as with a polarimeter or refractometer, and the result can be expressed as percentage clarity referred to a given thickness of a standard medium, such as distilled water or lacquer solvent.—S. S. A.

Tin salts; Reaction of —. A. Mazuir. Ann. Chim. Analyt., 1920, 2, 9.

WHEN 2 c.c. of a neutral or alkaline solution of a stannic or stannous salt is mixed with 2 c.c. of 10% potassium iodide solution and 2 c.c. of concentrated sulphuric acid is added, a yellow crystalline precipitate of tin iodide is obtained. The precipitate is soluble in chloroform, alcohol, and dilute hydrochloric acid and is decomposed by ether with the liberation of iodine. Arsenic and antimony salts yield a similar reaction, but the arsenic iodide is quite insoluble in hydrochloric acid and the

antimony iodide forms a brick-red flocculent precipitate. Other metals of the antimony group do not interfere with the reaction.—W. P. S.

Lead and bismuth; Separation and determination of —. G. Luff. *Chem.-Zeit.*, 1920, 44, 71.

A nitric acid solution of the two metals is treated with ammonia, added drop by drop, until a slight turbidity forms; this is dissolved by the addition of a few drops of nitric acid, saturated ammonium nitrite solution and sodium nitrite solution are added, the mixture is diluted to about 200 c.c., and heated to boiling; when nitrogen ceases to be evolved, the precipitate of bismuth hydroxide and basic nitrate is collected on a filter, washed with hot water, then dissolved in nitric acid, the solution evaporated and the residue ignited and weighed as Bi_2O_3 . The ignited oxide is treated with nitric and hydrofluoric acids to remove silica, then washed to remove traces of alkali, and again weighed. The filtrate from the bismuth precipitate is acidified with acetic acid and the lead precipitated and weighed as chromate. With modification, the method may be used for the separation of bismuth and cadmium.—W. P. S.

Calcium and magnesium; Estimation of — in different saline media. E. Canals. *Bull. Soc. Chim.*, 1919, 25, 655–658. (See this J., 1919, 159 A, 797 A.)

For the volumetric estimation of small amounts of magnesium it is recommended that the magnesium should first be precipitated as magnesium ammonium phosphate and then one of the two following methods adopted. The precipitate is washed centrifugally with dilute ammonia solution and then dissolved in just sufficient 5% hydrochloric acid, the phosphoric acid in this solution being estimated by titration with an uranium solution (1 c.c. = 0.5 mgrm. P_2O_5), using cochineal as an indicator; or, the precipitate is washed with a saturated aqueous solution of sodium phosphate and then dissolved in hydrochloric acid, the ammonia present being estimated colorimetrically by means of Nessler's reagent. The results are accurate to 0.2 mgrm. of magnesium by the first and to 0.01 mgrm. by the second method.—W. G.

Manganese; Sensitive reaction for —. P. H. Hermans. *Pharm. Weekblad*, 1919, 56, 1344–1346.

CARON and RAQUET's reaction (this J., 1919, 574 A), in which a red coloration is produced by oxidising agents in manganese solutions after addition of potassium oxalate and acetic acid, was discovered independently by the author, and incidentally employed as a test for nitrogen tetroxide in presence of chlorine and bromine. It may also be used for the detection of nitrites. Suitable oxidising agents are potassium permanganate and bichromate, lead peroxide, nitrous acid, nitrogen tetroxide, etc. Some substances, although in themselves powerful oxidising agents, have a negative effect, but can be made effective by addition of osmic acid. Such are hydrogen peroxide, potassium persulphate, chlorine, bromine, iodine, chloric acid, iodic acid, and potassium ferricyanide. For the manganese test, lead peroxide is most suitable, as it is stable and gives no coloration alone. The test is sufficiently sensitive to indicate 0.07 mgrm. manganese, or 0.03 mgrm. sodium nitrate, in 1 c.c. of solution. The red coloration is due to formation of a complex alkali manganese oxalate.—W. J. W.

Cadmium vapour arc lamps. Bates. See IIa.

Phenol. Fischer and Breuer. See III.

Phenols. Fischer and Glund. See III.

Iron in iron ores. Brandt. See X.

Hydrogen electrode. Bailey. See XI.

Amylolytic action. Waksman. See XVIII.

Brewing water. Lüers. See XVIII.

Gluten in low-grade flour. Bouyer. See XIX A.

Hydrocyanic acid. Menaul and Dowell. See XIX A.

Glycogen. Thienlin. See XX.

Urea. Ganassini. See XX.

Dichlorethyl sulphide. Vablick and others. See XX.

Acetoacetic acid. Arreguine and Garcia. See XX.

Acetone. Goodwin. See XX.

Mercury in organic combination. François. See XX.

PATENTS.

Furnace-gases; Apparatus for ascertaining the composition of —. [Determination of carbon dioxide.] F. Cossor, London. Eng. Pat. 138,156, 23.1.19. (Appl. 1716/19.)

A MEASURING vessel of known capacity is connected to the furnace-flue by way of a hand-controlled stop-valve, and also to a closed vessel containing a solution of caustic potash through which the gas from the measuring vessel is caused to pass. A vertical tube, having a capacity fully equal to that of the measuring vessel, depends from the latter, and is connected by a U-tube to a reservoir of inert liquid (glycerin and water), the level of which coincides with the bottom of the depending tube; and an indiarubber pumping-ball is connected to the upper end of the reservoir through a tube provided with a venting plug and a non-return valve. The gas outlet of the absorption vessel communicates with a hand-controlled vent and with one limb of a manometer, the other limb of the latter being open, graduated, and extending at a slight upward inclination to the horizontal.—W. E. F. P.

Soluble gas constituents; Apparatus for determining —. F. W. Sperr, jun., Oakmont, Pa., and A. A. Kohn, Newark, N.J., Assignors to The Koppers Co., Pittsburgh, Pa. U.S. Pat. 1,316,602, 23.9.19. Appl. 3.12.18.

THE apparatus comprises two concentric chambers, connected in series and a coil in each chamber immersed in a gas-absorbing liquid therein, the length of the coil being greater than the depth of the liquid in which it is immersed. The gas under examination is passed through an injector jet into the lower end of one coil, and the injector action causes circulation of the absorbing liquid through the coil. The gas is afterwards passed in a similar manner through the other coil to remove the last traces of the soluble constituent.

Calculations depending on the relations between the pressures, densities, temperatures, and percentage compositions of gases; Instrument for facilitating — and recording of observations of these quantities. P. G. N. Ommaney, Westminster. Eng. Pat. 138,146, 7.1.19. (Appl. 484/19.)

Fat in milk. U.S. Pat. 1,329,183. See XIX A.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Aktieselskabet Myrens Verksted. Apparatus for drying solid moist substances. 8340. Mar. 20. (Norway, 7.2.18.)

Beeton, and Trufood, Ltd. Drying organic substances. 9044. Mar. 27.

Christie. Driers. 7966. Mar. 17.

Cloke and Smith. Separating and/or concentrating materials. 8591. Mar. 23.

Davies. Emulsifiers. 8954. Mar. 26. (U.S., 23.4.17.)

Ellerm (Ruzicka). Effecting reactions between gases and/or vapours. 8933. Mar. 26.

Fairweather (Aktiebolaget Kväveindustri). Heat treatment of masses or liquids by gases. 7978. Mar. 17.

Galvanizing Equipment Co., and Lane. Furnaces. 8059. Mar. 18.

Head. Roasting or calcining furnaces etc. 8901. Mar. 26.

Larsen. Burning watery materials in kilns. 8485. Mar. 22. (Denmark, 4.6.19.)

Martin and Richards. 8573. See X.

Merz. Evaporation of liquids with recovery of the vapours. 7778. Mar. 16.

Peters. Crystallising-pans. 8345. Mar. 20.

St. Barbe. Briquettes and process of making same. 7788. Mar. 16.

St. Barbe. Binding loose material for briquettes, road surfaces, etc. 7789. Mar. 16.

Sturgeon. Centrifugal separating-apparatus. 8091. Mar. 18.

Syndicaat Electro-Staal, and Vermaes. Rotary kilns. 7713. Mar. 15.

Waterhouse, and Waterhouse, Ltd. Manufacture of crucibles etc. 8046. Mar. 18.

Worthington Pump and Machinery Corporation. Filter press. 8798. Mar. 25. (U.S., 9.3.18.)

COMPLETE SPECIFICATIONS ACCEPTED.

18,471 (1914). Chantraine. Furnaces. Mar. 24.
18,473 (1914). Chantraine. Furnaces. Mar. 31.
10,455 (1918). Elektro-Osmose A.-G. Electro-osmotic process for removing water from materials. (135,817.) Mar. 24.

20,765 (1918). O'Brien. Continuous fractional distillation. (140,140.) Mar. 31.

21,927 (1918). Boland. Drying process and apparatus. (140,145.) Mar. 31.

4967 (1919). Lessing. Rings for filling absorption towers, distilling columns, etc. (139,880.) Mar. 24.

22,076 (1919). British Thomson-Houston Co. (General Electric Co.). Catalyzing material. (140,011.) Mar. 24.

367 (1920). Delaygne. Solution for absorption refrigeration processes. (140,356.) Mar. 31.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Bean. Method of producing gas. 8256. Mar. 19.

Carpenter. Purification of coal gas etc. 8699. Mar. 24.

Engelke. 9050. See III.

Field. Coal substitute. 7658. Mar. 15.

Franklin. Fuel. 8971. Mar. 26.

Good Inventions Co. Combustion of liquid etc. fuel. 8270. Mar. 19. (U.S., 6.6.16.)

Hart. Production of carbon from waste materials etc. 8718. Mar. 24.

Hislop. Fuel. 7783. Mar. 16.

International Coal Products Corp. Manufacture of briquettes. 8955. Mar. 26. (U.S., 18.2.18.)

Kormann. 7994. See XII.

Lund. Gas producers etc. 9010. Mar. 27.

Pfeifer. Production of gas etc. 8327. Mar. 20.

Poppe. 7998. See IX.

St. Barbe. 7788 and 7789. See I.

Smith, and Whitehall Petroleum Corp. Oil-distilling apparatus etc. 8104. Mar. 18.

Smith and Tulloch. Gas producers. 8593. Mar. 23.

Thompson. Furnaces for manufacture of gases etc. 8949. Mar. 26. (U.S., 1.6.18.)

Trotter. Production of gasoline. 7956. Mar. 17.

U.S. Industrial Alcohol Co. Liquid fuels. 8797. Mar. 25. (U.S., 12.10.17.)

Wade (Standard Oil Co.). Distilling petroleum. 8361. Mar. 20.

Williams. Manufacture of gas-purifying material. 8279. Mar. 19.

COMPLETE SPECIFICATIONS ACCEPTED.

987 (1918). Bouillon. Dehydrating peat. (140,112.) Mar. 31.

16,820 (1918). Soc. de Chimie et Catalyse Industrielles. Production of rich gases from petroleum residues. (140,128.) Mar. 31.

3916 (1919). Smith. Gasification of coal. (123,738.) Mar. 31.

12,923 (1919). Bildt. Producer-gas furnace. (139,957.) Mar. 24.

14,848 (1919). Steel. Manufacture of fuel from anthracite duff. (139,977.) Mar. 24.

26,395 (1919). Vickers and Dickson (Clayton). Composite fuel. (140,025.) Mar. 24.

III.—TAR AND TAR PRODUCTS.

APPLICATION.

Engelke. Treating hydrocarbon oils, tars, asphalts, etc. 9050. Mar. 27.

COMPLETE SPECIFICATIONS ACCEPTED.

15,440 (1919). Burt, Boulton, and Heywood, and Miles. Manufacture of carbazole. (139,981.) Mar. 24.

21,360 (1919). Morris and Co., and Morris. Sulphonation and chlorination of aromatic substances. (140,007.) Mar. 24.

IV.—COLOURING MATTERS AND DYES.

APPLICATION.

Ges. f. Chem. Ind. in Basel. Manufacture of substantive *o*-oxyazo dyestuffs, copper compounds thereof, and their application in dyeing. 8920. Mar. 26. (Switz., 1.5.19.)

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Bouillon and Worms. Apparatus for producing cellulose threads. 7634. Mar. 15.

British Cellulose and Chemical Manuf. Co., Briggs, Clotworthy, and Dreyfus. Manufacture of fibres and fabrics. 7929. Mar. 17.

British Cellulose and Chemical Manuf. Co., and Roy. Production of artificial filaments. 8880. Mar. 26.

Bronnert. Manufacture of viscose silk. 8000 and 8451. Mar. 17 and 22.

Fort, Lumsden, Mackenzie, and Robinson. Scouring vegetable yarns and fabrics. 8972. Mar. 26.

Goldberg. Protecting from fire inflammable substances. 7792. Mar. 16. (Ger., 27.6.19.)

Hart. Degumming ramie fibre. 8599. Mar. 23.

Langwell and others. 8115. See XVIII.

Minton. Drying paper etc. 8908—8911. Mar. 26.
 Paul. Recovery of grease from waste liquors from wool-scouring. 8766. Mar. 25.
 Soc. la Soie Artific. de la Voulte. Waterproofing and strengthening threads and fabrics. 9056. Mar. 27. (Fr., 11.2.20.)

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Fort, Lumsden, Mackenzie, and Robinson. Scouring and bleaching yarns etc. composed of vegetable fibres containing dyed effect threads or fibres. 8366. Mar. 20.
 Ges. f. Chem. Ind. in Basel. 8920. See IV.
 Tootal. Dyeing. 8391. Mar. 22.
 Vallays. Dyeing-vats. 7811. Mar. 16. (Fr., 21.3.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

7094 (1919). Saundevan, Ltd., and Evans. Fireproofing fabrics. (110,209.) Mar. 31.
 8360 (1919). Sharp. Means of fastening dyes in material, and waterproofing same. (110,222.) Mar. 31.
 14,599 (1919). Gaston and Rushton. Dye jigs. (139,971.) Mar. 21.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Enderli. Manufacture of formates. 7922. Mar. 17.
 Greatham. Extraction of potash from blast-furnace dust and recovery of metalliferous residue. 8380. Mar. 22.
 Nitrogen Corporation. Production of nitrogen compounds. 7665. Mar. 15. (U.S., 1.9.17.)
 Nitrogen Corporation. Production of ammonia. 7967. Mar. 15. (U.S., 17.3.19.)
 Poppe. 7998. See IX.
 Soc. Anon. l'Azote Français. Manufacture of ozone. 8471. Mar. 22. (Switz., 22.3.19.)
 Wilderman. Production of hypochlorites and chlorates. 7832. Mar. 16.

COMPLETE SPECIFICATIONS ACCEPTED.

7661 (1918). Hulin. Preparation of anhydrous metallic chlorides. (110,114.) Mar. 31.
 17,991 (1919). Barnes. Preparation of barium hydroxide from barium sulphide. (110,297.) Mar. 31.
 21,939 (1919). Barbe. See XVI.
 25,676 (1919). Salzwirk Heilbronn A.-G. Process for making sodium sulphate. (131,214.) Mar. 31.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Central School of Science and Technology, Imperial Trust, Mellor, and Moore. Manufacture of pottery etc. 8336. Mar. 20.
 Hancock. Manufacture of pottery and porcelain. 8747. Mar. 25.
 Marlow. Gas-fired ovens or kilns for tiles, pottery, etc. 8037. Mar. 18.
 Saavedra. Vitrefied product. 8183. Mar. 22.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Buckle. Manufacture of bricks. 8255. Mar. 19.
 Cochrane. Manufacture of cement from blast-furnace slag. 8761. Mar. 25.
 Fabriks Aktiebolaget Kronsten. Insulating and building material. 7901. Mar. 17. (Sweden, 19.3.19.)
 Kirkpatrick and Monnoyer. Manufacture of compositions for covering roads etc. 8001. Mar. 17.
 Poppe. Utilisation of combustion gases of brick etc. ovens, and manufacture of sulphurous products. 7998. Mar. 17.
 St. Barbe. 7789. See I.

St. Barbe. Manufacture of binders for road-making etc. materials. 7790. Mar. 16.
 Smith. Manufacture of concrete blocks, briquettes, etc. 7818. Mar. 16.

COMPLETE SPECIFICATIONS ACCEPTED.

16,530 (1914). Mellersh-Jackson (Krupp A.-G.). Manufacture of Portland cement. Mar. 31.
 14,789 (1919). Lambert. Hardening and preserving permeable soft woods. (139,974.) Mar. 21.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Annable. Extraction of copper from its ores. 7791. Mar. 16.
 Blair. Open-hearth furnaces. 7986 and 8098. Mar. 17 and 18. (U.S., 12.9.16 and 20.7.17.)
 Blanch. Manufacture of tungsten from its ores. 7618. Mar. 15.
 British Thomson-Houston Co. (General Electric Co.). Treatment of metals. 8213. Mar. 19.
 Champion. Cast-iron welding flux. 7657. Mar. 15.
 Clark, and Metallurgical Plant Construction Co. Heat treatment of metallic etc. objects. 8079. Mar. 18.
 Cochrane. 8761. See IX.
 Conroy, and Harper, Sons, and Bean. Furnace for tinning etc. 7771. Mar. 16.
 Duke. Alloys. 8701. Mar. 21.
 Edser and Wood. Concentration of ores by flotation. 8359. Mar. 20.
 Greatham. 8380. See VII.
 Hadfield. Manufacture of steel. 8103. Mar. 18.
 Marks (Scovill Manuf. Co.). Melting and reducing metals. 7663. Mar. 15.
 Martin and Richards. Treatment of pulverulent or granular material with gases, particularly roasting of ores etc. 8573. Mar. 23.
 Mathys (U.S. High Speed Steel and Tool Corp.). High-speed tool material. 8728. Mar. 24.
 Metallbank u. Metallurgische Ges. Bearing metals. 8727. Mar. 21. (Ger., 18.1.17.)
 Moeller and Schwere. Extraction of metals from complex ores. 8019. Mar. 18.
 National Malleable Castings Co. Steel. 8956. Mar. 26. (U.S., 21.5.15.)
 National Malleable Castings Co. Alloyed steels 8957. Mar. 26. (U.S., 21.5.15.)
 National Malleable Castings Co. Preparing iron for castings. 8962. Mar. 26. (U.S., 17.11.15.)
 Preston. Colouring metal. 7751. Mar. 16.
 Stock. Production of metals in electric furnace. 8719. Mar. 21.
 Sulman. Treatment of low-grade garnierite ores. 8268. Mar. 19.
 United Lead Co. Hard lead alloys. 8960. Mar. 26. (U.S., 2.10.18.)

COMPLETE SPECIFICATIONS ACCEPTED.

12,590 (1917). Krupp A.-G. Grusonwerk. Treatment of ores. (109,271.) Mar. 31.
 5811 (1918). Gontermann. Basic Bessemer process for producing steel. (115,027.) Mar. 31.
 2288 (1919). Boisselier. Solder for aluminium. (121,715.) Mar. 21.
 10,132 (1919). Cunningham, and Stein and Atkinson. Furnaces for heat treatment of metals. (110,238.) Mar. 31.
 13,597 and 17,785 (1919). Leathart, and Locke, Blackett, and Co. Refining lead. (140,278.) Mar. 31.
 14,111 (1919). Cunningham. Preparing aluminium or its alloys for electro-plating. (139,967.) Mar. 21.
 17,573 (1919). Wale and Wale. Composition for coating metals with tin or tin alloys. (110,294.) Mar. 31.
 23,746 (1919). Soc. des Alliages et Bronzes Forgeables. Tilting crucible or melting-pot furnaces. (110,015.) Mar. 21.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Beswick. Electric storage batteries. 8065. Mar. 18.

Marks (Scovill Manuf. Co.). Electric furnaces. 7664. Mar. 15.

Scovill Manuf. Co. Automatic control mechanism for electric furnaces. 8711. Mar. 24. (U.S., 26,618.)

Soc. Métallurgique du Frayol. Electric furnaces. 8444. Mar. 22. (Fr., 7,120.)

Stock. 8719. *See* X.

COMPLETE SPECIFICATIONS ACCEPTED.

10,455 (1918). Elektro-Osmose A.-G. *See* I.

14,106 (1918). Jungner. Primary galvanic batteries with unalterable electrolyte. (118,843.) Mar. 31.

20,375 (1918). Imbery. Electric muffle furnaces. (140,138.) Mar. 31.

21,831 (1918). Fenton. Electric furnaces. (139,843.) Mar. 24.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Arlt. Apparatus for separating oil or fat. 8805. Mar. 25.

Kormann. Treating oil. 7994. Mar. 17.

Paul. 8766. *See* V.

Quick. Treatment of animal and vegetable oils. 8354. Mar. 20.

Rankin and Self. Extraction of oils, fats, alkaloïds, etc., by volatile solvents. 7803. Mar. 16.

COMPLETE SPECIFICATION ACCEPTED.

6142 (1919). Naaml. Vennoots. A. Jurgens' Vereen. Fabr. Recovering oils and fats from soap-free waste water. (124,220.) Mar. 24.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATION.

British Thomson-Houston Co. (General Electric Co.). Protecting-compositions. 7944. Mar. 17.

COMPLETE SPECIFICATIONS ACCEPTED.

290 (1919). Flakes A/S., and Schou. Manufacture of painting and priming compositions. (140,147.) Mar. 31.

18,392 (1919). Barbe. Manufacture of a white pigment. (140,301.) Mar. 31.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Allenet et Cie. Vulcanising rubber. 8117. Mar. 18. (Fr., 15,120.)

Tudor (Tudor). Utilisation of waste rubber. 8810. Mar. 25.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Bray (Indian Wood Products Co.). Manufacture of catechin and catechu tannic acid. 8681. Mar. 24.

Knudsen. Tanning fish skins. 8416. Mar. 22.

Knudsen. Manufacture of fish glue and isinglass. 8417. Mar. 22.

Marris, and Walker and Sons. Process for tanning. 8861. Mar. 26.

Ross, and Walker and Sons. Epilation of skins. 8739. Mar. 25.

COMPLETE SPECIFICATION ACCEPTED.

7479 (1918). Weiss. Manufacture of horny masses from casein. (115,439.) Mar. 31.

XVI.—SOILS; FERTILISERS.

COMPLETE SPECIFICATIONS ACCEPTED.

18,536 (1919). Norsk Hydro-Elektrisk Kvaelfstof-aktieselskab. Fertilisers containing nitrogen and phosphoric acid. (132,496.) Mar. 24.

24,039 (1919). Barbe. Process for granulating cyanamide. (140,331.) Mar. 31.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATION.

Langwell, and Power Gas Corp. Fermentation of cellulose. 8115. Mar. 18.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Baker and Sons, Head, and Prescott. Treatment of food products etc. 7950. Mar. 17.

Becton, and Trufood, Ltd. 9044. *See* I.

Horlick. Food compound. 8310. Mar. 20. (U.S., 22,118.)

Marris, Ross, and Walker and Sons. Preserving animal and vegetable matter. 8860. Mar. 26.

Sherman. Preserving meat. 7960. Mar. 17.

Wood. Food compositions. 8229 and 8230. Mar. 19. (U.S., 19,319.)

COMPLETE SPECIFICATIONS ACCEPTED.

1083 (1919). Wanquier et Cie. Desiccation of potatoes and other vegetable substances. (122,422.) Mar. 31.

5028 (1919). Whyte and Fothergill. Apparatus for treating sewage and other effluents. (140,189.) Mar. 31.

6142 (1919). Naaml. Vennoots, Jurgens' Vereen. Fabr. *See* XII.

17,070 (1919). Chalas. Dry and soluble extract of raw meat. (129,639.) Mar. 31.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Bielouss. Treatment of aromatic materials. 8804. Mar. 25.

Commercial Research Co. Side-chain oxidation. 8208. Mar. 19. (U.S., 7,419.)

Imray (Monsanto Chemical Works). Production of saccharin. 8470. Mar. 22.

Lombaers. Extraction of caffeine from coffee beans. 8276. Mar. 19. (Holland, 13,619.)

Rankin and Self. 7803. *See* XII.

Soc. Anon. de Prod. Chimiques. Production of acetaldehyde from acetylene. 8648. Mar. 24. (Fr., 24,319.)

COMPLETE SPECIFICATION ACCEPTED.

9019 (1918). Pascal. Synthetic manufacture of ethyl alcohol and ethyl acetate. (140,115.) Mar. 31.

21,360 (1919). Morris and Co., and Morris. *See* III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATION.

Elliott. Photography. 8176. Mar. 19.

COMPLETE SPECIFICATIONS ACCEPTED.

4390 (1919). Smith. Screen plates and films for colour photography. (139,871.) Mar. 24.

28,247 (1919). Finnegan and Rodgers. Producing coloured photographs or cinematograph films. (140,349.) Mar. 31.

XXII.—EXPLOSIVES; MATCHES.

COMPLETE SPECIFICATION ACCEPTED.

26,827 (1919). Mellgren. Manufacture of igniting composition for matches. (140,026.) Mar. 24.

XXIII.—ANALYSIS.

COMPLETE SPECIFICATION ACCEPTED.

3171 (1919). Johnson and Brearley. Apparatus for testing the hardness of materials. (140,165.) Mar. 31.

I.—GENERAL; PLANT; MACHINERY.

Distillation and rectification. L. Gay. Chim. et Ind., 1920, 3, 157—166.

In a continuous column the part below the inlet for the liquid to be rectified serves for the volatilisation, and the part above for the rectification of the liquid. According to one method of working, in which each section of the lower part receives a supply of heat and the upper part is cooled throughout its entire length, the contact between the liquid and the vapour phases is reduced to a minimum. In the other method the whole of the heat is supplied to the lowest section, which is often a boiler, and the cooling is confined to the reflux condenser mounted on the top of the column; in this method the contact between the phases is at a maximum. It is shown that the second method is the more efficient, and a number of formulæ, tables, and curves are given from which it is deduced that in the second method the number of sections required in the lower or vaporising portion of the column is governed by the composition of the liquid to be rectified and is practically independent of the amount of heat supplied, whereas in the upper or rectification portion of the column the number of sections necessary does depend upon the amount of heat supplied to the bottom section, but has a minimum value which depends on the desired composition of the condensed liquid to be collected.

—W. H. C.

PATENTS.

Evaporation of water from aqueous liquids. A.-G. Kummel & Matter, Aarau, Switzerland. Eng. Pat. 123,716, 24.10.18. (Appl. 17,387/18.) Int. Conv., 26.2.18.

In the process in which the vapour given off from a liquid on evaporation is heated by compression and used to evaporate more of the same liquid, part of the compressed vapour is blown into the liquid by one or more open steam jets, while part passes through a coil or other known type of heat transmitter.—B. M. V.

Separating; Method of and apparatus for — or separating and recovering (and if desired washing) oil, grease, fat, or fatty matter or the like or other material from water or other liquids or for the separation of two liquids of different densities. L. Linden, London. Eng. Pat. 138,655, 19.7.18. (Appl. 7430/18.)

A SEPARATING chamber is divided into three compartments transversely to the direction of the flow of liquid. The first and third compartments are narrow and are formed by downward projections from the roof. The liquid enters at the liquid level at one end of the chamber, passes down the first compartment to the lower part of the chamber, and then horizontally across the chamber to the third compartment, where it passes upwards to the outlet at the same level as the inlet. Transverse baffle plates of short height prevent disturbance of the liquid in the middle compartment, whilst permitting light material to rise to the surface. This material is withdrawn through a pipe at the liquid level or an opening in the roof by raising the head of liquid. Fatty material may be first melted and washed by the injection of steam through pipes below the floating material. Below the level of the horizontal current of liquid are placed transverse baffle plates of short height for directing the heavy material, falling out from the current, into pockets formed in the bottom of the chamber. These pockets have exit valves below, and are provided with perforated pipes for the treatment of the deposited sludge before removal if desired.—J. H. J.

Mixing machines. J. R. Webb, Wolverhampton, and Ransome & Sons Machinery Co., Ltd., London. Eng. Pat. 139,018, 11.3.19. (Appl. 6096/19.)

A SHAFT revolves in a trough-like vessel and carries a number of discs or wheel-like mixing members inclined to the shaft and to each other. The trough-like container need have only one aperture for filling and emptying, being rotated through about 180° for the latter purpose.—B. M. V.

Condensing apparatus for refrigerating machines. L. Chew and W. F. Jennings, London. Eng. Pat. 139,361, 14.5.19. (Appl. 12,131/19.)

ONE or more vertical series of horizontal tubes is divided into groups by headers at the ends of the tubes, the groups decreasing in number (or size) of tubes from the bottom upwards, so that the velocity of the vapour, which passes upwards inside the tubes in zig-zag fashion and decreases in volume because of condensation, may remain constant. The tubes are cooled by a water spray outside, and their ends are reduced in diameter so that they may be fastened (e.g., by welding) into the headers and yet lie very close to each other in order to maintain a uniform film of cooling water. The interiors of the headers are provided with baffles sloping downwards to effect separation of the liquid and vapour.—B. M. V.

Drying cylinders; Steam inlet and water outlet of —. J. O. Lundberg, Borge, Norway. U.S. Pat. 1,331,994, 24.2.20. Appl., 24.6.19.

THE hollow trunnion of a drying cylinder is provided with a removable cap which does not rotate with the cylinder; the cap carries a steam inlet pipe, a water outlet pipe extending into and bending down to near the lowest point of the cylinder, and a window to permit observation of the water tube.—B. M. V.

Dryer. R. C. Newhouse, Wauwatosa, Assignor to Allis-Chalmers Manufacturing Co., Milwaukee, Wis. U.S. Pat. 1,332,137, 24.2.20. Appl., 26.6.18.

A CYLINDRICAL casing contains a number of tubes extending the length of the casing. The tubes have a segmental cross-section, and are so arranged that an annular chamber is formed between the casing and the tubes, and radial spaces separate the individual tubes. The annular chamber and the radial spaces are all in communication with one another.—L. A. C.

Dry-kiln. O. W. Degen, Alameda, and W. Degen, San Francisco, Cal. U.S. Pat. 1,332,164, 24.2.20. Appl., 9.8.18.

A CHAMBER containing the goods to be dried is connected with a furnace by a duct; the furnace is provided with a chimney and also with a sheet metal air-pipe leading from the outer air across the fire to a point in the duct leading to the dryer, so that clean warm air is drawn into the dryer. Dampers are arranged so that the products of combustion can be sent, as desired, up the chimney or into the dryer along with the clean air.—B. M. V.

Filter [; Ultra —]. J. F. Wait, Niagara Falls, - N.Y. U.S. Pat. 1,331,732, 24.2.20. Appl., 30.8.17.

AN ultra-filter is constructed of a colloidal membrane supported by a closely woven wire fabric with a smooth surface next the membrane. The wire fabric is backed by a plate with grooves for leading away the filtrate. The whole is fixed in an apparatus adapted to withstand high pressures, with charging and pressure connections on one side of the filter and discharge and negative pressure connections on the other side.—J. H. J.

Separator; Revolving ——. A. B. Carstens, Monterey, Mexico, Assignor to The American Metal Co., Ltd., New York. U.S. Pat. 1,331,802, 24.2.20. Appl., 1.10.18.

A SEPARATOR for molten materials of differing specific gravity consists of a rotary cylindrical vessel provided with two sets of tap holes diametrically opposed in the ends. The axis of the cylinder is slightly inclined to the horizontal, and axially disposed openings in the ends serve for the admission of material and heat to the interior.—T. St.

Heating or cooling apparatus. R. Achard, Lyon, France. U.S. Pat. 1,332,992, 24.2.20. Appl., 26.12.17.

A NUMBER of short doubled-walled tubes (the spaces between the two walls being narrow) are arranged horizontally in groups, with vertical pipes connecting alternate ends of the tubes. The liquid to be heated or cooled is passed through the vertical pipes and the short tubes, and the heating or cooling medium through the jackets of the double-walled tubes.—B. M. V.

Atomising liquids for purposes of drying, concentration, mixing, or reaction; Apparatus for ——. L. Galland, Berlin-Wilmersdorf. Ger. Pat. 307,152, 21.10.17.

Rows of jets are grouped in one or more clusters in an atomising or drying chamber. Each row or cluster has its own separate supply of compressed air and liquid. Drying of the atomised liquid is effected more quickly than is possible by the use of long atomising tubes passing through the chamber.—J. S. G. T.

Kilns for burning cement, lime, dolomite, etc.; Compressed air inlet for ——. Amme, Giesecke und Konegen A.-G., Brunswick. Ger. Pat. 315,775, 11.5.18.

THE mouth of the inlet is divided into channels by a grating composed of rods of rhombic cross-section, each of which can be separated into two parts of triangular cross-section. The inner side of the grating, which is the more liable to damage by heat and mechanical wear, is therefore easily replaced, whilst the sound rear portion can be retained; the device also facilitates the clearing of the narrowest parts of the air channels if these become choked.—D. F. T.

Filter-leaves. A. R. Peck, Los Angeles, Cal., U.S.A. Eng. Pat. 139,390, 22.10.18. (Appl. 16,240/19.) SEE U.S. Pat. 1,282,280 of 1918; this J., 1919, 34 A.

Filter-press and the like. J. Miller, Assignor to G. Fletcher and Co., Ltd., Derby. U.S. Pat. 1,331,350, 17.2.20. Appl., 3.6.19. SEE Eng. Pat. 126,265 of 1919; this J., 1919, 397 A.

Refrigeration; Process of ——. W. R. Ormandy, London, Assignor to Seay Syndicate, Ltd., Manchester. U.S. Pat. 1,331,356, 17.2.20. Appl., 13.4.17.

SEE Eng. Pat. 107,040 of 1916; this J., 1917, 917.

Grinding-mill and tumbling comminuting-body therefor. R. C. Newhouse, Assignor to Allis-Chalmers Manufacturing Co., Milwaukee, Wis. U.S. Pat. 1,331,964, 24.2.20. Appl., 29.4.15.

SEE Eng. Pat. 101,055 of 1916; this J., 1916, 954.

Cooling chambers. Eng. Pat. 139,383. SEE XIXA.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Coking plants; Standardisation of ——. O. M. Schadeck. Stahl u. Eisen, 1919, 39, 1349—1350.

THE necessity of standardisation of existing coking plants is discussed. No actual standards are suggested, but the author recommends the standardisation of pipes, dampers, arches, and the like, and the construction of standard units in new plants.—A. G.

Coal gas; Composition of the unsaturated hydrocarbons present in ——. F. S. Sinnatt and L. Slater. Analyst, 1920, 45, 85—87.

COAL gas was passed at the rate of 10 litres per hr. through wash-bottles containing bromine; at the end of the absorption period the liquid in the bottles was washed with dilute sodium hydroxide solution, then with water, and dried over anhydrous potassium carbonate. Two experiments were made, 700 litres of gas being used in the first and 1500 litres in the second, the weights of liquid bromides obtained being 235 and 520 grms. respectively. The liquid was distilled under 15 mm. pressure, the fractions boiling below 100° C. and between 100° C. and 145° C. being collected separately. The fraction boiling below 100° (15 mm.) was then fractionally distilled. From the results of the two experiments it is calculated that the composition of the unsaturated hydrocarbons was respectively ethylene, 83.8, 84.3; propylene, 13.4, 11.8; butylene, 1.7, 2.3; and amylene, 1.0, 1.5% by vol. The gas contained 3.5—4.5% of unsaturated hydrocarbons.—W. P. S.

[Gas] purification; Oxide ——. G. W. Anderson. Gas J., 1920, 149, 688—690.

THE best working results are obtained when 3—4 cb. ft. of oxide is provided for every 100,000 cb. ft. of gas per annum. Figures are quoted from working results of a number of plants running on gas containing 1.0—1.2% H₂S and 2% of air. Four purifier boxes were connected in series, the first one being disconnected and recharged as soon as any H₂S was detected at the outlet of the third box. The oxygen-content of the purified gas should not be higher than 0.2—0.3% and the temperature should not exceed 26°—30° C., the inlet temperature being 20° C.; 0.25% of steam per 1000 cb. ft. of gas may be admitted to keep the oxide moist. A mixture of "Lux" oxide with 30%—40% by volume of bog-ore is recommended. Such a mixture, operating under the conditions mentioned, can be discarded after once or twice revivifying. The same effect is obtained by the rotation system, which has been successful chiefly in plants where the quantity of available oxide in the purifiers was small in proportion to the amount of gas produced. The size of the purifying plant has an important bearing on its efficiency, and the design of the plant should proceed on the following lines:—If the gas velocity be 0.2 in. per sec., the section of each box should be 0.7 sq. ft. per 1000 cb. ft. output in 24 hours. If the total depth of oxide in each box exceeds 24—30 in., a divided flow of gas should be resorted to. For regeneration of the oxide outside the purifiers 0.67—0.87 sq. ft. of surface area is required per 1 cb. ft. of oxide in the purifiers. An estimate of the plant necessary to deal with a yearly output of 450 million cb. ft. of gas is given.—A. G.

Benzene [petrol] from lignite tar; Production of — by cracking. F. Fischer and W. Schneider. Ges. Abhandl. Kenntn. Kohle, 1918, 3, 122—149. Chem. Zentr., 1919, 90, IV., 1074—1075. (See this J., 1919, 672 A.)

BENZENE has been obtained hitherto from lignite

tar either by heating under pressure or by superheating at normal pressure; the behaviour of the tar when heated to the boiling point for a long time has now been examined. The fractions of tar from Riebeck's montan works which boil above 275° C. were partially decomposed when heated at 300°–315° C. for 13 hours. The specific gravity fell, and 20% distilled below 200° C., 73% below 250° C., and 88% below 275° C. Protracted heating of the tar fraction at 350°–500° C. lowered the specific gravity of the decomposition distillate to 0.81–0.83, and a yield of 66.9% was obtained, which fell to 63% after an acid-alkali wash. The products boiling below 300° C. may be regarded as newly formed and amount to 46.8%. The distillate has the composition:—B. pt. 60°–100° C., 4.2% (sp. gr. 0.681 at 20° C.); 100°–150°, 6.9% (0.743); 150°–200°, 11.3% (0.7799); 200°–250°, 19.7% (0.8198); 250°–300° C., 32.4% (sp. gr. 0.8502 at 20° C.). The yield of products boiling below 150° C. was less than is obtained by the other methods previously used. The products obtained by the different methods are compared in the following table:—

Lignite tar cracked by	Specific gravity of fraction.		Bromine number.	
	below 100° C.	100°–150° C.	below 100° C.	100°–150° C.
1. Heating under pressure ..	0.667	0.758	69	58
2. Slow heating .	0.681	0.743	112	98
3. Superheating at ordinary pressure ..	0.715	0.818	150	97

The products under Series 3 were treated with zinc chloride, which reduced the bromine number more than the alkali-acid wash. The yield of unsaturated compounds is much less where heating under pressure is used, probably because the compounds being retained for a time in an autoclave become polymerised. Experiments were made to see if polymerisation occurred in the protracted distillation as here practised. The tar was distilled and paraffin crystallised out at 5° C. After removing fractions boiling below 250° the tar was heated to 280°–330° C. The specific gravity of the distillate (0.88–0.89) indicated little decomposition. A further heating for 64 hours at 335°–350° C. gave after the acid and alkali wash 6.7% of a light oil up to 150° C., indicating considerable decomposition. The residue was a semi-solid mass (13.9%) from which a lubricating oil could be obtained with viscosity at 20° C., 35.5° (Engler), flash point 108°, sp. gr. at 20°, 0.992, setting point 11° (without stirring). The corresponding fraction from a tar rapidly distilled *in vacuo* had a viscosity at 20°, 19.2° (Engler), setting point 15° (without stirring), flash point 197°, sp. gr. at 20°, 0.965. The protracted distillation had led to a certain thickening of the oil. The disadvantages of the lignite benzines are the pungent smell and high percentage of unsaturated compounds. Decomposition under pressure reduces the latter, as also does washing and hydrogenation. In the future it is possible that these products will form the raw material for chemical manufactures.—H. J. H.

[Petroleum] oil-field emulsions. J. L. Sherrick. J. Ind. Eng. Chem., 1920, 12, 133–139.

To be accepted by transporting companies in the United States crude petroleum must not contain more than 2% of water, and this necessitates treatment of emulsions in the field. The emulsions in Goose Creek (Texas) oil-fields consist of salt solution and oil containing asphalt, and experimental

results obtained with them are applicable to the products of that district in general. When subjected to an electrical current the water particles migrated to the anode. The negative charge on the water particles could be neutralised and the water precipitated by the addition of cations which were strongly adsorbed, such as ferric ion. The results obtained with ferric chloride and ferric nitrate showed that both ions of an added electrolyte are effective in precipitating the charged particles. The effect of ether in reducing the stability of such emulsions is probably due to its solvent action upon the asphalt, rather than to any increase in the density of the two liquid phases. A mixture of ether and carbon bisulphide of the same sp. gr. as the emulsion will also cause water to separate. It is probable that the action of certain commercial preparations used for treating emulsions is due to their being hydrophilic colloids.

—C. A. M.

Paraffin was (pentacontane $C_{50}H_{102}$) found in Lancashire coal; Solid —. F. S. Sinnatt and M. Barash. Inst. Min. Eng., Nov. 11, 1919.

A DEPOSIT of several tons weight was found over 30 years ago in the Arley mine at a depth of 440 yards. This consisted of a dark brown, brittle, tasteless, and odourless wax. It contains no sulphur, nitrogen, ash, or halogens, and is practically insoluble in all solvents in the cold, but dissolves in hot benzene, toluene, chloroform, carbon tetrachloride, and aniline. The wax is unaffected by strong alkalis and acids, except by hot concentrated sulphuric acid. Hot liquid bromine has no action on the wax. On bleaching a hot benzene solution with animal charcoal, a white amorphous solid was produced, m. pt. 93.0° and solidified at 92.7° C. Under 15 mm. pressure the wax boiled at 420°–422° C., and about 85% distilled without decomposition. The sp. gr. is 0.9422 when cold and 0.7940 at the melting point. The substance has the formula $C_{50}H_{102}$, and is the highest homologue of the paraffin series so far discovered occurring naturally.

—A. G.

Heating Martin furnace with coke-oven gas. Springorum. See X.

Heating metallurgical furnaces. Gouvy. See X.

Chlorination of ethylene. Smythe. See XX.

Blown-out shots in coal mines. Hutchison and Barab. See XXII.

Cuprous chloride solution for gas analysis. Kauskopf and Purdy. See XXIII.

Combustible gases. Schmidt-Jensen. See XXIII.

PATENTS.

Fuel; Artificial —. L. Lane, Loughor, and D. H. Williams, Grovesend, S. Wales. Eng. Pat. 139,094, 2.7.19. (Appl. 16,523/19.)

THE fuel consists of a mixture of anthracite duff, waste pitch, sodium nitrate, carbonaceous shale, and/or "clod," with or without a small quantity of dried clay. The waste pitch is a grease produced in the lubrication of rolls or similar machinery or in the manufacture of tin-plate. "Clod" is mineralised mud mixed with carbonaceous clay. The coal is heated and mixed with the nitre before adding the waste pitch, and the mixture is briquetted and left to harden for 24 hours.—A. G.

Coal; Agglutinant for the conglomeration of —. L. A. E. Irazusta, Zarauz, Spain. Eng. Pat. 139,106, 7.8.19. (Appl. 19,477/19.)

COAL is briquetted with the aid of 3–4% of an agglutinant composed of 3% of tallow or other greasy substance and 97% of tar.—A. G.

Fuel; Artificial —. B. G. Seccombe and J. Sandiford, Manchester. Eng. Pat. 139,115, 25.8.19. (Appl. 20,800/19.)

COAL slack is briquetted with clay, lime, fibrous material, salt, nitre, alum, and sulphur.—A. G.

Pulverulent fuel installations for use in connection with furnaces. Quigley Furnace Specialties Co., Inc., New York. Eng. Pat. 126,638, 7.5.19. (Appl. 11,386/19.) Int. Conv., 8.5.18.

CRUSHED coal freed from iron by means of a magnetic separator is dried in a rotary dryer and conveyed to storage-bins which feed the pulverisers. A suction fan conveys air and pulverised coal from the pulveriser to a cyclone-separator from which the main body of air returns to the pulveriser and is used over and over again. The coal dust is delivered to a storage-bin with a gate discharge, through which it falls into a blowing tank comprising a large upright cylinder set upon the platform of a weighing-machine of the "quick reading, net" type. From the tank onward a compressed-air system of transportation for the pulverised coal is employed. The outflow of fuel from the tank is controlled by internal mechanism operated by a bell-crank and lever which can be locked in any desired position by a quadrant and latch device. The flow of fuel to any particular furnace on the distribution main can be regulated by the weight of fuel sent from the blowing tank. Accumulations of pulverised coal in the main can be cleared by means of an auxiliary compressed air line. The fuel is injected into the furnace by means of a jet, secondary air being supplied for combustion by means of a concentric pipe. The advantages of this system are that only such quantity of coal is pulverised as is needed for immediate use, and the powdered coal being kept in air-tight storage bins, oxidation is retarded, and hence spontaneous combustion is not likely to occur.—A. G.

Storage of acetylene or other gas; Fillings for tanks or the like intended for the —. A. U. Särnmark, Göteborg, Sweden. Eng. Pat. 123,534, 17.2.19. (Appl. 3927/19.)

THE filling material is composed of ceramic or other porous material reinforced by loose strands of metal wire, such as iron, of short length evenly distributed throughout the mass. To prevent entanglement the strands are straight or only slightly bent. The reinforced mass may be made into briquettes or slabs, which are inserted in the tanks. (Reference is directed to Eng. Pat. 23,856 of 1910; this J., 1911, 349.)—J. S. G. T.

Liquid hydrocarbons; Refining —. A. E. Dunstan, Sunbury-on-Thames. Eng. Pat. 139,233, 3.6.18. (Appl. 9114/18.)

OXIDISABLE sulphur compounds present in mineral oils are removed by the action of an alkaline solution of a hypochlorite, such as sodium hypochlorite.—A. E. D.

Motor fuel for internal combustion engines. G. F. Dinsmore, Boston, Mass. U.S. Pat. 1,331,054, 17.2.20. Appl., 19.3.19.

CRUDE wood naphtha is mixed with petroleum distillate (kerosene). The acetone is dissolved by the kerosene, and the solution is separated from the undissolved methyl alcohol.—A. E. D.

Oil-cracking still. J. L. Gray, St. Louis, Mo. U.S. Pat. 1,331,909, 24.2.20. Appl., 21.2.17. Renewed 13.11.19.

OIL is superheated in a cast-iron cylindrical casing containing straight lengths of wrought-iron pipe arranged parallel to the axis of the cylinder and embedded in its wall. These pipes are connected by means of return bends, so as to form continuous

conduits passing from side to side from the lower to the upper part of the casing.—C. A. M.

Liquid fuel; Production of —. Konsortium für Elektrochem. Industrie G.m.b.H., Munich. Ger. Pat. 315,290, 30.8.17.

ACETALDEHYDE and paraldehyde are mixed in proportion adjusted to give the desired volatility. The mixture, which burns with a non-luminous and non-smoky flame, has approximately the same calorific value as methylated spirit.—H. J. H.

Lubricating oil [from mineral oils]; Process for obtaining — by means of sulphurous acid. Allgem. Ges. für chem. Industrie m.b.H., Berlin. Ger. Pat. 315,289, 20.7.17.

THE portion soluble in liquid sulphur dioxide which is extracted from suitable hydrocarbon oils is treated with dry ammonia gas, and the ammonium salts thus formed are removed. The excess of ammonia is expelled by passing air through the heated oil. The product contains an extremely small quantity of ammonium sulphite and ammonium sulphate, which remains dissolved in the oil or finely suspended. The lubricating oil produced has a high viscosity and a very low solidifying point.—J. F. B.

Aluminium chloride residues [from treatment of mineral oils]; Utilisation of —. L. S. Abbott, Port Arthur, Tex., Assignor to Gulf Refining Co., Pittsburgh, Pa. U.S. Pat. 1,332,018, 24.2.20. Appl., 1.7.19.

PART of the carbon in the residues is burned to heat the remaining carbon to incandescence, and a current of heated chlorine is then passed through the incandescent mass.—W. J. W.

Lubricating oil purifying and reclaiming systems. The De Laval Separator Co., New York, Assignees of F. W. Kennedy, Trenton, N.J., U.S.A. Eng. Pat. 134,196, 13.8.19. (Appl. 19,904/19.) Int. Conv., 23.10.18.

Combustion products; Apparatus for generating — under pressure. C. M. Pattison, Pentre, Wales. U.S. Pat. 1,331,523, 24.2.20. Appl., 27.11.18.

SEE Eng. Pat. 129,725 of 1917; this J., 1919, 673 A.

Hydrocarbons; Treatment of liquid — for the transformation thereof. N. C. and C. E. Cassal and B. H. Gerrans, London. U.S. Pat. 1,330,844, 17.2.20. Appl., 12.9.17.

SEE Eng. Pat. 117,087 of 1916; this J., 1918, 501 A.

IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

PATENTS.

Defecation sludge obtained in the manufacture of sugar; Production of terpenes and other products by dry distillation of —. H. Stoltzenberg, Berlin-Wilmersdorf. Ger. Pat. 316,503, 8.8.17.

To prevent decomposition the sludge is dried immediately it is produced without the addition of any other substances, and the dried product is distilled. The oily and aqueous fractions of the distillate are worked up separately to resin substitutes, pyridine bases, acetone, and Pyrrole Red. An alternative method is to separate the sludge into three fractions by precipitation and treat these separately: a resinous fraction yielding a high percentage of terpenes and resin; a fraction with a high nitrogen content yielding mainly bases; and a fraction rich in fatty acids yielding principally acetone.—L. A. C.

Electric incandescent lamps. Siemens und Halske A.-G., Siemensstadt, Germany. Eng. Pat. 112,264, 22.11.17. (Appl. 17,211/17.) Int. Conv., 29.12.16.

In electric incandescence lamps, more particularly those known as gas-filled carbon lamps, rare gases specially purified to remove the last traces of nitrogen are used as the lamp-filling medium. The life of the filament is much increased thereby and its load capacity considerably augmented.—J. S. G. T.

Leading-in conductor [e.g., for electric lamps]. J. H. Anderson, Cleveland, Ohio, Assignor to General Electric Co. U.S. Pat. 1,331,085, 17.2.20. Appl., 28.8.15.

THE conductors are composed of a metal having a higher coefficient of expansion than the vitreous material into which they are sealed, and they are provided with one or more thin radial flanges at the sealing point.—B. V. S.

Vacuum tubes; Production of electropositive electrodes in —. W. Kossel, Munich. Ger. Pat. 307,674, 26.1.18.

A LESS electropositive material is converted into a more electropositive substance *in vacuo* in the absence of oxygen. As an example, amalgams are in general more electropositive than the corresponding metals, e.g., zinc.—J. S. G. T.

Discharge tubes; Incandescence cathode for —. F. Skaupy, Berlin. Ger. Pat. 310,712, 16.6.18.

In cathodes of the Wehnelt type the support for the material affording emission of electrons is composed of osmium. The range of possible substances which may be employed as sources of electronic emission is thereby extended on account of the higher temperature at which osmium may be used compared with platinum and iridium.—J. S. G. T.

III.—TAR AND TAR PRODUCTS.

Lignite tar; Action of concentrated and fuming sulphuric acid on the fraction 200°–300° C. of —. W. Schneider and O. Jantsch. Ges. Abhandl. Kenntn. Kohle, 1918, 3, 191–99. Chem. Zentr., 1919, 90, IV., 1073.

THE fraction 200°–300° C. of a tar from the Riebeck Montan works freed from phenols and bases was shaken repeatedly with concentrated sulphuric acid and 59.4% was dissolved. The process was continued with fuming acid containing respectively 9% SO₃ and 15% SO₃, when a further 29.2% of the oil dissolved, leaving 11.2% of a colourless oil, 90% of which distilled between 200° and 300° C. and had sp. gr. 0.772 at 20° C. The residue solidified on cooling. The same fraction of a low temperature coal tar treated similarly left almost the same percentage of residue insoluble in acid, but a larger proportion dissolved in the concentrated acid. It is concluded that the proportion of paraffin is approximately the same in the fraction of both tars, whereas the lignite tar fraction contains the smaller proportion of unsaturated hydrocarbons (soluble in strong sulphuric acid) and a larger proportion of aromatic and hydroaromatic hydrocarbons (soluble in fuming sulphuric acid). 500 c.c. of a lignite distillation tar fraction (200°–300° C.) washed free of phenols and bases was treated once with 550 c.c. of concentrated sulphuric acid, leaving 48% undissolved. The acid tar treated with water gave 18.2% of neutral oil and 21% of oily sulphonic acid. The neutral oil was a moderately viscous lubricant, 40% of which distilled over up to 287° C. when decomposition set in.—H. J. H.

Catechol. B. Elsner. Monatsh., 1919, 40, 361–362.

AT dilutions not greater than 1%, with excess of cold barium hydroxide solution, catechol gives a precipitate of a very sparingly soluble barium salt, C₆H₂O₂.Ba₃11₂O, in the form of silver-grey leaflets with a pearly lustre. Since resorcinol and quinol give no such precipitate, the reaction is suggested as a means of separation, which is probably superior to that of depending upon the use of benzene.

—J. K.

PATENTS.

Sulphonation, nitration, and chlorination of aromatic substances; Apparatus applicable for the —. H. N. Morris and Co., and H. N. Morris, Manchester. Eng. Pat. 139,234, 5.6.18. (Appl. 9256/18.)

THE apparatus consists of a number of reaction chambers each divided into two compartments, one above the other, with communicating passages and condensing chambers above; the upper compartment of each reaction chamber is fitted with two or more atomisers through which the reacting substances are introduced and the lower compartment with coils for the control of the temperature.

—D. F. T.

Carbazole; Purification of —. South Metropolitan Gas Co., and W. Kirby, London. Eng. Pat. 139,441, 11.11.19. (Appl. 28,279/19.)

THE precipitate produced by the addition of water to a solution of carbazole in concentrated sulphuric acid is obtained in comparatively large crystals which can easily be removed by filtration if the dilution is effected by exposure to a moist atmosphere.—D. F. T.

Side-chain oxidation. [Preparation of benzoic acid from toluene.] V. Coblenz and H. W. Walker, Assignors to Commercial Research Co., New York. U.S. Pat. 1,332,028, 24.2.20. Appl., 7.4.19.

BENZOIC acid is manufactured from toluene by "stratifying" this with nitric acid of 20–43% concentration at a temperature somewhat below the boiling point of the toluene, the materials being left in contact until reaction is complete and escaping vapours condensed and returned to the reaction mixture.—D. F. T.

Retene and its derivatives; Preparation of aromatic nitro-compounds from —. R. Arnot, Zürich. Ger. Pat. 315,623, 2.8.18.

RETENE, retenequinone, or high-boiling resin oil (tar tallow) is treated with nitric acid of sp. gr. greater than 1.43 in the presence or absence of fuming sulphuric acid. *Examples.*—Dinitroretene, prepared by the action of nitric acid of sp. gr. 1.48 to 1.52 at 15° C. on retene or tar tallow, is a flocculent yellow compound soluble with difficulty in aqueous alkalis, and soluble in most organic solvents; on reduction it forms an amine which can be diazotised and coupled with the usual components. Nitroretene-sulphonic acid, a yellowish-brown compound, is prepared by the action of nitric acid and 20% oleum on retene.—L. A. C.

Naphthalene and other crystalline substances which can be sublimed; Purification of —. Mitteldutsche Teerprodukten- und Dachpappenfabr. P. Riehm, Grifte. Ger. Pat. 315,958, 6.3.18.

NAPHTHALENE, phthalic anhydride, camphor, indigo, arsenious acid, cinnabar, ammonium chloride, or the like, is purified by fractional sublimation in an apparatus consisting of an inner chamber into which the vapour is first led, one or more outer chambers surrounding the inner chamber on all sides, and a final cooling chamber. In the case of naphthalene, large crystals of a high degree of purity are de-

posited in the inner chamber; the product in the outer chambers is somewhat less pure, while the last fraction deposited in the final chamber is of an oily nature.—L. A. C.

Coumarone resin. Ger. Pat. 302,543. See XIII.

IV.—COLOURING MATTERS AND DYES.

Azo-derivatives of indoxyl. J. Martinet and O. Dornier. Comptes rend., 1920, 170, 592—594.

WHEN the requisite amount of a diazo compound is added to a mixture of ice and an indoxyl melt neutralised by sulphuric acid, azo-derivatives of indoxyl are readily obtained. The authors have, in this way, prepared 2-methyl-, 3-methyl-, 4-methyl-, 2,4-dimethyl- and 2,3,5-trimethylbenzeneazindoxyls and α - and β -naphthaleneazindoxyls. All of these compounds decompose on melting. (See further J. Chem. Soc., 1920, i., 335.)—W. G.

[*Light*] filters. Miethe and Stenger. See XXI.

PATENT.

Purifying substances by sublimation. Ger. Pat. 315,958. See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Textiles; Testing the mildew resistance of —. B. S. Levine and F. P. Veitch. J. Ind. Eng. Chem., 1920, 12, 139—141.

IN testing the resistance offered by fabrics to mildew the incubation period should not be less than 30 days under aerobic conditions of growth in subdued light, and at a suitable humidity and temperature. Six discs about 1½ in. in diameter are cut from the fabric to be tested and immersed in running (or frequently changed) water for at least 2 days to remove water-soluble fermentable or fungicidal substances. They are then pressed between absorbent paper and placed in six bacteriological Petri plates containing 10 to 15 c.c. of agar jelly free from nutrient substances. The plates, which must not be air-tight, are incubated for 7 to 10 days in a closed chamber at 20°—25° C. If a well-developed growth has appeared the test is discontinued. Otherwise the discs are inoculated with stock cultures of *Alternaria*, *Cladosporium* and a pink *Mucor*, and the incubation renewed for 3 to 4 weeks. After this period the amount, nature, and extent of the growth, and the amount of discoloration and the strength of the fabric are noted, and the sample classified in accordance with an arbitrary scale in which 0 represents susceptibility to mildew with tendering of the cloth, and 10 "mildew-proof," with no growth of mould and no discoloration of the cloth. Laboratory results thus obtained agreed fairly well with the results of practical tests in which pieces of treated and untreated cloth were stretched in the form of tents and exposed to weather conditions for upwards of a year.—C. A. M.

Flax and hemp fibres; Differentiation of —. R. Haller. Neue Faserstoffe, 1919, 1, 229—230. Chem. Zentr., 1920, 91, II., 48.

THE fibres of flax and hemp show quite different swelling phenomena on treatment with concentrated caustic soda. Flax swells uniformly, and the contents of the central canal show up distinctly, whilst the fibre substance becomes uniformly transparent. In the case of hemp the contours become irregular, the fibre remains non-transparent, and the wall of the central canal does not stand out

distinctly. The diameter of the flax fibre increases on an average by about 83%, and that of the hemp fibre by only 25%. The flax fibre in contact with the alkali makes likely worm-like contortions; the hemp fibre also bends about but less vigorously. On treatment with cuprammonium reagent the flax does not show barrel-shaped swellings, but the hemp fibre does. If the fibres are dyed with a strong solution of Dianil Blue PH in concentrated caustic potash lye and examined under the microscope, the flax shows knotty swellings and cross lines at irregular intervals which are more deeply dyed; in the case of hemp there are numerous cross lines, but these are not appreciably darker in colour than the rest of the fibre.—J. F. B.

Aeroplanes; Material and dopes for the planes of —. A. Fuchs. Chim. et Ind., 1920, 3, 167—172.

DETAILS are given of the various materials used for planes and of the dopes or varnishes used to render them waterproof. Cellulose acetate and other materials used as bases for the dopes are discussed, as well as the most appropriate solvents. Materials for increasing the plasticity of the film when dry and for reducing its liability to fire, the methods of applying the dopes, of drying the films, and of recovering the vaporised solvents are considered.

—W. H. C.

PATENTS.

Fibrous plants [yucca]; Process of treating — for the recovery of fibres. W. Loehr, Bloomington, Cal. U.S. Pat. 1,330,127, 10.2.20. Appl., 28.5.19.

YUCCA plants are crushed, boiled in an alkaline solution until the cellular tissue is softened, treated with dilute solution of alum, washed in cold water, and the fibre then shaken and dried.—J. F. B.

Vegetable fibres; Process for making — lustrous. P. Kraiss, Dresden, and O. Röhm, Darmstadt. Ger. Pat. 315,398, 24.9.18.

FIBRES in the raw or manufactured form are treated for a long time with a solution of the enzymes of the pancreatic gland at a moderate temperature, then washed and dried. Flax, ramie, and nettle fibres so treated acquire a permanent silky gloss.

—J. F. B.

Papers for use as a dielectric for electrical condensers and other purposes. F. Haigh, Stoke-on-Trent. Eng. Pat. 138,226, 9.4.19. (Appl. 8969/19.)

EXTREMELY thin tissue paper is coated with a homogeneous substance possessing good electrical insulating properties, such as pyroxylin, collodion, nitro-cellulose, or cellulose acetate. The thin paper is supported by a backing of a heavier paper, the two papers being pressed together in a moist condition during manufacture (Eng. Pat. 24,981 of 1911). The coating is then applied to the thin paper while in contact with the support, and the backing is removed before the paper is used.—J. F. B.

Paper, yarns, and fabrics; Process for waterproofing —. H. T. Böhme A.-G., Chemnitz. Ger. Pat. 315,412, 13.12.16.

THE goods are treated with water-soluble oils and sizing agents. Complete waterproofing of paper is effected by precipitating the oil from its aqueous emulsion by means of weak acids or acid salts on the fibre, with the simultaneous use of sizing agents. The oils may be added to the pulp in the hollander.

—J. F. B.

Sponge; Manufacture of an elastic artificial — from plant structures. J. Hach and E. Dreyfuss, Kaiserslautern. Ger. Pat. 315,185, 9.2.18.

THE leaves and stems of various peat mosses, especi-

ally *Sphagnum cymbifolium*, are torn up in a "rag-devil" and treated in the half-dry condition with calcium hydroxide, the excess of which is subsequently neutralised with dilute acetic acid. The cell walls are thereby hardened or "tanned" and permanently preserve their elastic condition. The mass has the properties of a natural sponge.

—J. F. B.

Artificial silk; Apparatus employed in manufacture of —, J. Clayton, Coventry. Eng. Pat. 139,104, 5.8.19. (Appl. 19,297/19.)

Paper making machines; Dryer for —, J. E. Alexander, Port Edwards, Wis. U.S. Pat. 1,390,366, 10.2.20. Appl. 26.11.17.

Drying cylinders; [Doll-head bearings of] textile and like —, E. Farrell, Salford. Eng. Pat. 139,261. (Appls. 1876, 25.1.19, 7073, 21.3.19, and 9681, 16.4.19.)

Impregnating or treating textile fabrics and other materials; Apparatus for —, J. D. Taylor, London. U.S. Pat. 1,331,729, 21.2.20. Appl. 14.11.17.

SEE Eng. Pat. 106,805 of 1917; this J., 1917, 869.

Adhesives. Ger. Pats. 316,080 and 316,234. See XVII.

Saccharification of cellulose. Ger. Pat. 304,400. See XVI.

Saccharification of cellulosic material. Ger. Pat. 304,399. See XIXa.

Product for nitration from wood cellulose and cotton. Ger. Pat. 300,844. See XXII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Printing white reserves on wool. M. Battagay. Bull. Soc. Ind. Mulhouse, 1919, 85, 103—141.

THE strong reducing properties of wool distinguish it from cotton. Consequently discharges on wool must be brought about by reduction. Theoretical considerations, supported by experiment, indicate the hydrosulphites—especially if they be mixed with a covering material such as zinc oxide and lithopone—to be most suitable. A suitable paste is 200 grms. of sodium tungstate, 200 grms. of thickening (e.g., 1 part of maize starch, 1 of water, 8 of gum tragacanth), 500 grms. of hyalidite, and 20—50 grms. of glycerin. The tungstate is fixed in a bath of a barium salt. The substituted amines, formed by reduction of the dyestuff, unite with the wool and determine the possibility of future discoloration. Naphthylamines substituted in the 1- and 8-positions give bad discolorations. *p*-Phenylene-diamine is inert. In the latter part of the paper the chemical and physical properties of wool are summarised, and its relationship to the proteins is shown.—A. J. H.

Discharges on wool. E. Jaquet. Sealed note 1432, Dec. 12, 1903. Bull. Soc. Ind. Mulhouse, 1919, 85, 142. Report by M. Battagay, *ibid*, 142—143.

A METHOD for obtaining white discharges on wool by means of a mixture of sodium hydrosulphite, albumin, zinc oxide, and gum is described. Battagay reports this discharge to be liable to discoloration (see preceding abstract).—A. J. H.

PATENTS.

Washing textiles. L. Elkan Erben, G.m.b.H., Berlin. Ger. Pat. 315,835, 21.11.17.

AN electric current is passed through a solution of saponin and sodium chloride whilst this is being used for washing textiles.—L. A. C.

Water-glass; Means for preventing the harmful effects of fabrics cleaned with —, W. Reichel, Munich. Ger. Pat. 316,293, 16.1.17.

FABRICS cleaned with water-glass are rinsed afterwards in a dilute solution of ammonium chloride to prevent them from causing irritation on coming in contact with the human skin.—L. A. C.

Colouring product and tinctorial mordant, R. H. and M. H. Vegas, Buenos Aires, Argentina. U.S. Pat. 1,330,992, 17.2.20. Appl. 17.6.18.

SEE Eng. Pat. 129,761 of 1918; this J., 1919, 760 A.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sodium perborate and perborate soap powders; Determination of available oxygen in —, H. Trickett. Analyst, 1920, 45, 88—91.

THE iodometric method yields the most trustworthy results, but, in the case of soap powders and the like previous removal of the soap is necessary. A method which may be applied directly to perborates and soap powders consists in treating the perborate solution with sodium hypochlorite solution in a nitrometer and measuring the volume of the liberated oxygen. The results obtained agree with those found by the iodometric method. Titration of perborates with permanganate is not very reliable; the best results are obtained by adding a slight excess of the perborate solution to a definite volume of permanganate solution acidified with sulphuric acid, and then titrating the excess with permanganate. The titration should be carried out at 15°—20° C., and the acid concentration should be 5 of sulphuric acid on the total volume. The gasometric determination of perborates by decomposition with permanganate is untrustworthy.

—W. P. S.

"Buffer" system; primary phosphate-bicarbonate-free carbonic acid, in place of the system; primary phosphate-secondary phosphate. W. Windisch and W. Dietrich. Woch. Bran., 1920, 37, 81—83.

THE authors investigated the changes of surface tension of solutions of eucupine dihydrochloride resulting from the liberation of the capillary-active free base (cp. Traube, this J., 1915, 853) under the influence of secondary potassium phosphate before and after treatment of the solutions with carbon dioxide. It is concluded that excess of carbon dioxide effects a practically complete conversion of secondary potassium phosphate into the primary salt and potassium bicarbonate. In living and breathing tissues, and in fermenting liquids, therefore, all the phosphate must be present as primary salt in equilibrium with potassium bicarbonate and free carbon dioxide. Primary and secondary phosphates can only coexist after production of carbon dioxide by respiration or fermentation has ceased.

—J. H. L.

Water-glass; Colloidal method for increasing the volume of adhesive —, J. D. Macdonald. J. Ind. Eng. Chem., 1920, 12, 174—176.

ON adding an electrolyte to water-glass the colloidal silica is coagulated, but if the solution is stirred during the addition, and for some time afterwards,

the gel particles cannot coalesce, but are redissolved, i.e., "peptised." By adding brine about two-thirds saturated in a thin stream to the water-glass whilst this is mechanically stirred it is possible to increase the volume of the water-glass solution up to about 25% without impairing the viscosity or adhesive properties of the liquid, which will contain from 3 to 4% of sodium chloride. The final viscosity of the solution may be varied to a considerable extent by small changes in the concentration of the brine. The treated adhesive does not deteriorate when kept out of contact with the air.

—C. A. M.

Lead chloride; Vapour pressure of —. E. D. Eastman and L. H. Duschak. U.S. Bureau of Mines, Tech. Paper 225, June, 1919. 14 pages.

In connection with the treatment of ores by a process involving a chloridising roast and volatilisation of the chlorides of the metallic constituents, the vapour pressure of lead chloride was determined. The method employed was a determination of the weight of substance required to saturate a quantity of inert gas at constant temperatures and pressures. The vapour pressures for temperatures between 500° and 850° C. are given and are shown on a graph, as well as those for solid lead chloride down to 400° C. Values for the vapour pressure estimated from the "smoothed" graph are: 400°, 0.00174; 500°, 0.141; 600°, 2.82; 700°, 22.4; 800°, 116; 900°, 433 mm. of mercury at 0° C. The melting point was determined as 495° C., and the boiling point as 945° C. at 754 mm. The heat of vaporisation at the melting point was calculated to be 40,600 cal.

—W. J. W.

Marine algæ as foods. Gloess. See XIXa.

Titration of mixed acids. Kolthoff. See XXIII.

Halogens. Lamb and others. See XXIII.

Ammonium molybdate. Neubauer and Wolferts. See XXIII.

PATENTS.

Nitric acid; Manufacture of highly concentrated — from dilute nitric acid. A. Clemm, Mannheim. Ger. Pats. (A) 303,313 and (B) 304,233, 2.12.16.

(A) DILUTE nitric acid is converted into barium nitrate by the addition of barium sulphide. The barium nitrate is freed from water and heated with concentrated sulphuric acid, and the residual barium sulphate is reduced. Strontium sulphide may be employed instead of barium sulphide. (B) The dried barium nitrate is treated with an excess of concentrated sulphuric acid at the ordinary temperature, and the mixed acid obtained, which is of such a concentration that it may safely be transported in iron vessels, is separated from barium sulphate by decantation or filtration in a closed heated filter. Residual nitric acid is removed from the barium sulphate by washing with concentrated sulphuric acid, and the sulphuric acid remaining in contact with the barium sulphate is utilised by adding the requisite amount of barium nitrate and heating the mixture, as described in the previous patent.—L. A. C.

Nitric acid; Concentration of aqueous —. H. Pauling, Berlin-Lichterfelde. Ger. Pat. 305,553, 31.5.17.

THE aqueous nitric acid and the dehydrating agent with which it is mixed, e.g., sulphuric acid, are heated, before or after mixing, and the heated mixture is submitted to the action of a current of dry air or other inert gas, also heated if necessary;

the evolved nitric acid vapours may be further treated with sulphuric acid. A similar process can also be applied to the dehydration of other substances, such as alcohol and acetic acid.—D. F. T.

Nitrogen oxides; Process for recovery of —. *Process for recovery of nitrous oxides.* P. A. Guye, Geneva, Switzerland. U.S. Pats. (A) 1,331,104 and (B) 1,331,105, 17.2.20. Appl., 4.1.19.

(A) NITROGEN oxides are recovered from a mixture with other gases by oxidation and subsequent liquefaction by refrigeration, the rate of oxidation being regulated so as to produce a mixture of oxides of nitrogen which will not solidify above 0° C. (B) Nitrogen oxides are recovered from a mixture with other gases by liquefying them at a temperature not exceeding -20° C. by means of a refrigerating liquid capable of dissolving nitrogen oxides without chemical interaction.—W. J. W.

Alumina; Manufacture of —. K. J. P. Orton and G. W. Robinson, Bangor. Eng. Pat. 139,005, 26.2.19. (Appl. 4827/19.)

AN aluminiferous mineral containing a high percentage of iron oxide, e.g., slate, is finely powdered and heated with 3—4 times its weight of nitre cake to a cherry-red heat for a short time. The product is lixiviated with water, and the slightly acid solution, diluted to about 15,000 parts per 100 parts of the mineral, is heated to 70°—80° C. until no more hydrated ferric oxide separates. Alumina and any remaining ferric oxide are precipitated from the filtered solution, and the ferric oxide is removed by the usual method.—L. A. C.

Zinc sulphate; Process for the purification of solutions of —. Comp. des Prod. Chim. d'Alais et de la Camargue, Paris. Eng. Pat. 139,443, 17.11.19. (Appl. 28,471/19.) Int. Conv., 12.9.19.

COPPER or/and cadmium sulphates present in a solution of zinc sulphate are removed by placing plates of copper or cadmium respectively and zinc, externally connected by an electrical conductor, in the solution. A difference of potential is set up between the two plates; copper (or cadmium) is deposited on the plate of the same metal, and the equivalent weight of zinc is dissolved from the zinc plate. A plate of carbon or of a metal less readily attacked than zinc by the SO₄ group is employed instead of copper or cadmium if the sulphates of both these metals are present.—L. A. C.

Ammonia-saturator. A. Roberts, Evanston, Ill., Assignor to American Coke and Chemical Co., Chicago, Ill. U.S. Pat. 1,331,784, 24.2.20. Appl., 17.4.15. Renewed 9.5.19.

THE apparatus comprises a tower closed at the upper end and communicating at its lower end with a hopper which has a bottom discharge, and is provided with a baffle parallel to one side. Slanting baffles project alternately from the opposing walls across the internal space of the tower over which the liquid flows from a connecting pipe at the top. Gas intake and discharge connexions communicate respectively with the bottom and top of the tower.

—W. J. W.

Carbon dioxide gas; Process of and apparatus for producing —. R. C. Newhouse, Wauwatosa, Assignor to Allis-Chalmers Manufacturing Co., Milwaukee, Wis. U.S. Pat. 1,332,138, 24.2.20. Appl., 27.6.19.

A gas rich in carbon dioxide is produced by treating incandescent carbon with a compound containing oxygen under such conditions that the percentage of nitrogen in the product is a minimum, then burning the gas produced to calcine a carbonate, and collecting the resulting gases.—L. A. C.

Sulphur present in natural sulphates; Utilisation of —. Badische Anilin und Soda-Fabrik, Ludwigshafen. Ger. Pat. 305,123, 29.9.17.

THE gas obtained by heating sulphates, e.g., gypsum, and a combustible material in a current of air is separated from free sulphur, mixed with air, and passed over a catalyst consisting of an infusible material saturated with a solution of an iron salt. The catalytic material is first brought to the temperature required to start the reaction, but as the reaction proceeds, i.e., oxidation of carbon monoxide and sulphur compounds to carbon dioxide and sulphur dioxide, the heat of combustion is sufficient to maintain the desired temperature.—L. A. C.

Magnesium carbonate or ammonium magnesium carbonate; Manufacture of —. "Lipsia" Chem. Fabr., Mügeln. Ger. Pat. 306,335, 25.11.17.

A SOLUTION containing ammonium carbonate, such as the filtered liquor obtained in the ammonia-soda process, is treated with the requisite quantity of free ammonia, and added to a solution of a magnesium salt.—L. A. C.

Sulphur dioxide; Manufacture of — from calcium, barium, and strontium sulphites. Chem. Fabr. vorm. Weiler-Ter Meer, Uerdingen. Ger. Pat. 307,121, 23.3.18.

SULPHUR dioxide is prepared by heating calcium, barium, or strontium sulphite with magnesium salts, such as crude magnesium chloride or sulphate, or mixtures of the same.—L. A. C.

Nitrogen and hydrogen; Synthetic production of compounds of —. H. Harter and H. Braun, Berlin-Schöneberg. Ger. Pat. 310,623, 18.7.17.

IN the synthetic production of compounds of hydrogen and nitrogen the contact mass is constructed of iron tubes and rods. The rods are so treated that iron, ferrous oxide, and ferric oxide are all present in the mass. The conditions of the reaction are so adjusted that hydrogen peroxide is formed during the reaction, and to assist in its production suitable metals capable of combining with hydrogen are added to the iron. The contact mass is packed in a number of narrow iron tubes arranged within a wider iron tube.—L. A. C.

Salts; Removal of iron from inorganic —. E. Natho, Essen-Bredency. Ger. Pat. 315,837, 31.5.18.

SATURATED solutions of aluminium, potassium, and sodium sulphates can be freed completely from iron by treating them with sulphite-cellulose waste liquor; alternatively dilute solutions may be treated and then concentrated by evaporation; the iron is precipitated as iron-tannin compounds together with dextrans.—D. F. T.

Glauber's salt; Removal of iron from —. E. Natho, Essen-Bredency. Ger. Pat. 315,940, 31.5.18.

THE salt is treated with an oxidising agent, such as bleaching powder, and calcium chloride, and then heated to 400° C.; the iron is eliminated as volatile ferric chloride; if no oxidising agent is used a temperature of 1400° C. is necessary.—D. F. T.

Mercuric oxide; Manufacture of high-grade —. Consortium für Elektrochem. Industrie, Munich. Ger. Pat. 315,657, 1.5.18.

MERCURIC oxide containing much free mercury, such as is obtained by the electrolytic method of production, is treated with solutions of organic substances, preferably such as possess the character of protective colloids. The free mercury then separates rapidly from the suspension.—D. F. T.

Sulphur; Purification of —. J. J. Hood, London. U.S. Pats. (a) 1,331,615 and (n) 1,331,646, 24.2.20. Appl., 13.12.19.

LIQUID sulphur is passed through (a) ignited alumina or (n) ignited magnesia.—L. A. C.

Chlorides; Manufacture of metallic —. W. Hoop, Stockport, and E. Newbery, Manchester. U.S. Pat. 1,331,257, 17.2.20. Appl., 26.8.18.

SEE Eng. Pat. 131,039 of 1918; this J., 1919, 718 A.

Magnesium chloride; Production and utilisation of —. G. H. Bailey and G. W. A. Foster, Assignors to The British Aluminium Co., Ltd., London. U.S. Pat. 1,331,688, 24.2.20. Appl., 29.9.17.

SEE Eng. Pat. 109,996 of 1916; this J., 1917, 1177.

Ammonia; Production of — from cyanamide. B. Brodtkorb, Notodden, Assignor to Norsk Hydro-Elektrisk Kvaestofaktieselskab, Christiania, Norway. U.S. Pat. 1,332,100, 24.2.20. Appl., 4.9.18.

SEE Eng. Pat. 120,034 of 1918; this J., 1919, 322 A.

Alumina; Manufacture of —. E. E. Dutt, Jabalpur, India. U.S. Pats. (a) 1,332,113 and (n) 1,332,115, 24.2.20. Appl., 18.10.18. (n) Renewed 29.11.19.

SEE Eng. Pats. 118,155-6 of 1917 and 120,838 of 1918; this J., 1918, 580 A; 1919, 74 A.

Potassium chloride; Manufacture of —. E. E. Dutt, Jabalpur, India. U.S. Pat. 1,332,114, 24.2.20. Appl., 18.10.18.

SEE Eng. Pat. 116,438 of 1917; this J., 1918, 466 A.

Aluminium chloride residues. U.S. Pat. 1,332,018. See II A.

Purifying substances by sublimation. Ger. Pat. 315,958. See III.

Water vapour. Eng. Pat. 137,547. See XXIII.

VIII.—GLASS; CERAMICS.

Glass batches; Use of mixtures of sulphate and water-glass as a substitute for alkalis in —. L. Springer. Sprechsaal, 1920, 53, 21.

VARIOUS mixtures of sodium sulphate and water-glass have been used as substitutes for other sources of soda in the manufacture of glass. The composition of water-glass varies considerably, but if the material is analysed and the results of the analysis used in calculating the amounts of soda, silica, and sodium sulphate required, the use of water-glass introduces no technical difficulty and increases the fusibility of the batch.—A. B. S.

Crystal glass; Use of specially pure Glauber's salt for the production of high-grade —. K. Killer. Sprechsaal, 1920, 53, 93-94.

GLAUBER'S salt can be used as the sole means of introducing soda into high-grade glass provided the salt is sufficiently pure. The chief impurities are sodium chloride, iron, and free sulphuric acid.

—A. B. S.

Soda-lime silicate glass; Electrical conductivity of —. R. Ambronn. Ann. Physik, 1919, [4], 58, 139-161. Chem. Zentr., 1920, 91, 11, 175-176.

MEASUREMENTS of the electrical conductivity of thirteen different glasses containing only Na₂O, CaO, and SiO₂ showed that the relation between conductivity and temperature can be expressed by a simple exponential equation in which the exponent is the reciprocal of the absolute tempera-

ture, and that the conductivity is zero unless the sum of the calcium and sodium atoms is at least 11 to each 100 atoms of the glass substance; in this connexion 1 atom Na is equivalent to 1 atom Ca.

—L. A. C.

Magnesia bodies. A. Berge. Sprechsaal, 1920, 53, 1—3, 9—11.

CERAMIC wares containing magnesia mature at a lower temperature than others, and the author has endeavoured to ascertain whether the use of magnesia bodies might be a means of saving fuel in the production of pottery. It was found that some of the German magnesium-bearing deposits are useful substitutes for the magnesite imported from Austria. The author examined 27 mixtures of magnesia with quartz, kaolin, and felspar, and describes the results obtained. It is concluded that the addition of magnesia is a suitable means of lowering the maturing point of various bodies without introducing serious technical difficulties. Some clay should be present in order to provide the necessary plasticity, about 10—15% being usually sufficient. Discoloration and blisters occur if the earlier stages of burning are hurried, as vitrification commences before all the carbonaceous matter has burnt out. The lower maturing point should effect a saving in fuel, but unless magnesia of sufficient purity can be obtained at a low price, the cost of the ware will not be reduced by the use of magnesia.—A. B. S.

Fireclay mixtures for high temperatures. R. M. Howe. Blast Furnace and Steel Plant, 1920, 8, 157—160.

VARIOUS materials which are commonly added to mortars made for use at high temperatures are often very harmful. They are added to increase the strength of the mortar at low temperatures and to reduce the shrinkage of the clay or porosity of the mixture. The addition of ground silica or alumina is useful, but Portland cement, lime, asbestos, water-glass, salt, and carborundum all lower the fusing point of fireclays to which they are added. The addition of 15% lowered the fusing point of fireclays to the following extent: Portland cement 320° C., lime 400° C., asbestos 250° C., water-glass 50° C., salt 500° C., and carborundum 20° C. A mixture of fireclay with 10% of asbestos and 10% of water-glass had a fusion point below cone 11 (1350° C.) as compared with 1730° C. for the clay alone. When this mixture was used as a mortar for laying bricks built four courses high and then heated under a load of 11 lb. per sq. in. to 1250° C., a large part of the mortar was fused and squeezed out of the joints. Arches similarly treated collapsed. The principal trouble in the use of fireclay alone as mortar is due to the shrinkage of the clay, and this may be avoided by the addition of a suitable proportion of grog or ground firebrick. Such an addition increases rather than diminishes the heat-resistance of the mortar and gives strong and firm joints even at 1350° C.—A. B. S.

Glazes; Transparent — for ivory and white ware. G. Heinstein. Sprechsaal, 1920, 53, 31—32, 44—46.

THE yellow colour of glazes containing lead oxide, alumina, or quartz is not suitable for ivory and white ware. The yellow tint disappears when the lead oxide is completely replaced by any other metallic oxide, and by replacing only 0.25 equivalent of lead oxide a good ivory glaze may be produced. If a little boric acid is added somewhat less lead may be used. The following three mixtures are recommended:—*Frit*: calcined soda, 15.90, 31.80, 44.70; chalk, 10.01, 20.02, 30.03; red lead, 17.32, 14.42, 57.11; Zettlitz kaolin, 7.76, 44.00,

43.00; quartz, 72.36, 75.38, 75.38; crystallised boric acid, 0.00, 62.03, 62.03; *Mill mix*: frit, 261.29, 289.62, 248.76; Zettlitz kaolin, 18.12, 20.70, 20.70 parts by weight. The first is suitable for ivory and inferior whites, and the second and third are suitable for ivory and good whites. Zinc oxide is the most satisfactory substitute for lead oxide in ivory glazes, but if alkaline-earth compounds must be used magnesia is the best substitute, then lime, strontia, and baryta in the order named. Zinc oxide and magnesia also reduce the tendency of the glaze to craze, except in the presence of boric acid.

—A. B. S.

Devitrification phenomena [in glazes], and their prevention. R. Berg. Sprechsaal, 1920, 53, 19—20.

WHEN a glaze which had been in regular use for earthenware was applied to wall tiles and fired in a wall-tile oven, at the same temperature, for the same length of time and using the same coal as before, it showed marked "blinding," especially near the edges. The defect was found to be due to the absorption of a portion of the glaze by the body, and it was cured by making the glaze more viscous by the addition of 5% of zinc oxide. If the slight opacity produced by zinc oxide is objectionable, the proportion of lime and potash or the total number of RO oxides may be increased.—A. B. S.

PATENTS.

Glass manufacture. B. G. Bealor, Aspinwall, Pa. U.S. Pat. 1,331,796, 24.2.20. Appl., 7.6.17.

AN apparatus for making glass consists of a tank, a glass-drawing well formed of refractory shields within the tank, a pair of movable refractory blocks, provided with fluid conduits and with their opposite faces spaced apart to an increasing distance at the glass-line as the blocks are increasingly immersed. Means are provided for drawing a sheet of glass from between the blocks in a plane at right angles to their faces.—A. B. S.

[Glass] colour-fitter or absorbing screen for artificial light. M. Luckiesh and D. A. Dewey, Cleveland, Ohio, Assignors to General Electric Co. U.S. Pat. 1,331,937, 24.2.20. Appl., 21.8.15. Renewed 29.11.19.

A COLOUR filter for giving the light of artificial illuminants the character of daylight is made of glass containing manganese, copper, and cobalt oxides in the proportion of 1 part of manganese dioxide to 2 parts of copper and cobalt oxides.

—A. B. S.

Quartz and other materials fusible with difficulty; Formation of hollow articles from — by means of an electric arc. Deutsch-Englische Quarzschmelze G.m.b.H., Charlottenburg. Ger. Pat. 315,539, 23.1.18.

AFTER the electrodes have been placed in position and the materials charged into an electric furnace an arc is started by ionisation of the intervening air instead of by means of a combustible conductor, to prevent contamination of the mould by the ash from the conductor.—J. A. C.

Quartz; Melting — by means of carbon resistances. Deutsch-Englische Quarzschmelze G.m.b.H., Charlottenburg. Ger. Pat. 317,421, 31.7.18.

BEFORE melting quartz by means of electrically heated carbon resistances embedded in the same, the resistances are heated for a sufficient time to volatilise impurities present in the quartz. The carbon is protected by a layer of sand or other suitable material, and should not be heated so long that the whole or a greater part of it is converted into graphite.—L. A. C.

[*Tunnel kiln for*] heat treatment of bricks, pottery, and the like. A. C. Ionides, jun., London. Eng. Pat. 139,267, 31.1.19. (Appl. 2470/19.)

Bricks, pottery, etc., are fired in a tunnel kiln divided into sections, the accommodation in each section being proportional to the time required in that stage of the heating or cooling. The goods are heated by passing the products of combustion of a mixture of gas and air through the corresponding part of the kiln. The firing section of the tunnel communicates with three parallel and connected preheating and cooling tunnels. Preheating is effected by radiation from the cooling goods, the cars carrying fresh goods passing along a central track with a track containing cooling goods on each side of it. The gas burners are placed in the side walls of the tunnel and are of the type described in Eng. Pat. 136,296. They may be provided with extended combustion passages (Eng. Pats. 108,701 and 114,223; this J., 1917, 1086; 1918, 229A) placed transversely across the cars, and are supplied with a mixture of gas and air at constantly related pressures, mixing being effected prior to ignition in proportions suitable for complete combustion. Additional tunnels may be provided adjacent to the other tunnels to serve as a drying chamber, which is heated by a directly fired air-heating device at the end of the chamber.—A. B. S.

Porcelain. The British Thomson-Houston Co., Ltd., London. From General Electric Co., Schenectady, N.Y., U.S.A. Eng. Pat. 139,315, 13.3.19. (Appl. 6303/19.)

In the manufacture of porcelain a portion of the free silica usually employed is replaced by zirconia or the oxide of some other rare metal. When a mixture of 45% of clay, 35% of felspar, and 15% of zirconia or zircote cement (containing 81% ZrO_2 and 14% SiO_2) is made into porcelain, the product may be heated to 800° C. and plunged into water without loss of mechanical strength. The modulus of rupture of such zirconium porcelain was found to be 7400 as compared with 4390 for ordinary porcelain. The electrical resistances of both porcelains are alike at ordinary temperatures, but at 286° C. that of zirconium porcelain was found to be 2.43 megohms and that of ordinary porcelain only 0.832 megohm. The zirconium porcelain is less porous than ordinary porcelain.—A. B. S.

Silicon carbide; Manufacture of —. O. Hutchins, Assignor to The Carborundum Co., Niagara Falls, N.Y. U.S. Pat. 1,331,435, 17.2.20. Appl., 15.7.19.

Silicon carbide is made in the usual manner from a furnace charge of silica and carbon, of which the upper portion is richer in silica than the lower portion.—A. B. S.

Enamel for enamelling articles in sheet metal or cast metal. C. Musiol, Brussels. U.S. Pat. 1,332,058, 24.2.20. Appl., 12.6.19.

A AORON-FREE enamel for sheet or cast metal is composed chiefly of a triple silicate of aluminium, sodium, and calcium, mixed with a fluoride.

—A. B. S.

Furnaces. Eng. Pat. 139,276. See X.

IX.—BUILDING MATERIALS.

Portland cement raw mixture; Analysis of —. J. C. Witt. Philippine J. Sci., 1919, 15, 107—120.

THE methods employed for determining the calcium carbonate content of Portland cement raw mixture (on which the proportioning of the materials is usually based) are critically surveyed, attention being drawn to the causes of errors due to the

nature of the constituents themselves and to the various kinds of apparatus and procedure adopted.

—W. E. F. P.

Cement; Effect of fineness of —. D. A. Abrams. Proc. Amer. Soc. for Testing Materials, Vol XIX. Part II., 1919. Bull. 4, Structural Materials Research Lab., Lewis Inst., Chicago.

A LARGE number of experiments have been made on the effect of fineness of grinding of Portland cement on the qualities of the concrete made from it. The experiments covered fifty-one different samples from seven commercial mills, and the cements were ground to four to seven degrees of fineness; the amount of residue left on a No. 200 sieve by the different samples varied from 2 to 43% of the cement. For a given sample of cement, the strength of the concrete increased with the fineness for all proportions of mixture, consistencies of the wet concrete, qualities of the aggregate, and ages of the concrete; but the strength of the concrete does not vary according to the fineness of the cement if different cements are used. The fineness of the cement has a greater effect in increasing the strength of concretes having a small proportion of cement, but the effect decreases with the age of the concrete. An increase in strength of from 2.5 at 7 days to 1.4% at 12 months is shown for each 1% reduction in the residue of the cement on the No. 200 sieve. The hardening of the concrete is thus accelerated by finer grinding of the cement. The density of the concrete and its expansion or contraction when stored in water or air respectively are not dependent on the fineness of the cement. With the usual concrete mixtures containing 20 to 25% of cement, the strength was increased about 1% by an increase of 1% in the cement content, and an increase in the proportion of cement had a greater effect on the strength of poor mixtures than on rich mixtures. The strength of the concrete varied inversely with the amount of water used, and a stronger concrete may be obtained with equal ease of working by using a larger proportion of cement.

—W. F. F.

Slags; Disintegration of blast furnace —. K. Endell. Stahl u. Eisen, 1920, 40, 213—222, 255—262.

CHEMICAL analysis does not give any definite means of distinguishing stable slags from unstable ones, though a high proportion of magnesia and alumina or more than 5% $FeO+MnO$ appears to favour stability. Thin sections of various slags were examined microscopically. As a rule stable slags contain notable quantities of the following groups of minerals distributed in a ground mass of indeterminate composition: melilites, resembling those found in some basalts, with a flocculent and zonal structure, feeble double refraction, refractive index above 1.51, straight extinction, optically monoaxial with negative sign; a second series of melilites with the same general optical characters, but greater refraction due to the introduction of ferrous or manganous oxide; varieties of olivine; opaque skeletons of magnetite or manganese oxides present in slags rich in iron and manganese; fern-like globulites of manganous and ferrous sulphide which appear to be characteristic of stable slags. Unstable slags are largely composed of disintegrating melilites, readily distinguished in polarised light by their grey blue colour and the characteristic appearance of their edges, by their higher degree of refraction and yellowish red interference colours. Variations in structure occur in old slags and in some of a special character. The stability or otherwise of a fresh slag can be predicted with a fair degree of certainty by micro-petrographic investigation, as stable slags do not contain disintegrating melilites after being exposed to the weather for two or three weeks.

Unstable melilites which do not disintegrate on weathering are formed by very rapid cooling and occur in the outer crust of large blocks of slag and in slag which has been allowed to flow through troughs. This is due to the fact (previously established by the author) that ferrous and manganous oxides rapidly diffuse from the molten interior to the cooler exterior of a silicious mass, thus enriching the latter and making it more stable than the former. The more slowly cooled interiors of large blocks frequently contain altered melilites which eventually cause the slag to fall to powder. On reheating for 48 hours at 1200° C. stable slags are not affected, but unstable ones show a large increase in the number of fragments of melilite, the increase being roughly proportional to the rapidity with which the slag was originally cooled. The changes effected by reheating are only visible in thin sections under the microscope. An increase in the size of the crystalline grains also occurs. An examination of a series of synthetic basic slags corresponding to those obtained in the Thomas and Gilchrist process of steel manufacture showed that the disintegration of slags is not likely to be due to calcium orthosilicate, but to mixed crystals of calcium aluminosilicates (melilites); and that the presence of manganous oxide or a mixture of manganous and ferric oxides in a slag increases its stability. Synthetic basic slags containing 11–13% Al_2O_3 and 3–4% Fe_2O_3 and/or MnO disintegrated readily, but those containing 17% Al_2O_3 were more stable, and those containing 21–22% Al_2O_3 showed no signs of disintegration. Similarly, synthetic slags containing 10% or more MgO were quite stable. Any disintegration likely to occur in large masses of slag takes place within the first fortnight. Disintegration is not primarily due to the chemical composition of the slag, but to its physical structure. Slags which are more than six weeks old and have shown no obvious signs of disintegration may safely be used as aggregate for concrete. The metastable constituents found in some slags are highly stable under ordinary atmospheric conditions.—A. B. S.

Light-weight artificial stone from blast-furnace slag; Schol process for the manufacture of —.

A. Guttmann. Stahl u. Eisen, 1920, 40, 326–331. The blast-furnace slag used as raw material should contain 35–38% SiO_2 and not less than 47–44% CaO . Manganese and magnesia should be present in small proportion only. The slag is prepared in a light and highly porous condition by projecting the molten material in a horizontal direction into a limited quantity of water so that the process is aided by the development of steam. The spongy mass has apparent specific gravity 0.14–0.22, and forms a serviceable insulator against heat and sound. It is crushed, sifted, and the portion between 3.5 and 12 mm.-mesh size used as basis. The moistened fines are mixed with quicklime in proportion 3:1. The latter is thereby slaked and more dry fines are added to bring up the proportion to 85%. This constitutes the cementing material. One part of cement and six parts of the granular basis are mixed wet and the product pressed into brick form in moulds. The bricks are subjected to steam heat at 60°–80° C. for 6–8 hrs., whereby considerable hardening occurs owing to the hydraulic properties of the mixture. The hardening continues when they are stored for a month in the open. This may be accelerated by a hardening under high-pressure steam.—H. J. H.

Refuse destruction. Grempe. See XIXb.

PATENTS.

Artificial stone or slabs of marble-like appearance; Production of —. H. Grönroos, Copenhagen. Eng. Pat. 138,799, 4.6.19. (Appl. 14,182/19.)

A moist mixture of equal parts of powdered porce-

lain or pottery, pipeclay, and glass-refuse is compressed in a mould under a pressure up to 2850 lb. per sq. in., and the resulting slabs are heated to 900°–1200° C. for 15 mins. so as to fuse the glass. When a glossy surface is required, a layer of crushed glass refuse is placed in the bottom of the mould prior to filling it with the mixture.—A. B. S.

Thermal insulating bodies; Manufacture of —.

H. J. C. Forrester, Birmingham. From International Isolations Kompani A./S.-Ikas, Christiania. Eng. Pat. 139,318, 15.3.19. (Appl. 6522/19.)

A HEAT-INSULATING material is made by mixing 2 parts of dried and finely ground "moler" (diatomaceous earth) with 3 parts of a granular or gritty substance such as dressed cork, peat, sawdust, etc., which has previously been heated to 150°–200° C. to cause it to expand and drive out part of the air and so prevent damage to the moulded pieces later. Water in small quantities is added slowly to the mixture with continual stirring until it attains a dough-like consistency, and the mass is then moulded, dried slowly, heated slowly to 250° C. and afterwards to about 1000° C. The final product has sp. gr. about 0.3.—A. B. S.

[Plaster] mixture; Plastic — and process for making the same. M. K. Armstrong, Winnetka, Ill. U.S. Pat. 1,331,554, 24.2.20. Appl., 2.7.19.

A PLASTER is made by deflocculating clay or clay soil in a fluid in the presence of an electrolyte and adding finely ground calcined gypsum.—A. B. S.

Kilns for burning cement etc. Ger. Pat. 315,775. — Sec I.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Ore-oxygen gasified directly or indirectly in the blast-furnace; Method for calculating the proportion of —. H. Thaler. Stahl u. Eisen, 1920, 40, 317–321.

THE reactions of oxygen in the iron blast-furnace are of three types:—(1) $\text{C} + \text{O} = \text{CO}$, (2) $\text{MO} + \text{C} = \text{M} + \text{CO}$, and (3) $\text{MO} + \text{CO} = \text{M} + \text{CO}_2$. Reaction 2 may be termed "direct" reduction and reaction 3, "indirect" reduction of the ore. If the material balance sheet of the furnace operation is known it is possible to calculate to what extent the direct and indirect processes contribute to the production of iron from its ore, and thus form an opinion as to the efficiency of operation. Indirect reduction corresponds to greater thermal economy and represents the ideal, although in normal practice only 60–65% of the reduction is of this type. In the results of eight calculations the lowest figure was 36.4%, and the highest 69.4%. When the actual CO content of the blast-furnace gas exceeds that theoretically obtainable from the blast alone, the "direct" gasified ore-oxygen equals three-fourths of the total gasified ore-oxygen plus one-fourth of the difference between the oxygen content of the CO actually in the gas and that obtainable from the blast alone, minus three-fourths of the oxygen content of the difference between the actual CO_2 content of the blast-furnace gas and that in the charge. If the CO content of the gas is less than that theoretically obtainable from the blast, the "indirect" gasified oxygen equals one-half the sum of the total gasified ore-oxygen and the difference between the oxygen content of the CO obtainable from the blast and that actually present in the gas.—H. J. H.

Blast furnaces; Comparison of electric and coke-fired —. M. Guédra. Rev. Mét., 1919, 17, 13–15.

In electric blast-furnaces at Trollhättan the heat

expended in obtaining 1000 kilos. of pig iron was 2,155,262 cals. The author's trials on coke-fired blast furnaces of 90 tons capacity using 1100 kilos. of coke per ton showed that 3,927,130 cals. was required to produce 1000 kilos. of pig. Thus there is a difference of 1,771,863 cals. in favour of the electric furnace, although only 60–70% of the electric energy was utilised. The calorific value of 1 kilo. of gas from the electric blast furnace is 2257 cals., against 638 for the coke-fired furnace. On account of the greater quantity of gas produced in the latter the total calories per ton of cast iron is 3,582,370, against 1,665,485 for the electric furnace, but a large part of the gas produced in the coke furnace is used to heat the blast to about 720° C.—T. H. Bu.

Coke-oven gas; Heating of a Martin furnace with cold —. F. Springorum. *Stahl u. Eisen*, 1920, 40, 9–13.

Gas, low in sulphur and with a high hydrogen content, is brought directly from the coke ovens and introduced at atmospheric temperature along with a blast of air into the furnace by means of tuyères. The tuyères are situated at either end of the furnace, the direction of the gas being regulated by means of reversible valves fitted with water-seals. The gas burns with a scarcely visible flame, giving a reducing atmosphere over the bath of metal. Tests of a few months' duration were carried out on two Martin furnaces of 30 and 100 metric tons capacity respectively, and the output and quality of the steel compared favourably with the output and quality of the steel from the same furnaces heated by means of ordinary producer gas.—J. W. D.

Heating metallurgical furnaces with heavy oil. A. Gouvy. *Chim. et Ind.*, 1920, 3, 143–156.

PETROLEUM in Russia during the war with the use of petroleum residue (mazout) has shown that heavy oil can be used with advantage for metallurgical furnaces. For the heat treatment of shells and for metallurgical work in general it is preferable to atomise the oil by compressed air and to avoid the use of steam. A cutting blowpipe flame which would burn the surface of the metal is avoided by providing a combustion chamber in front of the treating chamber so that the latter is filled with a tranquil flow of hot gas. The complete combustion of the oil is ensured by providing in the combustion chamber a bridge wall which becomes incandescent. For boiler firing the oil is atomised by steam into a brick-lined combustion chamber.—W. H. C.

Metallurgical processes in the acid and basic converter on the basis of spectro-analytical observations. L. C. Glaser. *Stahl u. Eisen*, 1919, 40, 73–80, 111–117, 188–191.

THE application of the spectroscope to the study of the converter flame yields additional information to that derived from its visual appearance, and besides portraying the purely metallurgical processes enables conclusions to be drawn as to the temperature and fluidity of the bath. Results of spectroanalytical observations on the acid process show that it is possible to ascertain when a desired carbon content has been reached and to control the composition of the metal bath. This results in a saving of additions. The heats can be satisfactorily tapped according to the spectrum. The spectroscope as applied to small Bessemer working is of value in determining the time of ignition of the carbon and in controlling the composition of the bath when making special material and producing cast ingot iron. In the case of the basic process it is possible by ascertaining the point of lowest carbon content to determine the degree of dephosphorisation. In both processes it is possible to judge the

progress of the blow, and to measure and regulate the necessary additions of scrap.—T. H. Bu.

Chrome-nickel steel; Influence of deoxidation and duration of solidification on electro —. E. Kothny. *Stahl u. Eisen*, 1920, 40, 41–45.

CHROME-NICKEL steel (C 0.40, Mn 0.42, Si 0.22, P 0.030, S 0.015, Cr 0.18, Ni 2.51) was prepared in an Héroult furnace and just sufficient ferromanganese and ferrosilicon added to remove red-shortness. The bath was then treated with two oxidising slags and half the charge at once poured. More ferromanganese was added to the steel remaining in the furnace, and, after half an hour, the second half of the charge was poured. Each half was cast into 3 ingots, the first portion being poured into a well heated sand mould, the second portion into an iron mould previously heated to 400° C., and the remainder into an iron mould at 50° C. The time taken for solidification of the two "sand" ingots was 35 min., and that for all four ingots cast in iron moulds was 13–15 min. The "sand" ingots solidified suddenly throughout, and showed a coarsely crystalline uniform structure when broken. The iron-mould ingots solidified progressively from the outside towards the centre and gave corresponding fractures. The fractures gave no indication of the difference in deoxidation treatment. The ingots were rolled down from 230² to 110² sq. mm. cross-section, and annealed for 10 hours at 700° C. Tensile and impact test-pieces were then prepared, some of which were tested without further treatment, and others after being quenched in water from 820° C. and reheated for 15 min. at 600° C. and at 250° C. respectively. The results showed that the quickly solidified, well deoxidised material corresponded in every way to commercial standards, but that incomplete deoxidation gave rise to a granular structure, and slow solidification to a fibrous structure in the heat-treated steels.—T. St.

Gold ore; An unusual process in the treatment of —. B. L. Gardiner. *J. Chem. Met. Soc.*, S. Africa, 1920, 20, 109–117.

THE ore consists of granulated quartz mixed with oxides and hydroxides of iron, and containing also a certain amount of iron and magnesium sulphates. Direct cyaniding extracted only about two-thirds of the gold, the remainder being so entangled in, and coated with oxidised iron compounds that finer grinding, preliminary acid washes, or the use of stronger solutions of cyanide did not improve the extraction. By baking the ore at 200°–300° C., however, iron hydroxides were decomposed with the formation of a porous mass of ferric oxide from which the gold was readily leached, an extraction of 90% being obtained in the laboratory. To carry out the process on the large scale a rotary dryer was used. Only that part of the ore which passes a screen of 1½ inch aperture is treated in the dryer, the remainder being treated in the usual way. The ore, as it comes from the dryer, is mixed with slaked lime (10 lb. per ton) and dumped into the sand leaching vats. By working in this manner the extraction was increased from about 63% to 86% at a cost of about 2s. 2d. per ton.—A. R. P.

Gold-silver alloys; Action of aqua regia on — in the presence of ammonium salts. W. B. Pollard. *Chem. Soc. Trans.*, 1920, 117, 99–103.

GOLD-SILVER alloys after a short time resist the action of aqua regia owing to the formation of a protective coating of silver chloride on the surface of the metal, but complete solution may be readily effected in the presence of ammonium chloride, or ammonium chloride and nitrate. With 5 grms. of each salt and 5–10 c.c. of aqua regia 0.5–1.0 gm. of alloy can be easily and quickly dissolved. On diluting the solution with water the silver chloride

is completely precipitated. A secondary reaction occurs during solution whereby some of the ammonia is oxidised to nitrogen, and also in some cases the formation of purplish-brown crystals, having the composition $3\text{AgCl}\cdot 4\text{AuCl}_3\cdot 8\text{NH}_4\text{Cl}$, was observed. This double salt is immediately decomposed by water with precipitation of silver chloride.

—G. F. M.

Powdered coal; Utilisation of — for heating copper smelting furnaces. G. de Venancourt. Rev. Mét., 1920, 17, 2—12.

THE theoretical advantages attending the use of pulverised fuel are found to be confirmed in practice in the case of reverberatory copper-smelting furnaces. The feed hoppers for the ore charges are best placed in double line on either side of the furnace. The material in the furnace is then continuously exposed to the gases of combustion and protects the sides from the heat. The output of the furnaces is increased even with inferior grades of fuel. Particulars of trials on furnaces of the water-jacket type are given, the furnaces being found to retain their heat well. Trials made by the Tennessee Copper Co. showed that a smaller percentage of coal was required than when coke was used for heating by previous methods. In a new type of furnace the pulverised coal was introduced independently of the air. With 50% coke in the charge the results were satisfactory, but not entirely when using powdered coal alone. Nearly all coals, even lignites, may be employed, but they must be dry, very fine, and contain a certain quantity of volatile material.—T. H. Bu.

Copper; Standardisation of —. O. Nielsen. Metall u. Erz, 1920, 17, 4—10.

THE author reviews previous proposals for standardising copper specifications, and also the current specifications of many railways etc. He recommends that the properties specified should be confined to those which come into question in the service for which the copper is required. Thus a specification stating the conductivity requirements should suffice for copper for electrotechnical purposes, except in special cases when it would be necessary also to specify certain mechanical properties. Only in the case of copper required for the preparation of alloys is it necessary or desirable to define the chemical composition.—T. St.

Zinc furnace; The combustion chamber of the Hegeler —. O. Mühlhaeuser. Metall u. Erz, 1920, 17, 1—4.

THE furnace is divided into two similar portions by a dividing wall which runs the whole length of the furnace and serves to support the back ends of the muffles in both portions. Each portion is divided into 18 sections, and each section contains 24 muffles in six rows of four, one above the other. Producer-gas is supplied at one end of the furnace to both portions equally, and passes forward through all the sections on either side of the central wall, air being supplied separately to each section in amount sufficient to consume an aliquot part of the gas. The air may be preheated, and the exhaust gases on leaving the last sections may be utilised for this purpose, or for heating boilers, or both. Baffle arrangements are provided between the sections to mix the gases in the gas stream and so ensure uniform heating of all the muffles.—T. St.

Zinc retorts; Permeability of —. O. Mühlhaeuser. Metall u. Erz, 1919, 16, 191—195, 219—233.

IN a series of experiments, each lasting several weeks, carried out to determine the permeability of the retorts during zinc distillation, daily determinations were made of the pressure in the furnace and in the retort, and of the nitrogen content of

the retort gases. From the tabulated results the author draws the following conclusions. The permeability of a new retort depends on the porosity of the material and the nature, length, depth, and number of small fissures developed during the drying, baking, and heating up in the furnace. It steadily diminishes during the first fortnight of use, and eventually attains an almost constant value which varies for each retort in the furnace. This is attributed to the gradual filling up of the cracks by the glazing of the outside of the retort by the heat, and by molten slag dropping from the roof of the furnace, as well as to the alteration in the chemical nature of the clay (transformation of the alumina and silica into zinc spinel, tridymite, and sillimanite). The permeability sometimes increases abnormally for a day or two and then falls back to its original constant value. This may be due to overheating of the retort, thereby loosening the glaze, or making it more liquid and, therefore, less able to withstand the pressure of the gases, or it may be due to the development of new fissures which are then gradually filled up from either the inside or the outside of the retort. The former condition may be counterbalanced by an increase of slag within the retort or by the oxidising action of the flame causing the separation of ferrite from the ferrous silicate and thus rendering it more viscous.

—A. R. P.

Zinc; Polishing and etching of — for micro-examination. H. H. Hayes. Inst. of Metals, 12.4.20. [Advance copy.] 1 page.

To study the effects of different methods of annealing zinc the following rapid method of polishing and etching was developed. After polishing on standard French emery papers the specimen is rubbed on chamois leather smeared with "Globe" polish and finally with the tip of the finger, then cleaned with water-free ether and dried in a hot air blast or on linen. The micro-section is held in zinc-tipped forceps and immersed in fuming nitric acid (sp. gr. 1.5) for 1 min. and immediately rinsed in water and dried in a hot air blast.—T. H. Bu.

Zinc; Solubility of — in cyanide solutions. H. A. White. J. Chem. Met. Soc., S. Afr., 1919, 20, 97—101.

WEIGHED and measured strips of thin sheet zinc were subjected to the action of solutions of potassium cyanide and calcium hydroxide for various periods, under conditions similar to those obtaining in practice during the precipitation of gold in extractor boxes; and the amounts of zinc dissolved per unit of area exposed were calculated. The consumption of zinc by cyanide in the presence or absence of free alkalinity, was greatly reduced by de-aerating the solutions; and the amount dissolved by aerated cyanide solutions was much greater when the metal was in contact with carbon than when in contact with lead. In aerated solutions containing 0.03% KCN, the degree of alkalinity had little effect on the rate of solution of zinc, whether the latter was freely suspended or in contact with lead. Alkalinity equivalent to 0.03% NaOH did not interfere with the precipitation of gold from cyanide solutions; but, in the absence of cyanide, practically no zinc was dissolved by lime water of strength equivalent to 0.04% NaOH. From de-aerated solutions of potassium aurocyanide no gold was precipitated by zinc, alone or in contact with lead, in the absence of free cyanide; and the best precipitation of gold was obtained by the use of a zinc-lead couple in solutions containing free cyanide. It is concluded that close regulation of cyanide strength, alkalinity, and aeration of the solution is necessary to keep the consumption of zinc within reasonable limits; and that a zinc consumption corresponding even approximately to the amount of gold precipitated is not attainable.—W. E. F. P.

Brass; Casting in high tensile —. N. J. Maclean. Inst. of Metals, 12.1.20 [Advance copy.] 18 pages.

Is experiments to produce an alloy which when cast in sand moulds would give results 50% better than Admiralty gun-metal, additions of manganese, vanadium, titanium, and cadmium, and later nickel and iron, to brass were tried. The alloy finally chosen was Cu 60%, Zn 31%, Ni 3%, Fe 2%, Pb 0.25%, Mn 0.5%, phosphor-tin 0.25%. A special hardener consisted of Cu 75%, Ni 15%, Fe 10%, which was melted in a crucible and granulated by blowing a stream into a tank of water. Special precautions were taken to keep the zinc content of the alloy and the pouring temperature constant. Scrap metal was melted into ingots, which were analysed. The charge was then made up of scrap, new metal, a quantity of hardener, and zinc. The alloy gave sound castings which, cast in sand moulds, had a breaking strength of 20 tons per sq. in., an elastic strength of 10 tons per sq. in., and an elongation of 15% on 2 ins.—T. H. Bu.

Aluminium; Action of hard waters on —. R. Seligman and P. Williams. Inst. of Metals, 11.4.20. [Advance copy.] 25 pages.

OXYGEN is absolutely essential to attack on aluminium by water. The presence of impurities in the aluminium is not on the whole detrimental, the electro-positive metals serving to restrain corrosion. Hard worked metal is attacked relatively easily, but corrosion does not predominate on hardened spots. The composition of the water is the chief factor, pitting being definitely dependent on the simultaneous presence of the chlorides and bicarbonates of either calcium or sodium and probably other metals. Corrosion is always accentuated where the products of corrosion can accumulate. The formation of blisters may be due to the presence of cavities in the aluminium. Treatment of the aluminium to augment the protective film of oxide normally covering the surface gave only slightly enhanced resistance, but chemical treatment of tap water is capable of removing its property of pitting aluminium. Additions of lime water and of small quantities of nitrates or chromates are effective. Effective protection can be obtained by use of an outside source of electricity or by contact with a more electro-positive metal such as tin or zinc.—T. H. Bu.

Aluminium; Protective coating for —. L. von Grotthuss. Metall u. Erz, 1920, 17, 39—40.

ALUMINIUM sheets are given a protective coating by making them the cathodes in a bath, maintained at 60°—65° C., consisting of a solution of a sulpho-compound of molybdenum, zinc sheets forming the anodes. After a short time the aluminium becomes coated with a dark-brown to black deposit which withstands rolling, bending, etc. Sheets thus protected are unaffected by tap- or sea-water.—T. St.

Lead sulphate; The problem of reducing —. F. N. Flynn. Eng. and Min. J., 1920, 109, 487—489.

THE usual method of working up fume containing lead sulphate by extraction of the zinc with sulphuric acid and smelting of the residue in the blast furnace after briquetting, presents considerable difficulties, and the author proposes the following wet method. The wet material is dried, and any acid evolved is recovered. The dry residue is extracted with a nearly saturated solution of sodium, calcium, or magnesium chloride containing a certain amount of ferrous chloride or other easily oxidisable salt to act as depolariser. After settling the clear liquor, containing approximately 1% of lead, is decanted and electrolysed using a lead or iron cathode and an insoluble anode (e.g., graphite,

magnetite, duriron). The amount of ferrous chloride present must be greater than that equivalent to the amount of lead precipitated during the operation. When the electrolyte contains only 0.01% of lead the electrolysis is stopped and the solution is treated with sulphur dioxide or calcium sulphite to reduce ferric chloride, and then agitated with lime to precipitate the accumulated sulphuric acid and thus preserve the solvent action of the solution for lead sulphate. This purified solution is used over again in the process. The lead is precipitated as an adherent deposit on the cathode, as nodules, and as powder. The cathodes are washed, melted and cast into pigs; the nodules and powder, after washing, are melted in a dross reduction furnace.—A. R. P.

Dross losses in metal melting. E. H. Schulz and H. Winkler. Metall u. Erz, 1919, 16, 215—218.

THE amount of dross produced on melting brass increases with the zinc content of the alloy and also with the fineness and degree of oxidation of, and amount of impurities in, the scrap-metal added. Thus, on melting a 2½-ton charge consisting of 67% scrap and 33% new metal a loss of 4% was obtained, whereas when the mixture consisted of 80% scrap and 20% new metal, the loss was 11% of the charge, and the relative amount of copper lost was also greater. An alloy of zinc with 2.89% of copper and 3.75% of aluminium was made by melting the latter two metals separately in small furnaces, pouring them into the molten zinc contained in crucibles, and casting the alloy in iron moulds. After removing any prills of metal from the dross and allowing for this, 0.51% of the zinc, 11.80% of the copper, and 12.20% of the aluminium were lost as dross. The total metal loss calculated on the final alloy was 1.4%, of which that due to zinc was 0.48%, to copper 0.39%, and to aluminium 0.53%. Two charges of zinc-aluminium (2.5% Al) alloy scrap were melted down, the first, consisting of 50% finely divided and strongly oxidised material and 50% of coarser and cleaner scrap, gave 69% of clean alloy; the second consisting entirely of the dirty scrap, yielded only 40.5% of alloy. These considerable losses were due to the large amount of oxidised material present holding up the metal so that it was necessary to employ a high temperature in melting with consequent volatilisation and oxidation of much of the zinc. Similar losses were obtained in melting scrap zinc; they varied from 10% to 26%, according to the amount of finely divided and dirty scrap present. Scrap aluminium is usually melted in a reverberatory, and the difficulty of separating metal and dross is much greater on account of the low specific gravity of the former. Hence the losses to be expected depend to a much larger extent on the amount of dirty and oxidised material present, and, while a loss of only 5% may be experienced with careful working on sheet or other coarse form of metal, with very fine, and oily turnings, the loss is likely to be over 20%.—A. R. P.

Welding; Comparison of the different methods of —. P. Schimpke. Metall, 1919, 243—245. Chem. Zentr., 1920, 91, II., 81.

WELDS made by the use of a coke fire or water-gas give the strongest joints. The use of hydrogen is economical only for sheets up to 5 mm. thickness. Acetylene-welding is superior to all other methods for sheets, whilst for thicker material (up to 12 mm.) water-gas is superior. Benzol-welding costs 50% more than acetylene welding, while the use of coal-gas is about as expensive as that of hydrogen. Welding in a coke fire is the most costly method. Autogenous and electrical welds are considerably stronger than soldered joints.—A. R. P.

Vapour pressure of lead chloride. Eastman and Duschak. See VII.

Blast-furnace slag. Guttman. See IX.

Blast-furnace slag. Endell. See IX.

PATENTS.

Steel; Rail —. C. H. Wills, Detroit, Mich. U.S. Pat. 1,331,175, 17.2.20. Appl., 6.7.17.

STEEL of the following composition C 0.35—0.055%, P not to exceed 0.1%, Si not to exceed 0.27%, Mn 0.7—1.10%, and Me trace—1.0%, is quenched from 1500°—1650° F. (815°—900° C.) and drawn at 1000°—1200° F. (540°—650° C.).—J. W. D.

Nickel-manganese steel; Process for improving non-magnetic —. F. Theubert, Berlin-Weissensee. Ger. Pat. 310,101, 19.5.18.

By the addition of 1—6% of silicon to nickel-manganese steel the elastic limit and specific electrical resistance are raised and the alloy can be more easily worked, while its non-magnetic character remains unaltered.—A. R. P.

Steel alloy. Stahlwerke R. Lindenberg A.-G., Remscheid-Hastent. Ger. Pat. 316,149, 18.7.15. Addition to 299,192.

A HARD and highly tenacious steel contains 0.1% less carbon than that described in the chief patent (this J., 1919, 908 A); if a case-hardened steel is required the carbon content is 0.08—0.15%.

—L. A. C.

Nickel-plated iron and steel objects; Process for preventing — from rusting. E. Kronenberg, sen., Seligen. Ger. Pat. 316,053, 2.12.17.

IRON or steel objects are heated in oil at 200° C., cooled, freed completely on the surface from oil, covered with a thin coating of copper, nickel-plated in a citric acid nickel bath using a weak current, and dried at 200° C.—L. A. C.

Heat treatment of metals; Furnaces for —. H. W. Brighton and P. Peakman, Manchester. Eng. Pat. 138,981, 4.2.19. (Appl. 2737/19.)

A CLOSED muffle heated externally by means of producer gas is combined with a generator for supplying gas to the interior of the muffle. The gas leaving the muffle is used to assist in heating it externally. Partially carbonised fuel from the generator is conveyed to the producer, where carbonisation is completed.—J. W. D.

Furnaces for hardening or tempering steel tools or for heating or annealing metals, glass, pottery, or the like. J. R. C. August, Halifax. Eng. Pat. 139,276, 12.2.19. (Appl. 3410/19.)

A GAS-fired furnace in which the combustion chamber has inclined sides, a roof with a rounded corner or baffle between the gas supply jet and the combustion chamber orifice, and a floor which gradually slopes upwards from the end where the gas and air are delivered to the end where they enter the furnace. The pipes for supplying the gas and air pass back and forth through a series of chambers connected by spaced conduits giving a good heating surface, these chambers being cast inside a hood arranged on the top of the furnace and heated by the products of combustion from the furnace.

—J. W. D.

Conglomerates of fine ores, iron filings, flue dust, purple ore, and the like; Solidifying —. C. Giesecke, Harzburg, Germany. Eng. Pat. 139,229, 4.10.16 (Appl. 3369/18.)

BLOCKS or masses of fine ore or the like formed in a press are fed evenly, together with finely-divided fuel into a shaft furnace, wherein they are submitted to such a degree of heating and subsequent cooling and are caused to pass through the various

zones of the furnace with such rapidity that the sintering heat does not extend to the core of the agglomerated masses, whereby a conglomerate is produced having a well-sintered crust with a less sintered or completely unsintered and consequently easily reducible core.—J. W. D.

Alumino-thermic method; Manufacture of metals and alloys by the —. British Thomson-Houston Co., Ltd., London. From General Electric Co., Schenectady, N.Y., U.S.A. Eng. Pat. 139,247, 3.1.19. (Appl. 212/19.)

FERRO-zirconium or ferro-silico-zirconium is produced by forming briquettes of the oxides of the metals, intimately mixed with finely-divided aluminium, a portion at least of which is in the flaky condition, and then igniting the briquettes.

—J. W. D.

[*Aluminium*] *solder.* Miralite, Ltd., and W. G. Grever, London. Eng. Pat. 139,402, 21.7.19. (Appl. 18,141/19.)

A SOLDER for soldering aluminium and alloys rich in aluminium consists of Al 2.4, P 2.4, Zn 26.2, Sn 69%.—T. St.

Flotation apparatus and process. J. M. Callow, Salt Lake City, Utah, and H. P. Corliss, Pittsburgh, Pa., Assignors to Metals Recovery Co., Augusta, Me. U.S. Pat. 1,331,238, 17.2.20. Appl., 3.7.19.

IN a flotation apparatus the air is delivered through a porous layer composed of a mixture of a synthetic resin and a granular substance moulded into form and hardened by the application of heat.—T. St.

Copper; Process for hardening —. D. T. Beckham, Geneva, Tex. U.S. Pat. 1,331,479, 24.2.20. Appl., 13.3.19.

COPPER is hardened by heating it to a high temperature and then subjecting it to continual pressure until cold.—T. St.

Smelting-furnaces; Open-side water-jacket for —. A. B. Carstens, Monterey, Mexico, Assignor to The American Metal Co., Ltd., New York. U.S. Pat. 1,331,803, 24.2.20. Appl., 1.10.18.

A WATER-JACKET for a metallurgical furnace, forming, for example, the bosh wall of a blast furnace, consists of a series of superposed separable cells arranged to overflow successively from one to the other and from the top to the bottom of the jacket.

—T. St.

Furnace; Open-hearth —. F. Carter, Minneapolis, Minn., and D. McLain, Milwaukee, Wis. U.S. Pat. 1,331,804, 24.2.20. Appl. 12.3.17.

A NARROW furnace chamber with a high roof and contracted end portions is provided with vertical air-intake ports extending the full width of the contracted portions, and inclined oil burners, which pass through the end walls of the furnace chamber and extend across the path of air from the vertical ports.—T. St.

Roasting furnace. H. Alinder, Assignor to Allis-Chalmers Manufacturing Co., Milwaukee, Wis. U.S. Pat. 1,332,152, 24.2.20. Appl., 2.4.17.

IN a furnace with superposed hearths the rabble arms carried by the central vertical shaft are detachable, and when in position are secured by a projecting locking device.—T. St.

Roasting zinc-blende or other material containing sulphur; Process and furnace for —. Fellner und Ziegler, and W. von Escher, Frankfurt. Ger. Pat. 312,383, 21.12.17.

THE roasting gases are led backwards over a fresh charge of ore in order to preheat and ignite it,

after which the gases pass forward again thereby completing the roasting. The operation is carried out in a furnace consisting of two rotating tubes, one above the other, the lower tube being progressively widened towards the discharging end. The air is led in at the charging end of the tube in which the final roasting is carried out, and prevented from overflowing into the other tube. At the discharging end of the former tube the hot gases pass into the second tube and ignite the charge therein. Working in this manner it is claimed that no external heat is necessary, as the final roasting is carried out in the presence of the hottest gases.—A. R. P.

Martin furnace; Process for substituting coke or the like for pig iron in operating a —. Thyssen und Co. A.-G., and F. Thomas, Mülheim. Ger. Pat. 316,027, 21.1.16.

COKE covered with a protective layer of milk of lime, calcium sulphate paste, water-glass, or the like, is added to the charge of a Martin furnace instead of pig iron; the covering material melts, rises with the slag, and allows the coke gradually to come in contact with the molten iron.—L. A. C.

Casting metal; Method of —. C. McKnight, jun., Sewickley, Pa. U.S. Pat. 1,331,816, 24.2.20. Appl., 20.12.19.

To reduce porosity and inclusions at or near the surfaces of cast ferrous alloys, the moulds are coated with a sufficient quantity of a material as will combine with the dissolved gases and metallic oxides in the molten metal without materially affecting the physical properties of the metal.—J. W. D.

Strain-hardened metals; Coated —. W. R. Whitney, Niskayuna, N.Y., Assignor to General Electric Co. U.S. Pat. 1,331,888, 24.2.20. Appl., 15.6.18.

FERROUS metals under strain are prevented from becoming brittle during treatment with a hydrogen-generating agent by coating them with a material impermeable to hydrogen.—J. W. D.

Sodium-lead alloy. W. Stockmeyer, Minden, and H. Hanemann, Charlottenburg. Ger. Pat. 304,244, 25.3.17.

THE alloy consists of lead and sodium with less than 0.5% Cu. For example, an alloy containing 1% Na and 0.2% Cu is more ductile than that containing no copper, and may be drawn hot without breaking.—A. R. P.

Autogenous cutting of metals; Process for the —. Deutsche Wildermann-Werke Chem. Fabr. G.m.b.H., and C. Waldeck, Lülsdorf. Ger. Pat. 307,179, 7.5.18.

THE metal is exposed to the action of a jet of chlorine or a mixture of chlorine and other suitable gases whereby volatile or water-soluble compounds of the metal are formed. Iron, zinc, aluminium, copper, and lead may be cut by the process, which is also suitable for use under water.—A. R. P.

Alloy. K. Hunger, Mülheim. Ger. Pat. 309,243, 6.1.18.

AN alloy suitable for use as bearing metal contains lead and about 5% of a mixture of two parts of zinc and one part of magnesium.—L. A. C.

Tin-plate scrap; Process for detinning —. Chem. Fabr. Buckau, Magdeburg. Ger. Pat. 309,277, 12.1.18.

THE operation is carried out by treating the scrap with undiluted chlorine at 300° C., and removing the vapour of tin tetrachloride from the reaction chamber as fast as it is formed. By this means attack of the iron is avoided.—A. R. P.

Tin; Process for recovering — from waste tin plate or the like. T. A. Eklund, Stockholm. Ger. Pat. 316,111, 20.3.19. Int. Conv., 1.2.19.

SCRAP tin or the like is treated with hydrochloric acid in the presence of an oxidising agent (an alkali nitrate), or tin sludge is dissolved in hydrochloric acid in the presence of an alkali nitrate, and the solution containing quadrivalent tin is used for detinning scrap tin plate, yielding a solution containing divalent tin. The solution obtained in either case is electrolysed, using a current of about 1 volt, an anode of carbon, block tin, or tinned iron, and a cathode of tin, iron, or other metal. Crystalline tin is deposited on the cathode, and the exhausted bath is re-oxidised and used for the treatment of more waste tin.—L. A. C.

Coating metals with lead; Process for —. Montag und Laube, Hannover-Linden. Ger. Pat. 310,176, 14.9.17.

THE metal is treated by the "contact" process to coat it with a thin film of lead which adheres tenaciously. Thicker coatings of lead are then readily deposited on this by electrolysis in the usual way.—A. R. P.

Metallic surfaces; Coating — with lead, tin, zinc, or alloys of the same. Berlin-Burger Eisenwerk A.-G., Berlin. Ger. Pat. 315,712, 17.12.18.

A THIN layer of the coating metal or alloy is first deposited on the metallic surface by electrolytic means, the article is then dipped in a molten bath of metal of the same composition, and is covered with a poor heat conductor to prevent rapid cooling. Iron surfaces are first treated with an acid, and then with a sand blast to remove carbon.—L. A. C.

Metal turnings; Production of briquettes from —. Magnet-Werk G.m.b.H. Eisenach, Eisenach. Ger. Pat. 315,989, 26.4.18.

METAL turnings or the like are compressed with simultaneous heating by means of an electric current.—L. A. C.

Copper baths; Revivifying electrolytic —. Hüttenwerk Niederschöneweide A.-G. vorm. J. F. Ginsberg, Berlin-Niederschöneweide. Ger. Pat. 316,048, 13.12.17.

COPPER anodes containing cuprous oxide, either alone, or with other anodes, are used in an electrolytic copper bath to avoid the necessity for adding copper sulphate to the bath.—L. A. C.

Graphite; Means for enclosing pieces of — in refractory metals. G. Ising and H. Borofski, Brunswick. Ger. Pat. 316,180, 3.10.18.

THE formation of metallic carbides on the addition of pieces of graphite to a molten metal, e.g., iron, to produce a metal having a surface with low friction, is prevented by first coating the graphite with copper or other suitable metal.—L. A. C.

Wrought iron; Mechanical puddling in the manufacture of —. H. Maplethorpe, West Bromwich. Eng. Pat. 139,359, 5.5.19. (Appl. 11,229/19.)

Iron and steel; Manufacture of —. H. B. Clapp, Wimbledon, Assignor to D. R. Blair, London. U.S. Pat. 1,330,846, 17.2.20. Appl., 23.12.18.

SEE Eng. Pat. 134,283 of 1918; this J., 1920, 28 A.

Zinc; Extraction of —. E. S. Berglund, Trollhättan, Assignor to S. Hult, Stockholm, Sweden. U.S. Pat. 1,331,740, 24.2.20. Appl., 12.9.18.

SEE Eng. Pat. 120,549 of 1918; this J., 1919, 467 A.

Open-hearth furnace. J. O. Griggs, Youngstown, Ohio. U.S. Pat. 1,331,989, 24.2.20. Appl., 2.6.19.
SEE Eng. Pat. 127,785 of 1918; this J., 1919, 540 A.

Brass borings, brass turnings, and like material; Separation and extraction of dust and iron from —. W. Sowden, Leeds. U.S. Pat. 1,332,078, 24.2.20. Appl., 20.7.17.
SEE Eng. Pat. 105,174 of 1916; this J., 1917, 556.

Alloys for thermo-couples. Ger. Pat. 306,359.
See XXIII.

XL.—ELECTRO-CHEMISTRY.

PATENTS.

Storage cells; Electric —. C. J. V. Fery, Paris.
Eng. Pat. 124,211, 7.3.19. (Appl. 5706/19.)
Int. Conv., 30.4.17.

In secondary cells of the type in which the electrolyte is incorporated with an absorbent material such as sand, pumice powder, or the like, the negative element is located horizontally at the bottom of the containing vessel, and the positive element is placed vertically and above the negative element. The negative element may be formed by pouring on to the bottom of the containing vessel a paste of litharge and water acidulated with sulphuric acid and embedding therein a leaden wire which is bent to serve as one terminal of the cell. The positive electrode may consist of a leaden cylinder having ribs, between which the active material is introduced, a spirally wound leaden wire serving to retain this material in place. The device eliminates deformation of the positive electrode and prevents sulphating of the negative electrode on open circuit.—J. S. G. T.

Spongy-lead electrodes [for accumulators]; Manufacture of —. E. Noll, Frankfurt. Ger. Pat. 315,592, 17.11.15.

A mixture of a reducible lead compound and a reducing agent such as charcoal or sugar, especially the latter, is heated under pressure. For example, plates composed of a mixture of lead oxide and reducing agent are heated by immersion in a lead bath, and are then cooled without access of air by immersion in water. Plates so made possess a very uniformly porous structure.—J. S. G. T.

Paper for use as dielectric. Eng. Pat. 138,226.
See V..

Washing textiles. Ger. Pat. 315,835. See VI.

Quartz articles. Ger. Pat. 315,539. See VIII.

Melting quartz. Ger. Pat. 317,421. See VIII.

Preserving materials. Eng. Pat. 139,100. See XIXA.

XII.—FATS; OILS; WAXES.

Olive oil; Lipolytic enzymes in —. T. M. Rector.
J. Ind. Eng. Chem., 1920, 12, 156–158.

EMULSIONS of 50 c.c. of olive oil, 4 grms. of gum tragacanth, 2 c.c. of toluene, and 50 c.c. of water, were vigorously shaken and left for periods up to 30 days in a closed flask. In the case of unheated oil the acid value rose progressively from 0.7 to 3.6. The acid value of oil which had been heated to 75° C. rose to 22.8, and that of oil heated to 100° C. to 10.3, whilst after heating to 125°, 150°, and 175° C. the oil showed values of 5.3, 3.1, and 1.7 respectively. The lipolytic enzyme in filtered olive oil retains its activity for several years. The "foots" obtained in the purification of olive oil contains an anti-

hydrolytic enzyme. For example, the acid value of an olive oil, emulsified as described, increased to 8.8 in 25 days, whereas after the addition of unheated "foots" the acid value only rose to 1.4. The effect of this anti-enzyme is nullified by heat.
—C. A. M.

Pine-seed oil. O. von Friedrichs. Svensk Farm. Tidsskr., 1919, 23, 445, 461. Chem.-Zeit., 1920, 44, Rep., 67.

THE fatty oil from the seeds of *Pinus silvestris*, L. is a light, yellow, mobile, nearly odourless oil. A sample had the following characters.—Sp. gr. 0.9307; solidif. pt., -30° C.; $[n]_D^{15} = 1.4799$, $[n]_D^{40} = 1.4714$; acid value 0.92; saponif. value, 190.7; Hehner value, 94.75; Reichert-Meissl value, 0.79; and iodine value (Hanus), 159.2. The oil contained about 0.11% of volatile fatty acids, mainly butyric acid. There was 90.4% of non-volatile fatty acids, 7.5% glycerol, and 1.29% of crude phytosterol (m. pt. 135° C.). The fatty acids consisted of about 3% of solid fatty acids (palmitic acid, with 5% of stearic acid) and 97% of liquid acids (36.2% oleic, 56.2% linolic, and 7.6% linolenic acid).—C. A. M.

Fir-seed oil. O. von Friedrichs. Svensk Farm. Tidsskr., 1919, 23, 500. Chem.-Zeit., 1920, 44, Rep., 67.

THE fatty oil, as obtained by extraction with ether in Sweden from the seeds of *Picea abies*, Karst. (*Pinus abies*, L.) is a pale yellow mobile oil, with a somewhat bitter taste, recalling that of turpentine, and a faintly aromatic odour. A sample had the following characters:—Sp. gr. at 15° C. 0.9295; solidif. pt., -26 C.; $[n]_D^{15} = 1.4801$, $[n]_D^{40} = 1.4718$; acid value, 1.0; saponif. value, 191.8; iodine value (Hanus), 154; Hehner value, 92.65; and Reichert-Meissl value, 1.06. The oil contained 91.6% of non-volatile acids composed of 0.7% of palmitic acid and 99.3% of unsaturated acids (oleic 42.75, linolic 49.55, linolenic 7.7%). It also contained about 0.14% of butyric acid. The phytosterol had the characteristics of sitosterol.—C. A. M.

Marine animal oils; Reactions in the hydrogenation of —. T. Arentz. Tidsskr. for Kemi, 1919, 16, [11–13]. Chem.-Zeit., 1920, 44, Rep., 67.

THE solid fats obtained by the hydrogenation of marine animal oils do not show the same relationships between their physical and chemical constants as do natural fats. The refractive index and sp. gr. are both higher than the anticipated values. This is due to the formation of isomeric compounds of acids of the oleic series, which have a higher m. pt. The relative absorption of hydrogen was greater in the case of C_{16} – C_{18} glycerides than in that of C_{20} – C_{22} glycerides. When the hydrogenation is rapid the hardening process takes place in stages, and relatively unsaturated compounds are present in association with those completely saturated.

—C. A. M.

Yellow-tail fish oil. H. A. Gardner and A. Reilly. Circ. 86, Paint Manufacturers' Assoc., U.S.A., and Nat. Varnish Manufacturers' Assoc. Feb., 1920. 4 pp.

YELLOW-TAIL fish oil is obtained from the *Seriola dorsalis* which is abundant on the Southern and Lower Californian coast. The oil is cloudy, of light colour, faint odour, and unpleasant taste, and has the following characters: Iodine value (Hanus), 177 (filtered oil, 180); saponif. value, 190; acid value, 0.6; sp. gr. 0.9322. On exposure on a glass plate it dried in 105 hours to a fairly firm film as compared with 95 hours for raw linseed oil. With an addition of 10% of lead and manganese linoleate liquid drier it dried in 15 hours as compared with 12 hours for raw linseed oil prepared in the same manner. When mixed with an equal

weight of raw linseed oil, with and without drier, the drying results were about intermediate between those for the two oils separately.—A. de W.

Coconut oil and palm-kernel oil; Solubility of — in alcohol and acetic acid. J. R. N. van Kregten. *Ölten en Vetten*, 1919, 4, 185. *Chem. Zentr.*, 1920, 91, II., 203.

THE solubility of coconut oil and palm kernel oil in alcohol and acetic acid may be employed for estimating the composition of mixtures of these oils, but previous estimations lack uniformity, as fatty acids were not removed. In obtaining the results tabulated below the coconut oil contained 0.032% and the palm-kernel oil 0.060% of fatty acids, calculated on a mol. wt. of 200. Different experiments showed a divergence of only $\pm 1^\circ\text{C}$. The influence of water in the solvent on the critical temperature of solution is shown in the following table; 1 gm. of oil and 5 c.c. of the solvent were employed in each determination:—

Solvent.	Critical temperature of solution.			
	Sp. gr. at 15°/15°C.	Vol. %	Coconut oil.	Palm-kernel oil.
Alcohol ..	0.7942	99.06	20.2°C.	28.7°C.
	0.7981	99.10	30°C.	38.8°C.
	0.8001	98.75	35.5°C.	44.2°C.
	0.8020	98.36	39.5°C.	47.9°C.
Acetic acid. .	Sp. gr. at 15°/4°C.	% Acetic acid.		
	1.0573	99.25	17.9°C.	32.5°C.
	1.0579	99.05	26°C.	41.5°C.
	1.0581	98.85	30.9°C.	46.5°C.

The critical temperatures of solution of various mixtures of the two oils are also given.—L. A. C.

Saponification values [of oils and fats]; Influence of ester transposition in the determination of —. A. M. Pardee and E. E. Reid. *J. Ind. Eng. Chem.*, 1920, 12, 129—133.

TEST saponifications of benzyl formate, benzyl acetate, and methyl benzoate were made in the presence of various alcohols, a tube 1 m. long being used as reflux condenser. The results obtained with ethyl alcohol as medium confirmed Henriques' conclusion (this J., 1898, 673, 853), that in saponification there is transposition of esters, the loss being more pronounced with benzyl acetate, in which case ethyl acetate is formed and greatly increases the volatility, than with methyl benzoate, which yields ethyl benzoate and decreases the volatility. Hence low results may be expected when acetates are saponified in the presence of ethyl alcohol unless effective condensers are used. Inaccurate results are also obtained with isobutyl alcohol, methyl alcohol, or iso-amyl alcohol as medium. The best medium for saponification in general is normal butyl alcohol, which gave excellent results even with benzyl acetate.—C. A. M.

Oils; Action of nitric acid on saponifiable —. L. G. Radcliffe and C. Polychronis. *J. Soc. Dyers and Col.*, 1920, 36, 65—76.

THE action of nitric acid upon oils varies with the nature of the fatty acids which they contain. Saturated fatty acids, such as stearic acid, are but little affected by cold strong nitric acid or by fuming 99% acid, but are oxidised by hot nitric acid, with the formation of isonitrostearic acid and other compounds. Unsaturated fatty acids, such as oleic acid, react very slowly with strong nitric acid in the cold, but when treated with fuming nitric acid, even at a low temperature, yield nitrated products, while at the same time water is formed, probably through the nitration of the

hydroxyl group produced by the action of the nitric acid on the double linkage. Nitration of such acids as oleic acid lowers the iodine value (e.g., from 89.4 to 7), whilst the nitrated product has a definite hydroxyl value. Products free from nitrogen are never obtained by saponification. Nitrated oleic acid is probably a mixture of several substances containing NO_2 or NO groups. The action of nitric acid upon the glycerides of such unsaturated acids is probably of a similar character. Ordinary strong (72%) nitric acid has but little action upon such unsaturated glycerides. The nitration of castor oil, ricinoleic acid, and tricinolein proceeds in a similar manner, the hydroxyl groups forming nitric esters, while a nitrogen derivative is also formed of the nitro-oleic type. For the nitration of saponifiable oils at low temperatures nitric acid of sp. gr. 1.5 to 1.52 is required, although Brightman (this J., 1917, 985) has shown that under certain conditions castor oil can be readily nitrated by means of dilute nitric acid. The use of sulphuric acid with the nitric acid causes the reaction to be more energetic, but does not increase the amount of nitrogen in the products, which are much darker in colour. Sulphonated oils may be readily nitrated by boiling them with dilute (50—60%) nitric acid. Various fish oils contained from 3.19 to 3.43% of nitrogen after nitration, whilst nitrated whale oil contained 3.15% and had iodine value 15.6. Nitrated castor oil can dissolve an appreciable amount of nitric acid and still remain clear.—C. A. M.

Sodium perborate. Trickett. See VII.

Trimethyleneglycol in glycerin. Rojahn. See XXII.

PATENTS.

Oils; Method of hydrogenating —. G. Martin, Manchester. Eng. Pat. 139,239, 27.11.18. (Appl. 19,567/18.)

OIL, etc. is hydrogenated by a continuous process in an apparatus of the type described in Eng. Pat. 129,461/16 (this J., 1919, 645 A). A portion of the oil is preheated, while another portion is mixed in a second tank with a catalyst and then heated to the same temperature as the first portion. The oil from both tanks is mixed and introduced into a reservoir, whence it falls in a thin stream through a series of connected conduits, contained in a steam-heated chest, and encounters a current of hydrogen issuing from perforated pipes within each conduit. The unabsorbed hydrogen rises into the oil reservoir, whence it is pumped back into the gas reservoir, whilst the hydrogenated oil passes through a cooler, and is then forced through a filter by means of a pump.—C. A. M.

Catalyser for effecting hydrogenation and process of making same. Process for producing catalysers for hydrogenation. Process for hydrogenation. Process for producing catalysers and for effecting hydrogenation therewith. B. W. Elder, Los Angeles, Cal., Assignor to The Allbright-Nell Co. U.S. Pats. (A) 1,331,903, (B) 1,331,904, (C) 1,331,905, and (D) 1,331,906, 24.2.20. Appl., 7.6.18.

(A) Block nickel is rendered catalytically active by pulverising it by mechanical means in the presence of a protective liquid. (B) A practically inactive catalytic substance is converted into a highly active catalyst by converting it into a powder by a combined process of rubbing, squeezing, and percussive action. (C) Oil and hydrogen are brought into intimate contact at a suitable temperature with a finely powdered nickel catalyst produced by mechanical means from block nickel. (D) Oil is made to flow over catalytically active abraded nickel surfaces at a suitable temperature and in the presence of hydrogen.—C. A. M.

Lipase; Preparations containing —. R. Willstätter, Munich. Ger. Pat. 316,504, 5.9.18.

STABLE concentrated lipase preparations are obtained by absorbing lipase from an emulsion or suspension of the same by means of kieselguhr, kaolin, tale, barium sulphate, charcoal, or the like.

—L. A. C.

Glycerin from soap lyas; Process for obtaining —. R. Blum and C. Francke, Berlin. Ger. Pat. 310,045, 3.5.18.

SOAP lyas are concentrated by evaporation, and treated with ozone or ozonised air, whereby sulphur compounds are completely oxidised and rendered innocuous, whilst the fatty acids are converted into solid hydroxy-acids which can be readily separated. The ozone treatment may also be applied prior to acidification.—C. A. M.

Galls; Manufacture of stable preparations [detergents] from animal —. O. Lietz, Kiel. Ger. Pat. 315,352, 27.9.16.

ANIMAL galls are treated in ammoniacal or alkaline solution with hydrogen peroxide, with or without the addition of hypochlorites, and the active constituents are precipitated as a hard mass by the addition of acid. The product may either be employed as a washing powder in the solid form mixed with alkaline carbonates, or may be dissolved in alkalis or alkaline salts.—L. A. C.

Detergents; Manufacture of —. G. Bethmann, Frankfurt. Ger. Pat. 316,210, 8.9.17.

A SOLUTION of a zinc or cadmium salt is added to the solution obtained by decomposing glue by means of an alkali or alkaline-earth oxide or hydroxide or a dilute mineral acid. The product, evaporated to a thick liquid or to dryness, lathers well, and may be used as a soap substitute.—L. A. C.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Oil absorption of pigments. H. A. Gardner and R. E. Coleman. Circ. 85, Paint Manufacturers' Assoc., U.S.A., and Nat. Varnish Manufacturers' Assoc. Feb., 1920. 13 pp.

THE oil absorption of a pigment is defined as the amount of oil absorbed by unit weight of pigment when mixed together in a manner independent of mechanical forces or influences developed by actual grinding. The limit of absorption is characterised by that stage at which the surface of each pigment particle becomes thoroughly wetted by the liquid. In determining oil absorptions a medium must be chosen which has little effect in varying the degree of dispersity of the pigment. The air-dried pigment is well shaken in a wide-mouth stoppered bottle to eliminate packed particles, and 20 grms. is then weighed into a small jelly glass, 2½ in. diam. at the top and 3½ in. deep. Raw, well-settled linseed oil is then run on to the centre of the quantity of pigment, either drop by drop or 0.5 c.c. at a time, diminishing the rate of flow as absorption proceeds. The unmoistened portions of the pigment round the sides should be brought on to the oil by means of a stiff-bladed palette knife in order to ensure equal distribution. The small lumps that form should be equally distributed throughout the mass by stirring lightly without using pressure. Oil is then dropped on to the formed balls or lumps of paste and not on to the dry pigment. The end point is judged by the disappearance of all dry pigment and by the paste lump becoming softer and smearing the sides and bottom of the glass on stirring round. The tem-

perature of the ingredients should be maintained at 70°–100° F. (21°–38° C.). The result is returned as c.c. of oil per 100 grms. pigment. The oil absorptions of several common pigments are given. On the basis of the figures obtained the authors state that when similar amounts of thinners are added to pastes derived from unit weights of different pigments, paints of similar consistencies are obtained. (Cf. de Waele, Proc. Oil and Colour Chemists' Assoc., II., 13, 115–118; this J., 1919, 395 R.)

—A. de W.

Ultramarine; Studies on the constitution of —. L. Bock. Z. angew. Chem., 1920, 24, 23–24. (See also this J., 1915, 561.)

FURTHER evidence of the relationship between ultramarines and aluminate-silicates (zeolites) is brought forward. In the ultramarines the sulphur compounds take the place of the combined water in the zeolites. Experiments on the action of ammoniacal silver chloride solution on artificial ultramarines indicate that the silica-rich sulphur-rich and silica-poor sulphur-rich ultramarine blues are definite compounds of formulae, $2(\text{Na}_2\text{O}, \text{Al}_2\text{O}_3, 3\text{SiO}_2)\text{Na}_2\text{S}_4$ and $3(\text{Na}_2\text{O}, \text{Al}_2\text{O}_3, 2\text{SiO}_2)\text{Na}_2\text{S}_4$ respectively. A table is given showing the known ultramarines and their relationship to natural and artificial silicates.—J. K.

Bauxite as a pigment. A. Cobenzl. Farben-Zeit., 1919, 25, 20. Chem. Zentr., 1920, 91, II., 86.

THE blue colour of bauxite is due to ferrous sulphide in an easily oxidisable colloidal form, and the aluminium hydroxide itself takes no part in the production of the colour. A similar colour may be observed in barium sulphate that has stood for a long time in moist packages. In this case the organic matter in the water has reduced a little sulphate to sulphide, which has then been attacked by the acid present in the sample, and the liberated hydrogen sulphide has reacted with the iron compounds present with the production of colloidal ferrous sulphide.—A. R. P.

Bauxite as a pigment. M. Ragg. Farben-Zeit., 1919, 25, 151. Chem. Zentr., 1920, 91, II., 86.

THE author agrees with Cobenzl (see preceding abstract), that the blue colour of bauxite is due to colloidal ferrous sulphide. The occurrence of blue bauxite indicates that the mineral is composed of the residues from the solution of aluminous limestones which have become mixed with organic material of animal and vegetable origin. The decomposition of the latter has then produced the hydrogen sulphide required for the production of the blue colour.—A. R. P.

Varnishes and boiled oils; Determining the acid value of —. H. A. Gardner and R. E. Coleman. Circ. 87, Paint Manufacturers' Assoc., U.S.A., and Nat. Varnish Manufacturers' Assoc. Feb., 1920. 9 pp.

ACID values of oils or varnish media containing colloidally dispersed phases cannot be determined by the usual method of solution in alcohol and titration with dilute alkali, as the end point is indefinite owing to absorption of the alkaline base by the disperse phase. By using a neutralised mixture of equal volumes of purified 90% benzol and denatured alcohol as solvent the colloidally dispersed phase is brought into molecular solution, and can then be titrated as usual. Uncertainty of end point may be overcome by allowing to stand one or two minutes and observing the upper layer of oil and solvent, which at neutralisation should be clear and faintly pink.—A. de W.

Dopes for aeroplanes. Fuchs. See V.

Yellow-tail fish oil. Gardner and Reilly. See XII.

PATENTS.

Coumarone resin soluble in alcohol; Method for producing a —. Rütgerswerko A.-G., Berlin. Ger. Pat. 302,543, 9.6.17.

CAUDE solvent naphtha or heavy benzol, without previous removal of the bases or phenols or after removal of the bases, and with or without the addition of phenols, is subjected to polymerisation by the action of sulphuric acid or of sulphonic acids; on account of the presence of phenols the coumarone resin obtained in the usual way by subsequent distillation is soluble in alcohol and therefore suitable for the preparation of varnishes.—D. F. T.

Resin; Production of — from wood containing the same. Aktiebolaget Cellulosa, Stockholm. Ger. Pat. 315,731, 20.5.16. Int. Conv., 26.4.16.

WOOD rich in resin, such as Swedish pine, is heated under pressure with sufficient concentrated alkali solution, with the addition of a soluble neutral salt such as sodium chloride, to dissolve the wood and convert the resin into the alkali salts of the resin acids. Fatty acids and lignin precipitated together with the resin are soluble in alcohol and resemble resin in their properties, and thus have no deleterious effect upon the product. Oil of turpentine, methyl alcohol, ammonia, and amines distil over if the vapour is allowed to escape during the heating.—L. A. C.

Dope or substitute for celluloid, vulcanite, and the like. W. T. Robinson-Bindley and A. W. Weller, London. U.S. Pat. 1,331,127, 17.2.20. Appl., 20.2.19.

SEE Eng. Pat. 134,565 of 1918; this J., 1920, 14 A.

Terpenes etc. from defecation sludge. Ger. Pat. 316,503. See IIb.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Caoutchoucs; Scientific principles for the recognition of artificial — in technical rubber analysis. C. Harries. Gummi-Zeit., 1919, 33, 222—223. Chem. Zentr., 1920, 91, II., 143.

THE rubber is swollen in ten times its quantity of chloroform and then treated with 5.5—6% ozono until a sample no longer decolorises bromine; the solution is cautiously evaporated below 20° C. in a vacuum, and after drying in a vacuum for several days the ozonide is decomposed by boiling for an hour with twelve times its weight of water. Ozonides of caoutchoucs derived from isoprene and dimethylbutadiene decompose fairly smoothly with very little resin formation. A little of the filtered solution is treated with a few drops of ammonia solution, then acidified with acetic acid and boiled, when, except in the case of abnormal sodium caoutchoucs, the vapours show Knorr's pyrrole reaction and redden a pine shaving which has been wetted with hydrochloric acid. Ordinary Elberfeld isoprene caoutchouc yields 46% levulinic aldehyde and 60% levulinic acid together with formic acid and a little acetylacetone. The sodium-carbon dioxide isoprene-caoutchouc of the Badische Co. behaves similarly but more easily yields acetylacetone and succinic acid. The estimation of the various decomposition products is effected in the manner which has been described earlier. "Normal" dimethylbutadiene-caoutchouc yields acetylacetone almost quantitatively. The most difficult estimation is that of butadiene-caoutchouc; this yields only succinic dialdehyde, the phenylhydrazone of which is unstable but is readily converted by dilute hydrochloric acid into a white polymeric base melting at 184°—185° C. Vulcanised rubbers should be first extracted with acetone and then ozonised in the pre-

sence of calcium carbonate; in this way the resulting aldehydes and ketones are preserved from resinification, whilst the sulphuric acid and the organic acids are found in combination with calcium.—D. F. T.

Synthetic caoutchouc; Analytical estimation of —. G. Hubener. Gummi-Zeit., 1919, 33, 361—362. Chem.-Zentr., 1920, 91, II., 143—144.

"METHYL-RUBBER," whether raw or lightly vulcanised, undergoes bromination slowly and yields a product containing a higher proportion of bromine than that expected for a tetrabromide. Extraction of the bromide with carbon bisulphide and titration of the insoluble portion showed that raw and lightly vulcanised methyl-rubber on bromination yield a tetrabromide $C_{11}H_{16}Br_4$ and a bimolecular proportion of methyl bromide; hard vulcanised methyl-rubber gives no methyl bromide.—D. F. T.

Rubber goods and reclaimed rubbers; Rapid determination of "golden antimony" (antimony sulphide) in —. W. C. Schmitz. Gummi-Zeit., 1918, 33, 2—3. Chem.-Zentr., 1920, 91, II., 144—145.

TWO grams of the material is heated with 45 c.c. of sulphuric acid in a 300 c.c. Kjeldahl flask for 20 min.; the cooled solution is then treated with 3 grms. of ammonium persulphate and gently reheated for 10—15 min. longer after it has become colourless. The cooled liquid is diluted, almost neutralised with ammonia regardless of any precipitate, feebly acidified with hydrochloric acid, and then saturated with hydrogen sulphide. The precipitate is dissolved in hydrochloric acid containing tartaric acid, and the filtrate, after expulsion of the hydrogen sulphide, is titrated with potassium bromate using methyl orange as indicator. If the original material contains no iron the precipitation can be omitted; the colourless solution in sulphuric acid is heated for 20 min. with 5 grms. of potassium sulphate; a little potassium metabisulphite is introduced and the sulphur dioxide expelled by boiling; after repeating this operation twice more the liquid is diluted with 40 c.c. of water, and 10 c.c. of hydrochloric acid and a considerable quantity of tartaric acid added; titration is then effected as in the earlier process. The whole estimation needs 2—4 hours.—D. F. T.

PATENTS.

Rubber; Process for regenerating vulcanised —. S. van Raap, Amsterdam, Holland. Eng. Pat. 122,188, 3.1.19. (Appl. 227/19.) Int. Conv., 8.1.18.

GROUND waste rubber is dissolved with the aid of mechanical treatment at 150° C. in the smallest possible amount of an oil, such as linseed oil or castor oil, which is capable of combining with sulphur; the mixture is then heated at 160°—170° C. with sufficient sulphur and for a sufficient time to convert the oil into a solid mass; after having been rolled this is subjected to further treatment, with or without the addition of new rubber, and is finally vulcanised.—D. F. T.

Rubber and other heavy plastic material; Machine for treating —. Machines for kneading or mixing rubber and other heavy plastic material. F. H. Banbury, Ansonia, Conn., U.S.A. Eng. Pats. 119,241 and 119,242, 17.9.18. (Appls. 15,085 and 15,086/18.) Int. Conv., 21.3.17 and 18.11.16.

XV.—LEATHER; BONE; HORN; GLUE.

[Tannery] lime liquors; Analysis of —. Report of committee of Soc. of Leather Trades' Chem. D. Burton. J. Soc. Leather Trades' Chem., 1920, 4, 32—41.

DEFLATION, swelling, solvent action on the

epidermis, interfibrillary substance and the true hide substance, and removal of the fat are the effects generally desired in the process of liming. The depilatory properties of a lime liquor can be improved by addition of sulphides which are represented by the amount of $N/10$ $ZnSO_4$ solution required per 100 c.c. of lime liquor, giving the figure s . The swelling depends on the alkalinity and the content of soda and salt. The total alkalinity a is determined by Bennett's method (this J., 1916, 479), and includes that derived from calcium hydroxide, sodium hydroxide, sodium hydrosulphide, ammonia, and lime in combination with the weakly acid decomposition products of the hide and epidermis. The swelling increases with the caustic alkalinity due to $NaOH$ and $Ca(OH)_2$, which can be determined by titrating 100 c.c. of the filtered lime liquor with $N/10$ HCl with phenolphthalein as indicator. Sodium hydrosulphide ($\frac{1}{2} s$) has no swelling action, but ammonia (c.c. $N/10$ HCl per 100 c.c. of lime liquor = m) has a slight swelling effect. Lime in combination with weak acids, $a - (c + \frac{1}{2}s + m)$, depresses the ionisation and also the solubility of the lime and consequently the swelling. Sodium hydroxide is included in the alkalinity, but it and the salt can be determined by Bennett's method (*loc cit.*) Salt is stated to depress the swelling. The solvent action of lime liquors depends on the acid products from the epidermal matter and dissolved hide substance, the amino-acids present, free ammonia, total nitrogen, and the bacteria present. The difference in the titration figures with acid, using phenolphthalein and methyl orange as indicators, is a measure of the acid products. The amino-acids are determined by Stiasny's method (this J., 1910, 771) and the total nitrogen by the Kjeldahl method. Plate cultures from a lime liquor on a slightly alkaline peptone gelatin give a measure of the gelatin-liquefying bacteria. It is necessary to adopt a definite method of filtration which will give a constant degree of clearness. The degree of hydrolysis is $100(178-a)/174$ where $a=2.8 K/\nu$, K being the Kjeldahl figure and ν the no. of c.c. of $N/10$ $NaOH$ per 50 c.c. of lime liquor in the Stiasny amino-acid determination. A list of the principal references to the modes of action and methods of analysis of lime liquors is given.—D. W.

PATENTS.

Liming and dehairing hides; Process of —. P. Remer, Milwaukee, Wis., Assignor to A. G. Wackenreuter and W. S. Shaw, Chicago, Ill. U.S. Pat. 1,331,723, 24.2.20. Appl., 14.6.18.

HIDES are first soaked in a weak lime liquor containing a moderate amount of sodium sulphide, then washed, and soaked in a materially stronger solution of lime unmixd with sodium sulphide.—D. W.

Condensation products [tanning materials] from formaldehyde and aromatic derivatives; Manufacture of —. A. G. Bloxham, London. From Durand & Huguenin S. A., Basle, Switzerland. Eng. Pat. 138,796, 27.5.19. (Appl. 13,356/19.)

TANNING materials are prepared by condensing a 1- or 2-aminonaphthalenemono- or disulphonic acid, or a mixture of the same, and an aromatic hydroxy compound which contains not more than one hydroxyl group to each nucleus and one or more salt-forming acid groups, with formaldehyde, or a substance which develops formaldehyde, in dilute solution, at 60° – 80° C. in the presence of a little sulphuric acid. The clear liquor obtained is neutralised with sodium carbonate and/or a hydroxide of a metal, the salts of which have a tanning action, e.g., chromium, aluminium, or iron.—L. A. C.

XVI.—SOILS; FERTILISERS.

Soil and its water content; Relations existing between the —. B. A. Keen. J. Agri. Sci., 1920, 10, 44–71.

A RESUME is given of investigations on moisture in soil and its behaviour under varying conditions. The subjects dealt with are the maximum water capacity and pore space, the permeability, the capillary effects and the water-retaining capacity, the capillary movement of soil water, the hygroscopic moisture, and the surface forces. The last subject is dealt with from the point of view of the colloidal hypothesis, and it is considered that one of the most important soil constituents is the colloidal clay, as it is associated with a small amount of pore space which modifies the movement of water in the soil interstices over a wide range of moisture content. A change in the moisture content is reflected in a change in the soil solution. Conclusions based on an examination of soil solution after it has been removed from the soil are open to doubt. The system, soil plus soil solution, must be treated as a whole.—J. H. J.

Nitrates; Washing out of — by drainage water from uncropped and unmanured land. E. J. Russell and E. H. Richards. J. Agric. Sci., 1920, 10, 22–43.

AN account is given of the results from three natural soil gauges in operation at Rothamsted since 1870. The soil received no manure, and was kept free from weeds. All the drainage water was collected, measured, and analysed. During the whole period there was a continuous, though remarkably slow, loss of nitrate from the soil. Originally the soil contained 3500 lb. of nitrogen per acre in the top 9 in.; in 1917 this had been reduced to 2380 lb., and the drainage water had carried away 40 lb. of nitrogen per acre per annum. All the nitrogen removed from the soil appeared as nitrate in the drainage water. The amount of nitrogen removed at any time varied with the rainfall, a high rainfall removing higher amounts of nitrate. On the average 1 in. of rain removed 1 lb. of nitrogen per acre. The slowness of the removal of nitrogen may be attributed to the ammonia and nitrate formed as a result of the decomposition of the organic matter being possibly assimilated by organisms, such as algae, fungi, bacteria, and that only on the death of these organisms is part of the nitrogen finally given out and part re-assimilated. The reliability of the results is shown by the fact that the total of the chlorine in the rain-water during the whole period agreed with the total of the chlorine in the drainage water.—J. H. J.

Ammonia in soil; Determination of —. D. J. Matthews. J. Agric. Sci., 1920, 10, 72–85.

DETERMINATIONS of ammonia in soil were made by the aeration method. The apparatus used consisted of a long, wide, aeration tube placed in a sloping position, the lower end being closed with a cork carrying the air inlet tube, and the upper end connected to the bottom of an absorbing apparatus, consisting of an upright tube with two bulbs and with a splash retainer in its upper end. 25 gms. of soil is placed in the lower part of the aerator, and 50 c.c. of a solution of sodium carbonate and chloride and 1 c.c. of paraffin are added. $N/50$ sulphuric acid and a few drops of methyl red are placed in the absorber, and a current of air washed through acid is drawn through the apparatus at the rate of 300 litres per hour for 6 hours. With most soils the efficiency of the apparatus was found to be over 98%. With soils containing much unstable organic matter, such as heavily-manured glass-house soils, it was found better to replace the

usual alkaline solution by a solution of magnesia in strong sodium chloride and to aerate for a fixed period of 3 hours. With soils rich in chalk, low results were always obtained unless the samples were very finely ground.—J. H. J.

PATENT.

Phosphoric acid from superphosphate manufacture: Utilisation of — in the saccharification of cellulose. F. L. Schmidt, Stolberg, and Chem. Fabr. Rhenania, Aachen. Ger. Pat. 304,400, 11.7.17.

PHOSPHATES are treated with a small excess of sulphuric acid so as to produce phosphoric acid instead of superphosphate; this phosphoric acid is employed for the saccharification of cellulose, and is then precipitated in the form of a high-grade fertiliser by the addition of lime or chalk.—J. F. B.

XVII.—SUGARS; STARCHES; GUMS.

Milk sugar [lactose] from whey; Extraction of —. H. E. Woodman. J. Agric. Sci., 1920, 10, 1—11.

EXPERIMENTS were made on the possibility of improving the method of extraction of lactose from whey. In the German method the whey is nearly neutralised with caustic soda, evaporated *in vacuo* at 60°–70° C. to a tenth of its volume, and allowed to cool. The sugar crystallises out and is separated by the centrifuge. The mother liquor is boiled, the precipitated protein filtered off, and the filtrate allowed to crystallise. A recovery of 75% of the sugar (3.85% on the whey) is obtained. By removing the protein by heating at 93° C. before evaporating, clarifying the filtrate with alumina cream, evaporating, and treating with charcoal and magnesium and calcium sulphates, a yield of 32% of lactose (on the whey) was obtained, containing 1.5% each of protein and ash, a greater degree of purity than by other methods. It was found that an extremely small amount of iron was dissolved from the vacuum pan during evaporation.—J. H. J.

Invertase; Detection of — in the pure honey of bees. A. Caillas. Comptes rend., 1920, 170, 589—592.

INVERTASE has been isolated, as its aqueous solution, from a sample of honey, and characterised, particularly in so far as its action on sucrose is concerned.—W. G.

PATENTS.

Adhesive compositions; Improving —. M. Sarason, Berlin. Ger. Pat. 316,080, 2.7.18.

ADHESIVE compositions from water-glass, sulphite-cellulose waste lyes, or the like, are improved by the addition of a small quantity of liquorice extract or glycyrrhizin.—L. A. C.

Adhesive; Manufacture of an — from sulphite-cellulose waste liquor. K. Hüttenes, Düsseldorf. Ger. Pat. 316,234, 14.2.18.

LEAD acetate is added to sulphite-cellulose waste liquor, and the solution is evaporated.—L. A. C.

Defecation sludge. Ger. Pat. 316,503. See IIA.

Saccharification of cellulose. Ger. Pat. 304,400. See XVI.

Saccharification of cellulosic material. Ger. Pat. 304,399. See XIXA.

XVIII.—FERMENTATION INDUSTRIES.

Enzymes. III. Invertase and other enzymes of germinated barley. D. Maestrini. Atti R. Accad. Lincei, 1919, [V.], 28, ii., 509—511. (Cf. this J., 1920, 244A.)

INVERTASE can be extracted from germinated barley dried at temperatures below 40° C. by 0.003 mol. % acetic or hydrochloric acid solution, and is present, not only in the emulsion, but also in the filtrate of the extract. To obtain an active liquid, the extraction should last at least six hours at 30°–35° C.; the enzyme acts best at about 50° C. and is destroyed at 55° C. The activity of malt invertase is destroyed in 48 hours by 0.003 mol. % potassium hydroxide solution. Germinated barley contains a catalase and an oxydase, but no maltase, lactase, or rennase; the extract does, indeed, coagulate milk, but such coagulation is due to the acidity of the extract, as it is produced even after the latter has been boiled.—T. H. P.

Fermentation; Action of copper on alcoholic —. K. Schweizer. Bull. Assoc. Chim. Sucr., 1919, 37, 160—173.

IN comparative experiments on the fermentation of sucrose solutions by yeast, in glass and copper vessels, fermentation ceased in the copper vessels when less than half of the sugar had been decomposed. The inhibitive influence of the metal was not prevented by previously cleaning it with sand or by conducting the fermentation in presence of calcium carbonate to neutralise the acids produced. A 6% brewery wort and a 24% laboratory malt wort fermented as well in copper vessels as in glass ones; only in the case of a very light laboratory wort was any unfavourable effect of the copper observed, and this was not very marked. It is probable that in ordinary worts the proteins protect the yeast from the toxic action of traces of dissolved copper by combining with the latter themselves. Copper (coins) previously cleansed by immersion in nitric acid exerted no inhibitive action on the fermentation of sucrose solution in experiments extending over two days. In further experiments it was found that cupric oxide added to fermenting sugar solutions has no adverse effect, but cuprous oxide has a powerful inhibitive action. Quantities of cuprous oxide larger than 0.013 gm. per 0.5 gm. of pressed yeast entirely prevented fermentation, whilst quantities less than 0.1 mgm. per 0.5 gm. of yeast had no inhibitive action.—J. H. L.

Wine making; Applications of the reducing action of yeast in —. Matthieu. Bull. Assoc. Chim. Sucr., 1919, 37, 174—175.

THE method of treating the yellow malady of wines by re-fermentation depends, for its effect, on the reducing action of the yeast on the oxidation products which are responsible for the colour, flavour, and aroma characteristic of the malady. Bitterness in wines may be partially or entirely eliminated by similar treatment, or simply by mixing the wines with active yeast. The "cooked" flavour acquired by musts when heated, *e.g.*, for the purpose of concentration or pasteurisation, disappears again during the fermentation. Bitterness or "cooked" flavour in red wines may be effectively treated by means of active yeast, but the colour is diminished at the same time owing to adsorption by the yeast. The practice of improving "flat" wines by contact with marc from the presses depends to some extent on the reducing action of the yeasts of the marc, but other factors, such as admixture with the new wine contained in the marc, also play a part; the benefit of this treatment is often lost if the regenerated wine is afterwards exposed to the air. Another application of the reducing action of yeast

consists in the preservation of the characters of young wine in wines kept in contact with active yeast; the presence of the yeast tends to prevent the formation of oxidation products which would confer the characters of old wines.—J. H. L.

"Buffer" systems. Windisch and Dietrich. See VII.

Invertase in honey. Caillas. See XVII.

Titration of mixed acids. Kolthoff. See XXIII.

PATENTS.

Ethyl alcohol and de-alcoholised beverages from fermented liquors; Manufacture of —. H. Wade, London. From J. Schneible, Chicago, U.S.A. Eng. Pat. 139,099, 18.7.19. (Appl. 18,041/19.)

See U.S. Pat. 1,311,421 of 1919; this J., 1919, 693 A.

Saccharification of cellulose. Ger. Pat. 304,400. See XVI.

Saccharification of cellulosic material. Ger. Pat. 304,399. See XIXA.

XIXA.—FOODS.

Milk; Differentiation between poor and adulterated —. T. R. Hodgson. Analyst, 1920, 45, 91—94.

THE author has applied the formulæ given by Lythgoe (this J., 1914, 1166) to a large number of samples of milk, and finds that the formulæ are untrustworthy for the purpose of distinguishing between poor and adulterated milk. For instance, a milk containing 0.30% of fat and 7.31% of solids-not-fat, and admittedly skimmed, gave results by the formulæ indicating that it was "poor but genuine."—W. P. S.

Cheeses; New method of testing —. A. Geake. J. Agric. Sci., 1920, 10, 86—89.

AN attempt was made to estimate the degree of ripeness of a cheese from the extent of proteolysis as measured by the amino-nitrogen. Sørensen's method of determining amino-nitrogen was applied as follows:—8 grms. of cheese is ground three times with 30 c.c. of acetone and the residue allowed to dry in the air. 3 grms. of this residue is shaken with 50 c.c. of N/10 potassium hydroxide for 1 hr., and the mixture is filtered. 20 c.c. of the filtrate is titrated to neutrality with N/10 sulphuric acid, using phenolphthalein as indicator. 10 c.c. of neutral formaldehyde solution is then added, and the solution titrated back as usual. The total nitrogen is determined in 5 c.c. of the cheese solution by Kjeldahl's method. The result is returned as percentage of amino-nitrogen in total nitrogen. The method was tested on various cheeses of known quality, and a comparison is made between the figures obtained and the quality expressed in commercial terms.—J. H. J.

Marine algæ; Utilisation of — as foods. P. Gloess. Monit. Scient., 1920, 64, 3—5.

CERTAIN seaweeds (*Fucaceæ*, *Laminariaceæ*), after proper treatment for the removal of salts, form a valuable food or fodder; potassium salts and iodine may be recovered from the extracted salts.

—W. P. S.

Lactose from whey. Woodman. See XVII.

Zinc in foods etc. Weitzel. See XXIII.

PATENTS.

Foodstuffs; Manufacture of —. H. Bollmann, Hamburg, Germany. Eng. Pat. 109,265, 27.8.17. (Appl. 12,297/17.)

OIL-CONTAINING seeds, yeast, dried fish, etc., are extracted with a mixture of 120 parts of carbon tetrachloride and 80 parts of 96% alcohol with the aid of heat. Fat, fatty acids, and bitter principles are thus removed, and the residue after removing the solvent is in a state suitable for use as a food-stuff.—J. H. J.

Refrigeration of fish and other foods. N. Dahl, Trondhjem, Norway. Eng. Pat. 138,788, 13.5.19. (Appl. 11,965/19.)

FISH are packed in containers to which a refrigerating liquid is admitted at the bottom and passes upwards through the mass of fish. The rate of passage of the liquid can be controlled so as to prevent the fish becoming compressed at the bottom of the container. The liquid overflows at the top of the container and is collected, re-cooled, and used again. The process is conducted in the open air. (Reference is directed to Eng. Pat. 125,076; this J., 1919, 788 A.)—J. H. J.

Materials [foodstuffs] subject to putrefaction or decay; Methods for preserving — and apparatus therefor. R. Pape, Bussum, Holland. Eng. Pat. 139,100, 21.7.19. (Appl. 18,140/19.)

THE substance is placed in a double-walled chamber, and the development of bacteria is prevented by "suppressing the negative electric field and negative electric earth field by superposing an artificial electric field on the electrical earth field and in an opposite direction thereto," or by passing moist air into the chamber which is insulated from the earth, the air being first passed over moist layers (the moistened walls of the chamber) or over metallic conductors.—W. P. S.

Chocolate and other materials; Cooling chambers for —. E. C. R. Marks, London. From Fours et Proc. Mathy, Liège, Belgium. Eng. Pat. 139,383, 6.6.19. (Appl. 14,442/19.)

AN endless band conveys the material through a chamber; cold air flows through the latter, passing first over the upper stretch of the band and returning either through the space between the upper and lower parts of the band or beneath the lower part of the band. The spaces in the chamber may be formed by the band itself. The air leaving the chamber may be dried, compressed, and delivered into a cooler mounted near the upper part of the chamber.—W. P. S.

Flour; Process of bleaching and improving —. E. T. Fegan, Minneapolis, Minn., Assignor to A. R. Sasse, Kansas City, Mo. U.S. Pat. 1,330,937, 17.2.20. Appl., 18.3.18.

FLOUR is subjected to the action of gaseous chlorine and ammonia under such conditions that a "precipitate of ammonium chloride" is formed.

—W. P. S.

Bran and similar products; Process of extracting —. L. C. Reese, Saginaw, Mich. U.S. Pat. 1,331,125, 17.2.20. Appl., 19.3.18.

BRAN is treated in a series of extractors with an aqueous medium at a temperature most favourable for the conversion of the starch into sugars and at which the albumin is not coagulated; the exhausted material from this first series is then extracted in a second series with boiling water, the hot extract obtained being cooled to the temperature of the liquid in the first series of extractors on its passage to the latter.—W. P. S.

Foods or other substances; Process of heat-treating — N. H. Fooks, Mount Airy, Md. U.S. Pat. 1,331,337, 17.2.20. Appl., 29.10.19.

A liquid is heated under pressure to a temperature above its boiling point, and the substance to be treated is fed continuously into the vessel containing the liquid, passing first through the gas space above the liquid, then into the liquid, and on emerging from the latter is discharged from the vessel.—W. P. S.

Cellulosic material; Saccharification of — with gaseous hydrochloric acid. Chem. Fabr. Rhénania A.-G., Aachen, F. L. Schmidt and G. A. Voerkelius, Stolberg. Ger. Pat. 301,399, 8.5.17.

THE material is first moistened with cold highly concentrated (40%) hydrochloric acid and then treated with gaseous hydrogen chloride. Example: 400 kilos. of sawdust is uniformly moistened with 400 litres of 40% hydrochloric acid and, after steeping for a short time, the mass is saturated with the acid gas. The acid is allowed to act upon the wood for about 6 hours, and is then drawn off by suction. If the residual acid be neutralised with lime or soda the product is directly available for use as a feeding material in which the whole of the cellulose of the wood is present in the form of sugar or dextrin. If it be desired to convert the dextrin into fermentable sugar the product of the reaction must be lixiviated and the solution heated for 2 hours at 120° C.

—J. F. B.

Potatoes; Preservation of —. Verein der Spiritus-Fabrikanten in Deutschland, Berlin. Ger. Pat. 315,853, 5.1.17.

A MIXTURE of finely-shredded potatoes and sufficient dry pulp, straw meal, or the like, to absorb the water in the potatoes, with or without the addition of molasses, yeast, or the like, is compressed by a pressure up to about 200 atm.—L. A. C.

Saccharification of cellulose. Ger. Pat. 304,400. See XVI.

XIXB.—WATER PURIFICATION; SANITATION.

Water; Determination of the rate of solution of atmospheric nitrogen and oxygen by —. Part II. W. E. Adeney and H. G. Becker. Sci. Proc. Royal Dublin Soc., 1919, 15, 609–628. (See also this J., 1919, 156 A.)

DETAILS of further experiments are given, together with formulæ by means of which the rates of solution of oxygen, nitrogen, and air in water may be calculated for any conditions of area exposed, depth, or initial degree of saturation, provided the water is kept uniformly mixed.—W. E. F. P.

Boiler feed water; Removal of dissolved gases from —. L. Jung. Stahl u. Eisen, 1920, 40, 321–326.

IN considering the suitability of feed waters the content of dissolved oxygen and carbon dioxide should not be overlooked (cf. Cobb and Dougill; this J., 1914, 403). A test is described in which polished iron plates were suspended in samples of water boiled in a small apparatus for several hours. The degree of corrosion was determined by the loss in weight and by microscopic examination. A condensed water from a turbine, although containing only 3.23 c.c. of oxygen per litre, was corrosive because, owing to the softness of the water, no protective coating of scale was formed. Although this water when condensed was practically free from

oxygen, tests showed that it had very readily taken up a further quantity. This emphasises the necessity for preserving such water in closed vessels prior to feeding again to the boilers. A sample of condenser water containing 5.0 c.c. oxygen per litre freed from oil by electrical treatment was the most corrosive of four samples examined, especially when mixed with fresh water from the public supply which contained 7.0 c.c. of oxygen per litre. A condenser water carefully maintained free from oxygen to which the test was applied for 13 hrs. showed scarcely any attack on the metal. Dissolved oxygen should be removed from a boiler feed when possible. Chemical methods such as a preliminary treatment with iron turnings are unsatisfactory. A preliminary heating followed by collection of the gases may be effective. Mechanical methods involving agitation under reduced pressure are effective in large-scale continuous work. In every case treated water should be stored in vessels out of contact with air, and the same precaution should be taken with condenser water which may be quite satisfactory when first collected.—H. J. H.

Refuse destruction; Economics of —. P. M. Grempe. Feuerungstech., 1920, 8, 86–87.

IN consequence of the shortage of some of the principal building materials during the war, experiments have been made in Germany with the clinkers produced by the destruction of refuse in municipal incinerators. Particularly successful results were obtained in the town of Fürth, in Bavaria. The clinkers from the incinerators contained 55.6% SiO₂, 11.7% Fe₂O₃, and 13.9% Al₂O₃. Of the 55.56% SiO₂, 2.15% is soluble in soda solution, and 3.11% (together with 1.80% Al₂O₃) soluble in 30% potash solution, showing the presence of hydraulic silicates. The clinker inhibits rusting, and therefore is suitable as an aggregate for reinforced concrete. The clinkers have been used for the manufacture of paving stones, kerb-stones for pavements, covers for cable-conduits, etc. In tests with concrete prepared from mixtures of cement, sand, and clinker in the proportions 1:2:4, 1:3:6, and 1:4:8, the crushing strength after keeping for the periods mentioned, in a moist condition, were respectively: 28 days, 232, 139, and 55.6 kilos. per sq. cm.; 90 days, 288, 171, and 71.2; 1 year, 322, 190, and 94.0 kilos. per sq. cm.—A. G.

PATENTS.

Sewage; Process of treating —. J. W. Wilson, San Diego, Cal. U.S. Pat. 1,331,735, 24.2.20. Appl., 4.12.16.

IN the treatment of a sewage comprising septic and antiseptic portions, the two portions are kept separate, and the septic portion is retarded in flow to permit of the solids breaking down. When this has been effected the sewage is introduced into a liquefying tank. After liquefaction is completed, the liquid is run out and added to the antiseptic portion of the sewage.—J. H. J.

Liquids; Process and apparatus for sterilisation or pasteurisation of —. A. Mulertz, Frederiksberg, Denmark. Eng. Pat. 132,237, 6.8.19. (Appl. 19,425/19.) Int. Conv., 31.8.18.

THE liquid is passed upwards through narrow vertical tubes surrounded by a steam jacket, and then downwards through a similar series of tubes provided with a cold-water jacket. The exit for the liquid is fitted with a counter-pressure valve which may be so adjusted that the liquid does not boil during the treatment.—W. P. S.

Separating liquids. Eng. Pat. 135,655. See I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Quinine salts; Detection of other alkaloids in —. I. M. Kolthoff. Pharm. Weekblad, 1919, 56, 451—459.

KERNER and Weller's method is not sufficiently accurate under all conditions, and has many disadvantages. The author has devised a test which is suitable for quinine sulphate but not applicable to other salts. The nitroprusside test is not considered to be sufficiently sensitive. The most accurate test for all quinine salts is De Vrij's chromate test, for the application of which to quinine bisulphate and quinine a few preparatory details are given. (See also J. Chem. Soc., 1920, ii., 203.)

—W. J. W.

Aribin; Identity of — with harman. E. Späth. Monatsh., 1919, 40, 351—359.

A COMPARISON of aribin, its hydrochloride, its aurichloride, its chloroplatinate, and its picrate showed their identity with harman and its derivatives. (See further J. Chem. Soc., 1920, i., 327.)

—J. K.

Scabiosin, a new glucoside hydrolysed by emulsin. E. Bourquelot and M. Bridel. Comptes rend., 1920, 170, 486—491.

FROM the roots of *Scabiosa succisa*, L. the authors have isolated, in addition to sucrose, a new glucoside, scabiosin, which on hydrolysis gives dextrose and a yellow substance insoluble in water.

—W. G.

Ketones; Reduction of aromatic —. W. D. Cohen. Rec. Trav. Chim., 1920, [IV.], 1, 243—279.

WHEN benzophenone is reduced in an absolutely neutral medium, e.g., absolute alcohol, by means of sunlight, benzopinacolone is the only product formed. The velocity of the reduction is affected by several factors such as the nature and concentration of the alcohol, and the amount of active light. It varies considerably with different aromatic ketones. (See further J. Chem. Soc., Apr., 1920.)—W. G.

Chloroform; Testing of —. Utz. Pharm. Zentr., 1917, 58, 1—5.

FOR the detection of decomposition products in chloroform, particularly those due to the action of sunlight, the benzidine test described originally by Budde (Geb. Militärsanitätswesen, No. 55, 120) is useful. A small quantity of benzidine is dissolved in 10 c.c. of the sample. With pure chloroform the solution does not exhibit any change when kept in the dark for 24 hours; if chloral is present, a faint pink coloration is obtained, and this shows a bluish tint after some time. Chloroform containing phosgene, hydrochloric acid, or chlorine at once yields a turbid solution, and a precipitate may form if the amount of these impurities is large—W. P. S.

Acids; Preparation of aliphatic — by catalytic oxidation of primary alcohols. A. Mailhe and F. de Godon. Comptes rend., 1920, 170, 517—519.

THE vapours of primary aliphatic alcohols readily undergo oxidation when passed over finely-divided copper at 260°—270° C. From 100 c.c. of ethyl alcohol the highest yield of acetic acid was 18.18 grms., together with 12 c.c. of acetaldehyde.—W. G.

Methyl chloride and bromide; Preparation of — from dimethyl sulphate. C. Boulin and L. J. Simon. Comptes rend., 1920, 170, 595—597.

METHYL chloride may readily be prepared by allow-

ing dimethyl sulphate to drop into hydrochloric acid (16° B., sp. gr. 1.125) at about 50° C., or into a concentrated solution of sodium chloride at 60°—65° C. Similarly methyl bromide may be prepared by using hydrobromic acid at 45°—50° C., or, preferably, a concentrated solution of an alkali bromide, slightly acidified with sulphuric acid, at 30°—35° C. In all these cases the yield is about 90% of theory.—W. G.

Ethylene; Chlorination of — in the presence of calcium chloride. J. A. Smythe. Gas. J., 1920, 149, 691—692.

ETHYLENE and chlorine react very readily in the presence of calcium chloride. A mixed product, containing 50—60% of ethylene dichloride is obtained. Much heat is disengaged, and the apparatus should be cooled during the operation. The products are purified by fractionation. The chlorinated product contains, on the average, 50% of dichloroethane, 25% of trichloroethane, and 25% of higher-boiling products. The calcium chloride apparently acts as a catalyst, but its activity may be due to the presence of a small amount of iron (1 in 50,000 estimated colorimetrically).—A. G.

Chromates; Photo-oxidation of organic compounds by —. J. Plotnikow. Z. wiss. Phot., 1919, 19, 40—56.

ETHYL alcohol is oxidised by ammonium chromate and bichromate in the presence of light of wave length $\lambda = 436\mu$ according to a linear equation. The velocity constant is proportional to the alcohol concentration, and approaches a maximum with increasing salt concentration. The temperature coefficient of the reaction is 1.02. (See also J. Chem. Soc., Apr., 1920.)—J. F. S.

Catechol. Elsner. See III.

Titration of mixed acids. Kolthoff. See XXIII.

PATENTS.

Acetone; Process of manufacturing —. H. W. Matheson, Shawinigan Falls, Canada. Eng. Pat. 138,679, 23.1.19. (Appl. 1729/19.)

ACETIC acid vapour is passed through a metal tube heated to 375°—525° C., and filled with metal balls coated with a catalyst, e.g., a mixture of 75 to 95% of lime and 25 to 5% of magnesia, or the oxide, hydroxide, carbonate, or acetate of any metals the acetates of which are converted to acetone on heating. The greater part of the acetone produced is condensed by passing the escaping gases through a water-cooled condenser, and the last traces are recovered by scrubbing the residual gases with water.—L. A. C.

Ether; Process for purifying —. C. Claessen, Berlin. Ger. Pat. 298,822, 28.11.15.

ETHER of sufficient purity for use in the gelatinisation of nitrocellulose, even if it has been prepared from denatured alcohol, is obtained by treating the crude product containing aldehydes with solid alkali hydroxides, with subsequent separation of the product from the hydroxides and aldehyde-resin.—L. A. C.

Sterile virus for medicinal use; Production of —. Quarzlampen Ges.m.b.H., Hanau. Ger. Pat. 314,859, 24.2.14.

RADIATIONS from chemically active sources, e.g., ultra-violet rays, are allowed to fall upon vaccine suspensions long enough to destroy harmful bacteria. The virus has a greater resistance to the bactericidal action of the rays, and its activity is reduced to a negligible extent only.—H. J. H.

Lecithin; Process for purifying —. C. F. Hildebrandt, Hamburg. Ger. Pat. 315,941, 31.12.15.

CRUDE lecithin is dissolved in water or other solvent, and sufficient dilute mineral or organic acid is added to precipitate the lecithin without saponification of the same, i.e., the acid concentration must be less than 1%. Impurities and adulterants remain in solution, and the product may be further purified by repeating the process.—L. A. C.

Formaldehyde, methyl alcohol, methyl formate, and acetone; Production of — by dry distillation of formates. K. Hofmann, Charlottenburg. Ger. Pats. (A) 316,216, 24.1.17, and (n) 316,217, 12.2.18.

(A) BASIC compounds formed during the low temperature dry distillation of formates, e.g., zinc formate, which hinder the production of formaldehyde, methyl alcohol, methyl formate, and acetone, and promote the formation of undesired compounds, are neutralised by the continuous or intermittent addition of formic acid during the distillation. (a) The production of formaldehyde etc. by the dry distillation of formates is increased by the presence of feebly acid compounds such as zinc or iron sulphate, precipitated silica, and zinc, iron, or stannic phosphate, and the decomposition takes place at a lower temperature. Whereas calcium and zinc formate yield respectively 17% and 30%, a mixture of 2.5 parts of zinc sulphate and calcium formate yields 50% of a mixture of formaldehyde and methyl alcohol in accordance with the equations: $-2\text{CH}_3\text{O}_2 = \text{CH}_3\text{O} + \text{CO}_2 + \text{H}_2\text{O}$ and $3\text{CH}_3\text{O}_2 = \text{CH}_3\text{OH} + 2\text{CO}_2 + \text{H}_2\text{O}$, and the decomposition is effected at $250^\circ\text{--}280^\circ\text{C}$. instead of about 100°C . higher. Excess of zinc sulphate, iron or acid stannic phosphate increases the yield of formaldehyde; precipitated silica promotes the production of methyl alcohol at the expense of formaldehyde.—L. A. C.

Arsenical compounds; Aromatic —. Rockefeller Institute for Medical Research, Assignees of W. A. Jacobs, W. H. Brown, M. Heidelberger, and L. Pearce, New York. Eng. Pat. 122,819, 21.10.18. (Appl. 17,172/18.) Int. Conv., 28.1.18. Addition to 120,385 (U.S. Pat. 1,280,123; this J., 1919, 268 A).

SEE U.S. Pat. 1,280,127 of 1918; this J., 1919, 268 A.

Aldehydes; Manufacture of —. A. I. Appelbaum, Trenton, N.J., U.S.A. Eng. Pat. 138,999, 19.2.19. (Appl. 4163/19.)

SEE U.S. Pat. 1,302,273 of 1919; this J., 1919, 877 A.

Tobacco; Process for improving —. K. Erslev, Nijmegen, Netherlands. U.S. Pat. 1,331,331, 17.2.20. Appl., 3.1.19.

SEE Eng. Pat. 121,598 of 1918; this J., 1919, 963 A.

Terpenes etc. from defecation sludge. Ger. Pat. 316,503. See IIb.

Side-chain oxidation. U.S. Pat. 1,332,028. See III.

Purifying substances by sublimation. Ger. Pat. 315,958. See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

[Photographic] sensitometer; New non-intermittent —. L. A. Jones. Phot. J., 1920, 60, 80—98.

Two instruments of the continuous exposure, variable time type are described. In the second

instrument, which is better adapted for routine work, a shutter, actuated by a solenoid and ratchet, moves quickly but discontinuously across the sensitive surface at definite time intervals. In the timing mechanism, which controls the solenoid, a cinema film, moved at a uniform speed by a governed electric motor, is provided with a series of collinear apertures spaced from one another at distances corresponding with the exposure ratios required; the distances are measured in terms of the edge perforations to allow for normal variations in the length of the cinema film. One arm of a lever rests on the film in line with the operating apertures, into which successively it drops as the film moves; the lever carries an electric contact point so that the current operating the solenoid is completed when the lever drops. Other perforations etc. are arranged so that the depression of a contact switch starts the whole apparatus, which continues in operation until the exposure is finished and is then automatically cut off. A number of exposure machines may be connected at a central station with corresponding timing mechanisms operated by the same motor. Tables are given showing the timing mechanism to be satisfactory when tested against a chronograph and showing the speed and gamma obtained for a photographic plate to be independent of the absolute exposure, not variable with it as is the case with intermittent exposure machines.—B. V. S.

[Light] filters; Ultra-violet transparent —. A. Miethe and E. Stenger. Z. wiss. Phot., 1919, 19, 57—68.

TARTRAZINE in solutions 1:1000—1:120,000 shows a maximum transparency from $300\text{--}308\mu$ in the most concentrated solution to $280\text{--}391\mu$ in the most dilute. Filter Yellow in the same concentrations has a maximum transparency $296\text{--}308\mu$ with the most concentrated solutions, which increases to $270\text{--}500\mu$ with the most dilute. Martius' Yellow in 1:1000 solution is transparent for wave lengths $321\text{--}330\mu$ and in 1:20000 solution for $296\text{--}374\mu$. Nitrosodimethylaniline is transparent in 1:90000 solution over the range $299\text{--}365\mu$. Fluorescein increases in transparency down to 260μ with increasing dilution. Eosin in 1:1000 is transparent, $368\text{--}390\mu$, and in 1:10000, $271\text{--}470\mu$. The quartz silver mirror is transparent over the range $308\text{--}330\mu$ with a 2 seconds exposure of an iron-carbon arc, whilst with 640 seconds exposure the transparency is over the range $302\text{--}388\mu$.—J. F. S.

[Photographic] development under tropical conditions. A. J. Agnew. Phot. J., 1920, 60, 120—122.

THE sensitive film, before development, is treated in a hardening solution containing formalin and a suitable salt (Eng. Pat. 128,377; this J., 1919, 602 A). The most useful salts are sodium acetate, borate, bicarbonate, chromate, citrate, oxalate, phosphate, sulphate, and tartrate; potassium and magnesium salts are not quite so effective, and ammonium salts are not generally suitable because of the possibility of the liberation of ammonia in development. The hardening process also reduces the rate of development and the tendency to fog, so that development is easier of control than is otherwise the case at high temperatures. Subsequent intensification and reduction processes are not interfered with. There has not been noticed any tendency to separation of the film from the glass after drying. No reticulation is produced, after the hardening process, by considerable variation in the temperatures of developer, fixing bath, and washing waters.—B. V. S.

Dye impression [photographic] printing process. F. W. Donisthorpe. Phot. J., 1920, 60, 119.

A NEGATIVE, on paper, glass, or other suitable support, is treated in a special preparing bath for

about 10 min., rinsed, and then treated with a dye solution for 3–10 min., according to the exposure and development of the negative, a longer immersion being required for over-development or over-exposure. After rinsing the dyed negative it is ready for the production of positives, in the colour of the dye used, by pressing on to it damp gelatin-coated or baryta-coated paper, contact for about 1 min. being requisite. After taking off, the print merely requires quick drying.—B. V. S.

PATENT.

Coloured photographic images and method of producing the same. J. M. Blaney, Verona, N.J., Assignor to Prizma, Inc. U.S. Pat. 1,331,092, 17.2.20. Appl., 22.5.18.

By treatment of a silver image with a solution of a suitable tin salt an insoluble tin compound is deposited on the image and this is then coloured by treatment with a dye solution.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Drying explosives in a tunnel dryer, and recovery of the solvents. G. Weissenberger. Z. ges. Schiess- u. Sprengstoffw., 1920, 15, 33–36.

The apparatus described consists of a tunnel having a separate compartment at each end with inner and outer doors, and serving as intermediate chambers for admitting or discharging bogeys containing the explosive without loss of vapour or heat from the main tunnel. By means of a fan, air is circulated through the tunnel in an opposite direction to the passage of the explosive, the air being heated before entering the tunnel, and after leaving the tunnel passing successively through a pre-cooler and two final coolers; the cooled air from the latter is utilised as a cooling agent for the pre-cooler and, at the same time, undergoes a preliminary heating before returning to the heater and tunnel. In the final coolers, the temperature is lowered to $-50^{\circ}\text{C}.$, and by this means the air is completely freed from moisture and volatile solvent and acquires a high drying capacity. To condense the volatile solvents from the mist in the coolers, the air should be passed through a centrifuge. Risk of fire from static charges of electricity may be prevented by earthing the machine. Means are provided for drawing samples and controlling the temperature, and the rate of passage of the explosive through the dryer may also be regulated. In comparison with ordinary air or vacuum dryers, the apparatus requires only half the fuel consumption, provides continuous and more complete drying and recovery of solvents, and demands little supervision.—W. J. W.

Nitrocellulose; Examination of —. A. Schrimpf. Z. ges. Schiess- u. Sprengstoffw., 1920, 15, 17–20, 38–41, 53–56.

For the estimation of ash, 5 grms. is saturated with 25 c.c. of pure nitric acid (sp. gr. 1.4) in a platinum dish and evaporated to dryness on a water-bath. The residue is heated on an asbestos plate and finally over the naked flame. After cooling it is treated with a little water, again evaporated, and ignited. Foreign matter and unnitrated cellulose are determined by treating 5 grms. with 125 c.c. of a clear, aqueous solution of sodium sulphide (200 grms. in 800 c.c.) in a porcelain dish and gently boiling for 10 min. The residue is washed with water on a dry tared linen filter, previously treated with sodium sulphide, hydrochloric acid,

and water, and dried. The weight of the residue gives the foreign matter from which the unnitrated cellulose is calculated by deducting the ash. Solubility in ether-alcohol is influenced by factors such as the strength of the alcohol and the manner in which the solvents are added. 2 grms. of nitrocellulose after drying over sulphuric acid in a vacuum desiccator is transferred to a cylinder containing about 100 c.c. of ether-alcohol (ether, sp. gr. 0.72, 2 parts; alcohol, 95–96%, 1 part), and rinsed in with a further 100 c.c. After periodical shaking for 6 hours, the nitrocellulose is allowed to settle, and 100 c.c. of the clear liquid is drawn off into a glass dish and evaporated on a water-bath, till the residue assumes a horny appearance, at which stage the drying is completed in a steam-oven. For nitrogen estimations the Schultze-Tiemann method is more accurate than the Lunge method. Owing to shortage of rubber, a modified apparatus has been devised, in which the connections are dispensed with. For determination of sulphuric esters Piest's method is employed (this J., 1913, 1087). In the case of nitrocellulose derived from wood cellulose which contains a considerable amount of calcium sulphate and calcium carbonate, it is necessary to estimate these in order to allocate the correct amount of sulphuric acid to the esters. *Stability tests.* An outline and discussion of various methods are given, but Bergmann and Junk's (this J., 1904, 953), which is the official test, is employed by the author.—W. J. W.

Explosives; Sensitiveness of — to frictional impact [and new pendulum device for measuring same]. S. P. Howell. U.S. Bureau of Mines, Tech. Paper 234, Sept., 1919. 17 pages.

For determining the sensitiveness of explosives more uniformly than is possible by the broomstick and raw hide mallet tests the Bureau of Mines have adopted a pendulum friction device. This comprises a smooth-faced steel anvil, 8.3 cm. \times 30.5 cm., with three grooves for holding the explosive, and a steel shoe faced with hardwood fibre, 8.3 cm. wide, the face having a radius of curvature of 26.7 cm. This shoe swings in a radius of 2 m. from a height of 1.5 m. and is weighted with an additional 20 kilos. For more severe tests a steel shoe without wood fibre is used. 7 grms. of explosive is taken for test, and ten trials are made, the anvil and shoe being thoroughly cleaned after each trial. Tests are carried out at 14° – $30^{\circ}\text{C}.$ Observations are made as to complete or partial explosion, burning, and cracking. An explosive is considered safe if it fails to give more than an almost indistinguishable local cracking. Increase of temperature failed to give discrimination between various explosives. With the steel shoe it was found that nitroglycerin-dynamites and gelatin-dynamites are more sensitive than black blasting powders, blasting gelatin, ammonia dynamites, and explosives containing organic nitrates other than nitroglycerin. Trinitraniline, tetryl, 50/50 amatol, and 60/40 sodatol have a higher sensitiveness than trinitrotoluene, picric acid, ammonium picrate, 85/15 and 80/20 amatol, and 80/20 sodatol. Potassium chlorate explosives are extremely sensitive but can be made sufficiently insensitive by addition of mineral or vegetable oils, or aromatic nitro-compounds. The presence of pyrites-bearing coal greatly increases the sensitiveness of chlorate explosives; even the friction of a copper needle caused ignition in some cases.—W. J. W.

Trimethyleneglycol; Determination of — in distilled (dynamite) glycerin and first runnings. C. A. Rojahn. Z. anal. Chem., 1919, 58, 433–442. The sp. gr. of glycerin (1.2653 at $15^{\circ}/15^{\circ}\text{C}.$) is lowered by the presence of trimethyleneglycol (sp.

gr. 1.0573) or water, or both. If the water content is known the amount of the glycol present may be calculated from the sp. gr. The water is determined by drying 2 grms. of the sample on asbestos under reduced pressure over phosphorus pentoxide; the drying should not be prolonged for more than 48 hours, as the glycol is slightly volatile. Graphs and tables are given showing the percentages of the glycol present in glycerin samples of varying sp. gr. and water content.—W. P. S.

Liquid air; Production and use of — for blasting. H. Diederichs. Stahl u. Eisen, 1920, 40, 52—56.

Liquid air containing about 85% of oxygen is now used to a considerable extent for blasting purposes in non-flery mines. With the use of suitable fillers capable of absorbing $3\frac{1}{2}$ to 4 times their weight of liquid air, it is possible to fire simultaneously 10 to 12 35-mm. cartridges and a still greater number of cartridges of larger diameter. The cartridges in their simplest form consist of paper or fabric containers filled with cork-carbon, cork-powder, soot, etc. They are immersed in the liquid air shortly before use and become saturated in 5 to 10 mins. They have a useful "life" after removal from the bath of 8 to 12 mins. Firing is effected by fuse, direct or in conjunction with a detonator cap, and also electrically. The liquid air is transported in 10-litre containers, made of special glass or porcelain, packed in outer wood or iron vessels.—T. St.

Coal mines; An analytical method for detecting blown-out shots in —. G. F. Hutchison and J. Barab. U.S. Bureau of Mines. Tech. Paper, 210, Oct., 1919. 18 pages.

CHEMICAL tests made on scrapings from a shot-hole in a coal mine, after a blown-out shot, in comparison with tests made on a normal sample of the same coal, enable conclusions to be drawn regarding the nature of the explosive fired and whether the explosive detonated completely or not. Results are given of the products formed under known conditions and examples of deductions drawn from results in unknown cases.—T. St.

Mercury fulminate; Theories regarding the grey and white varieties of —. A. Langhans. Z. ges. Schiess- u. Sprengstoffw., 1920, 15, 7—9, 23—24.

PATENTS.

Octonitroethylenediphenyldiamine; Preparation of —. Farb. vorm. Meister Lucius, und Brüning, Höchst. Ger. Pat. 300,082, 2.6.15.

OCTONITROETHYLENEDIPHENYLDIAMINE, a highly-sensitive explosive having the constitution $C_{12}H_2(NO_2)_8$, $N(NO_2)(CH_2)_2N(NO_2)C_6H_4(NO_2)_3$, is prepared by the action of strong nitric acid on 2.4.2'.4'-tetranitroethylenediphenyldiamine or on 2.4.6.2'.4'.6'-hexanitroethylenediphenyldiamine. The product is practically insoluble in organic solvents, but crystallises from concentrated nitric acid in colourless rhombic prisms of m. pt. 213° C. (decomp.).

—L. A. C.

Wood cellulose and cotton; Production of material for nitration from —. C. Claessen, Berlin. Ger. Pat. 300,844, 18.7.15.

Wood cellulose and cotton are mixed together with water to a thick pulp, dried, and further mixed. The bleaching may precede or follow the wet mixing. In this way equal parts of the materials can be mixed and nitrated together so as to yield a collodion dissolving to a clear liquid and equal in stability to that obtained from nitro-cotton.—H. J. H.

XXIII.—ANALYSIS.

Liquids; Apparatus for the extraction of —. K. Brauer and E. W. Ebert. Chem.-Zeit., 1920, 44, 214.

THE apparatus is similar in shape to a Soxhlet extractor, and is intended for the extraction of a liquid with a solvent of higher sp. gr.; in place of the usual siphon is a narrow tube extending upwards from the bottom of the cylinder and entering the side tube, up which the vapour of the solvent passes. When the solvent has collected under the liquid to a height level with the point at which the narrow tube enters the side tube, it (the solvent) flows back gradually into the extraction flask. A tap is provided between the cylinder and stem of the apparatus for the purpose of drawing off the portion of the solvent which remains in the apparatus when the extraction is completed.—W. P. S.

Filtration through filter-paper; Accelerated —. G. Bruhns. Chem.-Zeit., 1920, 44, 207.

THE rate of flow through a simple folded filter-paper may be increased by turning back the loose end of the three-fold layer so that it projects into the funnel like a tongue.—W. P. S.

Precipitates; Machine for washing —. E. Sinkinson. Analyst, 1920, 45, 94—97.

A JUNG at one end of a counterpoised rocking arm supports the funnel containing the precipitate to be washed; the wash water is admitted to the funnel through a rotating jet, and when a predetermined weight of water has collected in the funnel the rocking arm falls. This movement operates a commutator connected with a motor which controls a valve on the water supply and the latter is cut off; when the water has flowed from the funnel the rocking arm rises, the motor is reversed and the valve opened so that water is again admitted to the funnel. These operations proceed automatically as long as current is supplied to the motor.—W. P. S.

Platinum wire; Cheap substitute for — for flame tests. A. Ehringhaus. Centr. Min., 1919, 192.

THE platinum wire used for carrying out flame tests can simply and cheaply be replaced by a strip of filter paper. To test a solution, a strip of pure filter paper is folded several times and one end dipped in the solution. This end is then held in the outer flame of a Bunsen burner. If an insoluble salt is to be tested the strip is first dipped in dilute hydrochloric acid and a little of the salt is then sprinkled on the damp end. In either case a good, pure flame coloration is obtained, which lasts as long as the paper is prevented from burning by the moisture or the salt. A lasting, monochromatic flame can be obtained by dipping one end of a strip of paper in a vessel containing the dissolved salt, the other end being held in the Bunsen flame. The paper is prevented from burning by a crust of salt which soon forms.—E. H. R.

Halogens; Copper flame test for — in air. A. B. Lamb, P. W. Carleton, W. S. Hughes, and L. W. Nichols. J. Amer. Chem. Soc., 1920, 42, 78—84.

ONE part of halogen (free or combined) in 100 million parts of air may be detected and approximately estimated by allowing the air to pass over a roll of fine copper gauze heated to incipient redness, and afterwards sharply heating the coil in a hot colourless flame and observing the green flame produced. The quantitative determination is made by measuring the quantity of air which must pass over the coil in order that a green flame may be produced when the coil is subsequently sharply heated.

—J. F. S.

Methyl orange; Effect of a change in temperature on the colour changes of — and on the accuracy of titrations. H. T. Tizard and J. R. H. Whiston. Chem. Soc. Trans., 1920, 117, 150—156.

The depth of colour of methyl orange in solutions of varying acidity within the limits of sensitiveness (hydron concentration 10^{-1} – 10^{-6}) was determined at three different temperatures by means of a modified Donnan colorimeter, and from the results the following values for the dissociation constant of the indicator were obtained: 10°C. , $K=2.1 \times 10^{-4}$; 25°C. , $K=3.8 \times 10^{-4}$; 40°C. , $K=5.5 \times 10^{-4}$. It is shown from these results that the concentration of hydron necessary to produce any definite depth of colour is doubled by raising the temperature from 15° to 40°C. , and consequently for the quantitative determination of hydriens in a solution by the methyl orange method it is essential to maintain a constant temperature. The effect of a change in temperature on the accuracy of an ordinary titration is, however, very slight, since, for example, the increase in concentration of hydrogen ions with increased temperature in a solution of a salt of a weak base and a strong acid tends to cancel the effect of the increased dissociation of the methyl orange.—G. F. M.

Indicators; Sodium alizarinsulphonate and other —. Mestrezat. J. Pharm. Chim., 1920, 21, 185—192.

As compared with certain other indicators (phenolphthalein, methyl orange, litmus, laemoid, Porrier's Blue, etc.) sodium alizarinsulphonate has the advantage that it may be used for the titration of ammonia and phosphoric acid in addition to being a sensitive indicator for strong acids and alkali hydroxides. It cannot, however, be used for the titration of carbonates. Commercial sodium alizarinsulphonate is always alkaline in reaction and needs to be neutralised with sulphuric acid before use as an indicator.—W. P. S.

Acids; Titration of mixed — by conductivity methods. I. M. Keltchoff. Rec. Trav. Chim., 1920 [IV.] 1, 280—302.

The author's results confirm the work of Meerburg (this J., 1919, 880 A). It is not possible to estimate lactic acid in gastric juice by the conductivity titration method, but the method is applicable to the estimation of the total acidity of the juice. It is possible by this method to estimate, with a very fair degree of accuracy either small amounts of weak acids, such as acetic acid, mixed with larger amounts of strong acids, such as hydrochloric acid, or *vice versa*. The method is thus applicable to the estimation of mineral acids in vinegar, particularly when the amount present exceeds 1% calculated as hydrochloric acid. Weak bases may similarly be estimated in the presence of strong bases by the conductivity titration method.—W. G.

Ammonia; Distillation of —. B. S. Davisson. J. Ind. Eng. Chem., 1920, 12, 176—177.

THE use of the scrubbing device of block tin with tin condenser (this J., 1919, 443 A) is recommended for the distillation of ammonia in the determination of nitric nitrogen by the method previously described (this J., 1919, 332 A). For the distillation of ammonia in Kjeldahl's method the solution is slowly distilled in this apparatus for 20 mins., about 80 c.c. of distillate being collected. The condenser is then drained, the distillation continued more rapidly for 15 mins., the receiver then removed, covered to prevent absorption of carbon dioxide, and the liquid cooled and titrated. Under these conditions the last traces of ammonia are removed from the condenser, whilst the volume of solution in the receiving flask is kept small (to prevent hydrolysis of

the indicator salt), and that of the solution in the distilling flask is not reduced.—C. A. M.

Mercury; Determination of — by Glückmann's method. A. Abelmann. Z. anal. Chem., 1919, 58, 443—445.

GLÜCKMANN'S method (see Peters, this J., 1900, 931) yields unreliable results owing to the solubility of mercury oxalate and the tendency to the formation of basic salts. This may be prevented by acidifying the mercury solution with 2–3 c.c. of 5N nitric acid, using a large excess of oxalic acid for the precipitation, and adding 50 c.c. of saturated potassium nitrate solution before the excess of oxalic acid is titrated with permanganate solution. The potassium nitrate solution should be tested previously with permanganate to ensure the absence of reducing substances.—W. P. S.

Zirconium and titanium; Separation of — as phosphates. J. Brown and H. T. Madden. J. Amer. Chem. Soc., 1920, 42, 36—39.

A SOLUTION of zirconium and titanium sulphates containing 1–2% by vol. of sulphuric acid is treated with an excess of hydrogen peroxide and then with an excess of di-sodium or di-ammonium phosphate and the mixture allowed to stand overnight. The zirconium phosphate is filtered off and washed with water containing a few drops of sulphuric acid and a little hydrogen peroxide. If the precipitate is coloured yellow by titanium it is fused with sodium carbonate, extracted with water, and re-dissolved by fusing with potassium bisulphate and then treating with dilute sulphuric acid. The zirconium is then re-precipitated as above. The phosphate is converted into oxide by fusing with sodium carbonate, extracting with water, dissolving the residue in hydrochloric acid, and precipitating with ammonia. The precipitate is washed, dried, ignited, and weighed as the dioxide. The combined filtrates from the zirconium precipitation are boiled to decompose hydrogen peroxide and the acidity adjusted so that the sulphuric acid amounts to 2% by vol. An excess of di-sodium or di-ammonium phosphate is added, the mixture allowed to stand for an hour and filtered. The titanium phosphate is washed, converted into dioxide, as in the case of zirconium, and weighed.—J. F. S.

Cuprous chloride; Preparation of a hydrochloric acid solution of — for use in gas analysis. F. C. Krauskopf and L. H. Purdy. J. Ind. Eng. Chem., 1920, 12, 158—161.

COMPARATIVE tests showed that the efficiency of cuprous chloride solution for the absorption of carbon monoxide is not deleteriously affected by the presence of relatively large amounts of stannous and stannic chlorides. A satisfactory reagent is obtained by reducing cupric chloride in acid solution by means of stannous chloride, and if a small excess of stannous chloride is used the solution may be exposed to air during transference from one vessel to another without the cuprous chloride becoming oxidised. Further, the solution may be renewed, after saturation with carbon monoxide, by heating to 60° – 70°C. to drive off the gas, a few drops of concentrated stannous chloride solution being added to correct any oxidation which might occur.—S. S. A.

Carbon dioxide, oxygen and combustible gases; Estimation of — by Krogh's method of micro-analysis. H. O. Schmit-Jensen. Biochem. J., 1920, 14, 4—24.

KROGH'S method of gas analysis may be used to estimate hydrogen and carbon monoxide in very small gas samples. Hydrogen is absorbed by a solution of 0.66 gm. of colloidal palladium and 0.42

grm. of sodium picrate in 20 grms. of water, and carbon monoxide by an acid solution of cuprous chloride. By a modification of Krogh's apparatus carbon dioxide and oxygen may be estimated by absorption, hydrogen and methane by combustion, carbon monoxide either by absorption or combustion, and nitrogen by difference. The fractional combustion of hydrogen and methane is also possible if the bubble of gas has a volume of at least 80 cub. mm. The error in the readings is about 0.1 cub. mm. (See also J. Chem. Soc., April, 1920).—J. P. S.

Oxygen; Determination of — by the copper-ammonia-ammonium chloride reagent. W. L. Badger. J. Ind. Chem., 1920, 12, 161—161.

HEMPEL's method for the absorption of oxygen by means of metallic copper spirals covered with a solution containing equal parts of saturated ammonium carbonate solution and ammonia of sp. gr. 0.93, was the basis for a series of experiments in which varying strengths of ammonia and ammonium salts other than the carbonate were used. The best results were obtained with a mixture of one part of concentrated ammonia (sp. gr. 0.9) and one of water saturated with ammonium chloride, the specific absorption (this J., 1915, 853) of this mixture being 55—60 vols. This reagent is cleaner and has a longer life than pyrogallate; unlike phosphorus, it is unaffected by catalysts, and it is readily prepared and is active at almost any temperature. On the other hand, it cannot be used for mixtures containing carbon monoxide or acetylene, and when fresh it is liable to leave measurable amounts of ammonia in the gas.—S. S. A.

Zinc; Determination of — in organic materials (urine, faeces, foods, etc.) and remarks on the zinc content of reagents and apparatus. A. Weitzel. Arb. & Reichs-Gesundh.-Amt, 1919, 54, 476—493. Chem. Zentr., 1920, 91, II., 5—6.

Two methods of determining zinc in organic materials are described. In the first the material is incinerated, the residue dissolved in acid, the solution treated with hydrogen sulphide, and the filtrate precipitated with potassium ferrocyanide. The precipitate is filtered off, washed, dissolved in concentrated sulphuric acid, and the filtrate treated in the usual way with ammonium sulphide to precipitate zinc sulphide. The latter is dissolved in hydrochloric acid and again precipitated as sulphide from an acetic acid solution. After solution of the precipitate in hydrochloric acid the liquid is treated with caustic soda and the precipitate ignited and weighed as zinc oxide. In the second method the acid filtrate from the first hydrogen sulphide treatment is oxidised with nitric acid, and the iron precipitated as basic acetate, together with any phosphate present. The precipitate is redissolved and re-precipitated two or more times until no further zinc can be detected in the filtrate. From the combined filtrates the zinc is precipitated with hydrogen sulphide, and the zinc sulphide is converted into, and weighed as, oxide, as in the first method. While both methods give equally accurate results, the second is preferable, as it uses smaller quantities of reagents and may be carried out in a shorter time. The author lays stress on the danger of contamination with zinc from the use of Jena glassware in the analysis.—A. R. P.

Ammonium molybdate; Recovery of — from phosphoric acid determinations. H. Neubauer and E. Wolferts. Z. anal. Chem., 1919, 58, 445—448.

The mixed filtrates, precipitates, etc., are neutralised partially with ammonia, the molybdic acid is precipitated at 80° C. with sodium phosphate, and the yellow precipitate is collected, washed, and dried. Four kilos. of the dry yellow powder is dis-

solved in 6 litres of water and 4 litres of ammonia (sp. gr. 0.91), and a solution of 500 grms. of crystallised magnesium nitrate is added; the total volume should be not less than 11 litres. The ammonium magnesium phosphate is removed by filtration and the filtrate is concentrated, ammonia being added from time to time to prevent dissociation and separation of molybdic acid. Crystals of ammonium molybdate are removed occasionally until the solution has a volume of about 750 c.c. On cooling, magnesium nitrate separates with the ammonium molybdate, but may be removed by washing the crystals of the latter with a small quantity of water. The ammonium molybdate thus obtained is recrystallised, first from very dilute ammonia solution and then from water.—W. P. S.

Densities; Relations between relative —, absolute density, and apparent weight of solutions. O. Chéneau. Bull. Assoc. Chim. Suér., 1919, 37, 175—181.

A TABLE is given to facilitate the conversion of specific gravities, determined at 15° C., from one basis of reference to another. One column shows absolute densities (15° C. in *vacuo*/4° C. in *vacuo*) ranging from 0.7 to 1.8, to five places of decimals, and four parallel columns show the corrections to be added or subtracted to obtain corresponding values referred to any of the following bases: 15° C. in air/15° C. in air, 15° C. in *vacuo*/15° C. in *vacuo*, 15° C. in *vacuo*/15.5° C. in *vacuo*, and 15° C. in air/4° C. in *vacuo*.—J. H. L.

Spectrophotometry; Photo-electric — by the null method. K. S. Gibson. U.S. Bureau of Standards, Scient. Paper 349, Oct. 11, 1919. 28 pages.

Catechol. Elsner. See III.

Mildew resistance of fabrics. Levino and Veitch. See V.

Flax and hemp fibres. Haller. See V.

Sodium perborate. Trickett. See VII.

Portland cement raw mixture. Witt. See IX.

Saponification values. Pardee and Reid. See XII.

Coconut and palm-kernel oils. Van Kregten. See XII.

Varnishes and boiled oils. Gardner and Coleman. See XIII.

Caoutchoucs. Harries. See XIV.

Synthetic caoutchouc. Hübener. See XIV.

Antimony sulphide in rubber. Schmitz. See XIV.

Lime liquors. Burton. See XV.

Ammonia in soil. Matthews. See XVI.

Milk. Hodgson. See XIXa.

Cheese. Geake. See XIXa.

Quinine salts. Kolthoff. See XX.

Chloroform. Utz. See XX.

Trimethyleneglycol in glycerin. Rojahn. See XXII.

Nitrocellulose. Schrimpf. See XXII.

Blown-out shots in coal-mines. Hutchison and Barab. See XXII.

PATENTS.

Water vapour; Method of and apparatus for detecting — or either of the gaseous components thereof. H. E. F. Goold-Adams and W. R. Bousfield, London, and G. W. Todd, Newcastle-on-Tyne. Eng. Pat. 137,547, 28.9.17. (Appl. 14,030/17.)

THE gases suspected of containing water vapour, or either oxygen or hydrogen (which must first be converted into water by passing over a suitable catalyst) are passed over a salt such as an alkali chloride or nitrate, which is arranged to form part of an electric circuit. If the partial pressure of the water vapour exceeds the critical hydration pressure of the particular salt selected, the salt takes up water and allows a current to pass in the circuit, whereby a suitable indication may be given by means of a galvanometer or an electric bell operated by a relay. The salt is conveniently placed between a pair of wire gauze electrodes through which the gases are passed. The device is particularly applicable in cases where it is necessary to guard against an explosive mixture of oxygen and hydrogen in gaseous reactions, such as in the synthetic production of ammonia, in which cases a continuous sampling of the mixed gases is secured by means of a by-pass, and a salt is selected, of which the hydration pressure is approximately equal to the partial pressure of the aqueous vapour corresponding to the percentage of oxygen against which a warning is required.—G. F. M.

Base-metal alloys containing chromium and nickel for thermocouples. W. C. Heraeus, G.m.b.H., Hanau. Ger. Pat. 306,359, 17.6.17. Addition to 302,452 (this J., 1920, 86A).

By the addition of varying proportions of cobalt to nickel-chromium alloys thermocouples possessing linear, convex, or concave, thermo-electric power curves can be constructed. By using alloys containing cobalt a couple can be produced having a thermo-electric power more nearly corresponding to that of the platinum-platinum-rhodium couple between 500° C. and 1300° C. than is the case in the absence of cobalt.—J. S. G. T.

[*Electric*] *resistance thermometer.* W. C. Heraeus, G.m.b.H., Hanau. Ger. Pat. 316,416, 1.4.1919.

THE resistance element is composed of boron. This possesses a temperature coefficient some 10 to 20 times greater than most metals customarily employed. Apparatus used therewith can therefore be of smaller sensitivity and consequently cheaper.

—J. S. G. T.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Adams. Conversion of liquids, fluids, and oils. 9484. Apr. 1. (U.S., 11.4.19.)

Barrett Co. Controlling temperature reactions. 9505. Apr. 1. (U.S., 11.6.19.)

Caspari. Filtering-media and diaphragms resistant to halogens and corrosive fluids. 10,093. Apr. 10.

Drees and Frischer. Causing gases and liquids to react with one another. 9989. Apr. 9. (Ger., 20.10.17.)

Fuller Engineering Co. Pulverized fuel furnaces. 9123. Mar. 29. (U.S., 1.7.19.)

Henshaw, Whittell, and Holmes and Co. Apparatus for bringing liquids and gases into intimate contact. 9432. Apr. 1.

Lamhot. Rocking-furnaces. 9297. Mar. 31. (Fr., 3.4.19.)

Lamhot. Oil-fired furnaces. 9298. Mar. 31. (Fr., 3.4.19.)

Lamhot. Rotary hearths for furnaces. 9299. Mar. 31. (Fr., 3.4.19.)

Lewcock and Siderfin. Catalytic processes and contact reactions. 9957. Apr. 9.

Littleton. 9525. See XVIII.

Metcalfe. Tunnel kilns or furnaces. 9797. Apr. 7.

Motala Verkstads Nya Aktiebolag. Furnaces for pulverulent fuel. 9147. Mar. 29.

Schmiedel. Device for bringing gases or vapours and liquids into intimate contact. 9385. Mar. 31. (Ger., 11.8.19.)

Schuler. Grinding and abrading medium for tube mills. 9285. Mar. 31.

Schuler. Centrifugal separators. 9286. Mar. 31.

Selden, and Selden Co. Catalysis. 9766. Apr. 7.

Wade (Barrett Co.). Catalytic oxidation. 9504. Apr. 1.

COMPLETE SPECIFICATIONS ACCEPTED.

21,470 (1918). Fenton and Vivian. Rotary kilns. (140,514.) Apr. 8.

3904 (1919). Sonsthagen. Means for feeding material through a rotating cylinder whilst subjected to roasting, mixing, etc. (140,880.) Apr. 14.

4276 (1919). Du Pont de Nemours and Co. Apparatus for use in recovering solvents. (123,752.) Apr. 8.

5605 (1919). Benjamin. Tunnel kilns. (140,578.) Apr. 8.

11,042 (1919). Fawcett, Preston, and Co., and Taylor. Tubular heating appliances of vacuum pans etc. (140,635.) Apr. 8.

16,383 (1919). Leinart. See XIX.

26,358 (1919). Norman, and Aluminium Plant and Vessel Co. Filter-presses. (140,715.) Apr. 8.

2581 (1920). Alexander (United Filters Corporation). Continuous vacuum filters. (141,001.) Apr. 14.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Adams. 9484. See I.

Anderson and Meikle. Apparatus for treating oil or tar. 9267. Mar. 31.

Beard. Quenching hot coke etc. 9314. Mar. 31.

De Laval Separator Co. Purifying lubricating-oil. 9676. Apr. 6. (U.S., 23.4.19.)

Jackson. Liquid fuel for internal combustion engines. 9818. Apr. 8.

COMPLETE SPECIFICATIONS ACCEPTED.

55 (1919). Broadhead. Manufacture of gas or gases. (122,179.) Apr. 14.

17,805 (1919). Soc. de Fours à Coke et d'Enterprises Indus. Coke-ovens etc. (129,994.) Apr. 8.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

- Anderson and Meikle. 9267. *See* II.
 Atack and Clough. Manufacture of compounds of the anthraquinone series. 9305. Mar. 31.
 Barrett Co. Manufacture of tar. 9506. Apr. 1. (U.S., 7,2.20.)

COMPLETE SPECIFICATION ACCEPTED.

- 17,280 (1919). Silberrad. Manufacture of dinitrophenol. (140,955.) Apr. 14.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

- Glanzfaeden A.-G. Producing very fine artificial threads from cellulose solutions. 9100. Mar. 29. (Ger., 22,7.18.)
 Jackson (Griley-Unkle Engineering Co.). Tube-beaters for paper pulp. 9488. Apr. 1.
 Rintoul and others. 10,097 and 10,098. *See* XXII.
 Wade (Barrett Co.). Manufacture of fibrous material. 9503. Apr. 1.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

- Anderson and Meikle. 9268. *See* XX.
 Boocock, and Holmes and Co. Recovering ammonia from ammoniacal liquor. 9415. Apr. 1.
 Clayton. Apparatus for manufacture of oxygen compounds of sulphur. 10,004. Apr. 9.
 Klencko and Schmiedel. Production of sulphuric acid without chambers and towers. 9386. Mar. 31. (Ger., 8,8.19.)
 Mason. Method of making bisulphite of sodium etc. 9824. Apr. 8.
 Phillips. Manufacture of alkali silicates. 9994. Apr. 9.
 Wilderman. Processes and cells for electrolytic decomposition of alkaline salts. 9341. Mar. 31.

COMPLETE SPECIFICATIONS ACCEPTED.

- 5979 (1918). Taliani. Catalytic oxidation of ammonia. (140,833.) Apr. 14.
 17,167 and 19,135 (1918). Calvert. Production of cyanogen compounds or cyanide. (140,484.) Apr. 8.
 19,585 (1918). Hood. Purification of sulphur. (140,844.) Apr. 14.
 19,940 (1918) and 13,816 (1919). Chrystal. Recovery of ammonia. (140,505.) Apr. 8.
 21,533 (1918). Soc. Indus. de Produits Chimiques. Converting alkali monochromates into bichromates. (122,172.) Apr. 8.
 14,958 (1919). Norsk Hydro-Elektrisk Kvaelfstoffaktieselskab. Sulphuric acid containing nitrogen oxides. (130,966.) Apr. 8.

VIII.—GLASS; CERAMICS.

APPLICATION.

- Assié. Manufacture of an abrasive and refractory material. 9685. Apr. 6. (Fr., 9,8.17.)

IX.—BUILDING MATERIALS.

APPLICATIONS.

- Phillips. Heat-insulating materials. 9995. Apr. 9.
 Simon. Process for decolorising veneers of exotic woods. 9256. Mar. 30. (Fr., 9,1.18.)
 Smitheman. Brick-making process. 9161. Mar. 30.

COMPLETE SPECIFICATION ACCEPTED.

- 3470 (1919). Hoare. Utilising slag as a building and other constructional material. (140,874.) Apr. 14.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

- Alexander (Metal and Thermit Corporation). Formation of tungsten alloys. 9633. Apr. 6.
 Amalgamated Zinc (de Bavay's), Ltd. Recovery of lead and silver from ores etc. 9149. Mar. 29. (Australia, 29,3.19.)
 Dean. 9483. *See* XI.
 Electrolytic Zinc Co. Electrolytic treatment of ores containing zinc etc. 9445. Apr. 1. (Australia, 4,4.19.)
 Lovelock, and Try and Son. Electrolytic deposition of iron, and treatment of the deposits. 9251. Mar. 30.
 Metropolitan-Vickers Electrical Co. Treating magnetisable material. 9651. Apr. 6. (U.S., 4,4.19.)
 Pearson and Saunders. Furnace for roasting and smelting ores. 10,041. Apr. 10.
 Syndicat Electro-Staal, and Vermaes. Manufacture of iron from iron ores. 9250. Mar. 30.
 Talbot. Metallurgical furnaces. 9455. Apr. 1.

COMPLETE SPECIFICATIONS ACCEPTED.

- 10,997 (1918). Marsh. Treatment of metals or refractory materials, and electric furnaces therefor. (140,835.) Apr. 14.
 21,068—21,070 (1918). Soc. Anon. de Commentry Fourchambault et Decazeville. Alloys. (140,507—9.) Apr. 8.
 14,989 (1919). Tainton. Roasting ores or concentrates. (140,943.) Apr. 14.
 16,588 (1919). Sinclair. Metal-smelting furnaces. (140,948.) Apr. 14.
 23,747 (1919). Soc. des Alliages et Bronzes Forgeables. Tilting-crucible or melting-pot furnaces. (140,709.) Apr. 7.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

- Assié. Electric furnaces. 9686. Apr. 6. (Fr., 9,8.17.)
 Dean. Compound anode for electroplating. 9483. Apr. 1. (U.S., 12,12.17.)
 Electrolytic Zinc Co. 9445. *See* X.
 Lovelock, and Try and Son. 9251. *See* X.
 Moore. Electric furnaces. 9997. Apr. 9.
 Wilderman. 9341. *See* VII.

COMPLETE SPECIFICATIONS ACCEPTED.

- 10,997 (1918). Marsh. *See* X.
 19,388 (1918). Elison. Electric accumulator plates. (140,500.) Apr. 8.
 4382 (1919). Harris. Electrolytic cell. (140,563.) Apr. 8.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

- Borgen. 9853. *See* XIX.
 Erslev. Treatment of oil-containing vegetable materials. 9314. Mar. 31. (Holland, 9.4.19.)
 Scott and Whitehead. Extraction of oils from vegetable matter. 9798. Apr. 7.
 Tseng. Manufacture of soap. 9913. Apr. 9.

COMPLETE SPECIFICATION ACCEPTED.

- 21,410 (1918). Engel. Oil-extraction apparatus. (140,513.) Apr. 8.

XV.—LEATHER; BONE; HORN; GLUE.

COMPLETE SPECIFICATION ACCEPTED.

- 9874 (1919). O'Gorman and Schryver. Manufacture of adhesives. (140,911.) Apr. 14.

XVI.—SOILS; FERTILISERS.

COMPLETE SPECIFICATION ACCEPTED.

- 11,019 (1919). Jones, and Newell and Co. Apparatus for the manufacture of superphosphate manures. (140,915.) Apr. 14.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

- Jarraud. Ageing spirits and alcohol liquors. 9321. Mar. 31. (Fr., 17.4.19.)
 Littleton. Air-pressure filtering apparatus for pressing yeast etc. 9525. Apr. 1.
 Scott. Skimming yeast from fermentation vessels and separating fluid therefrom. 9920. Apr. 9.

COMPLETE SPECIFICATIONS ACCEPTED.

- 224 (1919). Pascal. Production of denatured alcohol. (140,527.) Apr. 8.
 16,421 (1919). Crossman. Apparatus for mash filtration. (140,946.) Apr. 14.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

- Borgen. Making margarine. 9853. Apr. 8.
 Candy. Purification of water. 9808. Apr. 8.
 Forget-Me-Not Flours, Ltd., and Hutchinsonson. Process for ageing flour. 9242. Mar. 30.
 Jones, Watson, and Woodlands, Ltd. Manufacture of flour and bread. 9086. Mar. 29.

COMPLETE SPECIFICATIONS ACCEPTED.

- 17,399 (1918). Cullen and Fulton. Manufacture of an egg substitute. (120,396.) Apr. 8.
 19,673 (1918). Erslev. Manufacture of artificial milk. (121,133.) Apr. 14.
 16,383 (1919). Lenart. Apparatus for purifying liquids. (140,677.) Apr. 8.

- 22,429 (1919). Alberts. Manufacture of cocoa powder. (140,703.) Apr. 8.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

- Anderson and Meikle. Distillation of organic acids. 9268. Mar. 31.
 Dufrasse and Moureu. Preparation of condensation products of acrolein. 9307 and 9308. Mar. 31. (Fr., 31.3.19.)
 Dufrasse, Moureu, Pougnet, and Robin. Stabilisation of acrolein. 9826. Apr. 8. (Fr., 8.4.19.)
 Imray (Soc. Chem. Ind. in Basle). Manufacture of mercury compounds of glucosides. 9667. Apr. 6.
 Lepape and Moureu. Manufacture of acrolein. 9306. Mar. 31. (Fr., 31.3.19.)
 Lowy Laboratory Inc. Arsenical compounds. 10,031. Apr. 9. (U.S., 4.10.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 2916 (1917). Haddan (Commercial Research Co.). Manufacture of chlorhydrins. (140,831.) Apr. 14.
 14,731 (1917). Marks (Bosshard). Preparation of glyoxal. (140,478.) Apr. 8.
 18,686 (1919). Chem. Fabr. vorm. Sandoz. Isolation of hyoscyamine. (131,283.) Apr. 8.
 21,281 (1919). Marks (Nitritfabrik A.-G.). Preparation of pyrogallie acid. (140,694.) Apr. 8.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

- Kann, and Octophototype Synd. Emulsion for photographic processes etc. 9678. Apr. 6.
 Scott. Photography. 9878. Apr. 8.

COMPLETE SPECIFICATION ACCEPTED.

- 4053 (1919). Wellesley and Sanders. Production of photographic films in natural colours. (140,560.) Apr. 8.

XXII.—EXPLOSIVES; MATCHES.

APPLICATION.

- Rintoul, Griffiths, Nolan, and Nobel's Explosives Co. Manufacture of nitrocellulose plastics and solutions. 10,097 and 10,098. Apr. 10.

COMPLETE SPECIFICATIONS ACCEPTED.

- 21,170 (1919). Hedberg. Manufacture of explosives. (140,967.) Apr. 14.
 21,433 (1919). Atlas Powder Co. Explosive compounds. (134,523.) Apr. 8.

XXIII.—ANALYSIS.

APPLICATION.

- Guillery. Testing mechanical properties of materials. 9146. Mar. 29. (Fr., 28.3.19.)

I.—GENERAL; PLANT; MACHINERY.

Graphic mixture calculations. Wa. Ostwald. Chem.-Zeit., 1920, 44, 241—242.

WHEN it is required, from two or more materials containing two or more constituents in known proportions, to produce another mixture containing the same constituents in other proportions, the calculation can be greatly simplified by the use of the graphic method. In the simplest case, for example, where it is required from mixtures containing respectively 2.5% and 10% of a constituent *a* to prepare one containing 8.25% of *a*, a horizontal percentage scale is plotted and at its extremities are erected perpendiculars on which are set off the proportions of *a* present in the two original materials. The two points thus obtained are joined by a straight line. On this line a point is found, the perpendicular distance of which from the horizontal line corresponds to 8.25%, and from this point a perpendicular is dropped which divides the horizontal line in the ratio in which the two original materials must be taken to give the required mixture. The method can be applied to problems of considerable complexity, and gives an approximate result when no exact solution is possible. (See also Donnan; this J., 1916, 3.)

E. H. R.

Filter plates and frames; Classification of — and their general usage. D. R. Sperry. Chem. and Met. Eng., 1920, 22, 493—496.

PATENTS.

Conducting a liquid and gas soluble in said liquid through a closed cycle of thermal operations; Apparatus for —. C. H. Beadle, Southampton. Eng. Pat. 139,732, 24.9.19. (Appl. 23,491/19.)

AN apparatus is described for rendering heat energy available as mechanical energy or for purposes of refrigeration by means of the cycle of operations described in Eng. Pat. 20,267 of 1914. Gas is evolved from a liquid under pressure by means of heat transferred from liquid and gas at another part of the cycle, the evolved gas does mechanical work in an engine and the heat thus lost is regained from an external source, which may be waste or natural heat; the reheated gas is absorbed in the liquid, and the latter then passes to the generator where gas is evolved. A single vessel, preferably consisting of a number of vertical concentric tubes, is used for the absorption and evolution of gas and the transference of heat. An automatic valve is provided to maintain the level of the liquid in a dome at the top of the gas-effusion tube and to regulate the flow of exhausted liquid from the high to the low pressure zone, a reservoir is provided to form a store of recuperated liquid, and a pump to return this liquid to the evolution chamber.

—B. M. V.

Refrigerating apparatus. C. H. Worthen, Beaver, Pa., Assignor to E. E. Worthen, Wellsburg, W. Va. U.S. Pat. 1,332,703, 2.3.20. Appl., 19.2.17.

LIQUID anhydrous ammonia expands through a nozzle at the lower part of an expansion coil; the mixed vapour and unevaporated liquid are led from the upper part of the coil to a separation chamber, whence the residual liquid passes down a pipe jacketing the pipe which supplies the original liquid ammonia, the latter being thus chilled; the residual liquid ammonia is drawn into the expansion coil at the nozzle together with the fresh liquid ammonia.—B. M. V.

Rings for filling absorption towers, distillation columns, or the like. R. Lessing, London. Eng. Pat. 139,880, 27.2.19. (Appl. 4967/19.)

EACH ring is provided with a diametrical partition which may extend the whole or only part of the way across, but must not be joined to the ring in such a way as to interfere with the flow of liquid when the ring is in a horizontal position.—W. H. C.

Filling material for absorption and reaction towers. Prym and Co., Buisbach. Ger. Pat. 317,166, 11.2.18.

THE filling is in the form of tubes made by bending sheet metal. One end of the tube is turned inwards so as to increase the surface, but not the length or diameter. The inner projection, which may be also welded or soldered on, may be corrugated or even worked into the form of a tube.—H. J. H.

Furnace; Producer gas —. C. K. E. Bildt, Norrviken, Sweden. Eng. Pat. 139,957, 22.5.19. (Appl. 12,923/19.)

FOR heating boilers etc. with solid fuel a small proportion of air is admitted through the fuel bed in a separate furnace, thus forming producer gas, and combustion is then completed by secondary air. To prevent the harmful effects on the boiler proper of the intense heat of the producer-gas flame, the combustion chamber of the furnace is surrounded by water tubes which lie close to each other and are mostly straight, though some may be bent to form openings for the entrance and exit of gases. The water-boxes in which the tubes are secured should be inaccessible to the flame, and the one next the boiler proper may be annular, thus forming an outlet for the flame.—B. M. V.

Furnace heating; Method of —. W. E. Renner, Somerville, Mass. U.S. Pat. 1,332,681, 2.3.20. Appl., 30.7.19.

A LARGE part of the gaseous contents of a furnace is allowed to circulate through a conduit leading from the furnace, past jets for supplying additional air and atomised combustible mixture, then back to the furnace. The jets are so inclined as to promote the circulation, the combustible mixture being supplied just before the gases re-enter the furnace.

—B. M. V.

Rotary furnaces; Composite lining for —. W. S. Rockwell, Assignor to W. S. Rockwell Co., New York. U.S. Pat. 1,333,313, 9.3.20. Appl., 8.9.19.

THE shell of a rotary furnace is lined with refractory material and a spiral metallic rib, formed in sections, projects through the lining from the shell into the inner cylindrical space and forms a feeding channel therein.—W. H. C.

Gas furnace. G. W. McKee, Rockford, Ill. U.S. Pat. 1,331,309, 23.3.20. Appl., 18.12.17.

FUEL gas is delivered tangentially into a row of cylindrical combustion chambers opening at the top into a common horizontal chamber which constitutes the heating zone.—W. F. F.

[Electrically] precipitating suspended material from gases. E. R. Wolcott, Assignor to International Precipitation Co., Los Angeles, Cal. U.S. Pat. 1,331,225, 17.2.20. Appl., 28.5.19.

THE material to be deposited is rendered conductive by injecting into the gases a finely-divided solid conducting substance, the latter being deposited with the suspended material.—B. N.

Electrodes of electric precipitators; Arrangement for connecting —. Siemens-Schuckertwerke G.m.b.H., Siemensstadt. Ger. Pat. 314,626, 6.12.18.

THE high-tension electrodes hang vertically from horizontal plates suspended by insulators from the roof of a gas duct. The precipitation electrodes rest on the floor of the duct, and are placed vertically between the high-tension electrodes, so that dead space is reduced to a minimum.—H. J. H.

Electrical precipitator with permeable electrodes. Siemens-Schuckertwerke G.m.b.H., Siemensstadt. Ger. Pat. 314,947, 4.6.18.

THE electrode on which the deposit collects consists of two or more individual electrodes of gauze or the like in electrical connection, but sufficiently widely separated to protect other metallic parts from the electric field. This is done to avoid deposition of material upon other parts such as the supports of the electrodes and the walls of the gas duct.—H. J. H.

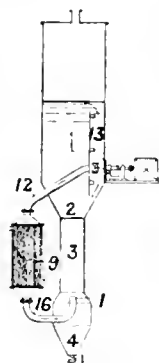
Electrical precipitation plant; Process for removal of dust from the settling surfaces of —. H. Pünig, Münster. Ger. Pat. 315,531, 28.10.17.

By means of a whistle, syren, or similar appliance sound waves are set up in the precipitation chambers with sufficient intensity to dislodge the dust which has settled out upon the surfaces.—H. J. H.

Evaporating and crystallising apparatus especially adapted for obtaining large crystals. C. Prache, Paris. U.S. Pat. 1,331,373, 17.2.20. Appl., 29.12.17.

THE apparatus comprises a vertical receptacle, 2, which is constricted in its lower part, 3, in which the crystals are separated. The solution is circulated downwards through the pipe, 12, by a forcing device in the space, 13, and passes through a heater, 9, into the pipe, 16, from which it is delivered at the outlet, 1. The small crystals in the space, 3, are thus suspended in the boiling solution until they grow to a predetermined size, when they fall into the space, 4.

—W. F. F.



Evaporator. C. F. Braun, San Francisco, Cal. U.S. Pat. 1,331,014, 16.3.20. Appl., 4.9.18.

THE evaporator is heated by a system of steam coils which are connected at one end to a supply and at the other to an exhaust manifold formed one on each side of a door that serves to close the opening through which the coils are introduced.—W. H. C.

Dryer and evaporator. C. Offenhauser, Philadelphia, Pa. U.S. Pat. 1,333,871, 16.3.20. Appl., 15.2.19.

A HORIZONTAL cylindrical drying drum is supported at its two ends which project through the two ends of an outer drum. The ends of the outer drum are attached to the inner drum, but means are provided for rotating the cylindrical shell of the outer drum. Hot gas is passed through the space between the drums to heat the inner drum, and an exhaust opening is provided in the upper part of one of the ends of the outer drum.—W. F. F.

Drying liquids; Apparatus for —. O. S. Sleeper, Assignor to Buffalo Foundry and Machine Co., Buffalo, N.Y. U.S. Pat. 1,331,389, 17.2.20. Appl., 25.4.16.

A DRYING drum, supported in horizontal bearings,

dips into the liquid to be dried, which is contained in a pan. The end walls of the pan are in contact with the drum, and the front and rear walls of the pan are adjustable vertically so that they may be in contact with the cylindrical wall of the drum. The liquid is circulated through a pipe, the inlet of which is in the bottom of the pan and the outlet at the top, close to the periphery of the drum.—W. F. F.

Dryer. L. R. Christie, Pittsburgh, Pa. U.S. Pat. 1,332,380, 2.3.20. Appl., 6.1.19.

THE dryer consists of a rotary drum having an outer and an inner shell. The heating gases pass through the inner shell, and the material to be dried passes through the annular space between the shells. Means are provided in the annular space for lifting the material and depositing it on the outer surface of the inner shell.—W. H. C.

Dryer. B. R. Andrews, Braintree, Mass. U.S. Pat. 1,322,657, 2.3.20. Appl., 25.1.18.

THE material is supported on trays which extend transversely across a drying tunnel. The tunnel is provided with baffles on opposite sides to cause the current of air which is passed through it to take a circuitous course. Means are provided to prevent the current of air from passing through any space left between the edge of the baffles and the material.—W. H. C.

Drying process and apparatus. F. P. Boland, Providence, R.I. U.S. Pat. 1,332,506, 2.3.20. Appl., 15.1.19.

A DRYING medium such as air is passed in succession through a small heated area and twice through the material to be dried.—B. M. V.

Dryer; Centrifugal —. G. H. Elmore, Swarthmore, Pa. U.S. Pat. 1,334,023, 16.3.20. Appl., 7.10.16.

A ROTARY annular screen mounted on a vertical shaft is cleaned by means of internal scrapers which are mounted to rotate coaxially with the screen. The scrapers are periodically and automatically moved towards and away from the screen, but out of contact with it.—W. F. F.

Filter-press. A. L. Bausman, Chicopee, Mass., Assignor to National Equipment Co., Springfield, Mass. U.S. Pat. 1,331,453, 17.2.20. Appl., 2.10.18.

THE filter-plates are pressed together by means of a hydraulic plunger, the face of which abuts against a filter-plate. A filtering cloth is arranged on the outside face of each plate, and is pressed against the plate by a collecting ring. A plunger ring surrounds and protects the edges of the cloth, and a packing is provided between the plunger ring and the plunger plate which is independent of the filtering cloth. A spacing device is provided between an abutment on the plunger plate and the plunger ring by means of which the plunger may be allowed to enter the plunger ring and press against the collecting ring to effect filtration or may be prevented from doing so.—W. F. F.

Filter-press device. G. F. Miller, New York. U.S. Pat. 1,333,869, 16.3.20. Appl., 7.6.19.

A FILTER-PRESS frame is provided with recesses in its two faces into which removable members carrying the filter cloths are fitted.—W. F. F.

Bag-filter. E. W. Deming, New York. U.S. Pat. 1,331,692, 23.3.20. Appl., 2.11.16.

THE filter is formed as an open-ended tubular member, and means are provided for supplying the liquid to be filtered, a washing liquid, and a gas, to both ends of the filter.—W. F. F.

Cooling apparatus [for gases]. I. Hechenbleikner, Assignor to Chemical Construction Co., Charlotte, N.C. U.S. Pat. 1,331,500, 24.2.20. Appl., 17.10.18.

AN apparatus for cooling gas comprises a vertical cylinder containing the gas, which is inserted into the open top of a surrounding tube which is closed at the bottom and which is of slightly larger diameter than the cylinder. The cooling liquid is supplied by means of a central vertical tube passing downwards through the cylinder and having its outlet near the closed bottom of the outer tube, so that the cooling liquid passes upwards around the cylinder and overflows at the upper end of the outer tube.—W. F. F.

Centrifugal machine. T. A. Bryson, Troy, N.Y., Assignor to Tolhurst Machine Works. U.S. Pat. 1,331,692, 24.2.20. Appl., 31.12.17.

A CENTRIFUGAL basket open at the top is surrounded by a casing which receives the liquid, and is provided with a bottom closure extending inwards to meet the perforated bottom of the basket. The top of the casing is closed by movable lids, and means are provided for heating the space within the casing.—W. F. F.

Centrifugal separators; Means for controlling the proportion of liquids separated in —. A. J. E. Munters, Assignor to Aktiebolaget Pump-Separator, Stockholm, Sweden. U.S. Pat. 1,331,779, 24.2.20. Appl., 21.12.18.

THE drum of the separator rotates on a vertical axis, and an outlet slot is provided in the neck of the apparatus which projects upwards, and is surrounded by a ring having a peripheral recess co-operating with the slot.—W. F. F.

Impact-pulveriser; Rotary —. C. J. Tomlinson, West Allis, Wis., Assignor to Allis-Chalmers Manufacturing Co., Milwaukee, Wis. U.S. Pat. 1,331,969, 24.2.20. Appl., 23.12.15.

A PAIR of rotors are arranged in a casing and are provided with peripheral projections which impact upon one another during rotation. Means are provided for feeding the material first to one of the rotors only, and then to the space between the co-operating impact surfaces.—W. F. F.

Comminuting-machine. O. Kutsche, Pittsburg, Pa. U.S. Pat. 1,332,220, 2.3.20. Appl., 12.9.18. Renewed 7.7.19.

THE material is crushed by grinding balls in a race or chamber supported by a rotating shell, the balls being kept in position by fixed means carried from above by an outer dust-proof casing.—W. H. C.

Comminuting apparatus. O. Kutsche, Pittsburgh, Pa. U.S. Pat. 1,332,850, 2.3.20. Appl., 10.10.16. Renewed 18.7.18.

A ROTATING base is provided with balls having a contour to fit the base. Material to be ground is fed to the centre of the base, and air under pressure is introduced simultaneously, while the ground material is removed at a lower pressure.—A. E. D.

Pulverising mill. G. F. Hurt, New York. U.S. Pat. 1,334,701, 23.3.20. Appl., 31.5.17.

THE bottom of the grinding chamber is formed as a hollow neck extending upwards into the chamber, and a driving shaft extends upwards through the neck, with an annular space between. The shaft carries revolving grinding rollers, co-acting with a fixed grinding ring, and air is circulated through the grinding chamber so as to draw the fine suspended material through the annular passage in the neck and prevent it from collecting on the moving parts of the mill.—W. F. F.

Waste-heat boiler. J. E. Bell, Brooklyn, N.Y., Assignor to The Babcock and Wilcox Co., Bayonne, N.J. U.S. Pat. 1,332,284, 2.3.20. Appl., 3.2.15.

WASTE gases from an industrial furnace are repeatedly passed vertically up and down over the inclined tubes of a water tube boiler set in the waste gas flue, and provided with cross-baffles so that practically all the waste heat is transferred to the boiler. Suction is provided so that at least 2000 lb. of waste gases per hour are provided per square foot of flow area.—A. E. D.

Gaseous compounds; Process of and apparatus for the oxidation and reduction of —. W. Koehler, Cleveland, Ohio. U.S. Pat. 1,332,730, 2.3.20. Appl., 8.10.18.

VARIOUS gases are mixed at a common point (e.g., by converging jets) and their temperature raised at that point (e.g., by an electric arc).—B. M. V.

Percolating and concentrating apparatus. J. U. Lloyd, Cincinnati, Ohio. U.S. Pat. 1,332,908, 9.3.20. Appl., 6.6.18.

FLUID menstruum circulates downward through a percolator and thence by a pipe to the bottom of a concentrator, which is surrounded by a series of jackets adapted to receive either steam or water. The fluid leaves the concentrator at the top and passes into the top of a condenser, from the bottom of which it passes by a pipe back to the top of the percolator.—W. F. F.

Oxidising apparatus [; Comminuting, rabbling, and —]. J. R. MacMillan, Assignor to Niagara Alkali Co., Niagara Falls, N.Y. U.S. Pat. 1,333,327, 9.3.20. Appl., 20.12.18.

AN oxidising chamber having a smooth flat hearth is mounted above and heated by a furnace. A grinding and stirring element carried by a rotating vertical shaft traverses the hearth and grinds the material on it.—W. F. F.

Liquefaction and separation of gas mixtures such as air; Process and apparatus for —. R. Mewes, Berlin. Ger. Pat. 315,511, 5.9.16. Addition to 290,080.

A PORTION of one of the components is withdrawn from the separation chamber or from another suitable part of the apparatus, somewhat compressed, and then returned in countercurrent to the separation chamber. After giving up heat, the gas being treated is expanded with partial liquefaction by being sprayed into the chamber, effecting some cooling here by admixture. The higher-boiling fraction, after passing through such a cycle, is sprayed into the separation chamber at the point where the fresh mixture enters, whilst the lower-boiling fraction is sprayed into the coldest portion of the chamber. From the high-pressure compressor a gas is drawn which is free from water and carbon dioxide.—H. J. H.

Filling containers with a definite quantity of liquefied gas, e.g., sulphur dioxide; Process for —. G. Giemsa, Hamburg. Ger. Pat. 315,659, 23.6.18.

THE container has separate inlet and outlet openings, the latter being furnished with a siphon tube, the lower end of which marks the desired level of the liquid in the container. In this way the necessity for weighing the liquefied gas is avoided, as also the risk of over-filling the vessel.—H. J. H.

Catalysing material. The British Thomson-Houston Co., Ltd., London. From General Electric Co., Schenectady, N.Y., U.S.A. Eng. Pat. 140,011, 8.9.19. (Appl. 22,076/19.)

SEE U.S. Pat. 1,299,641 of 1919; this J., 1919, 415 A.

Fibrox (silicon oxycarbide) may be used as a support for other catalysts, e.g., platinum, in addition to nickel.

Heat exchanger, especially for firing installations. L. Honigmann, Aix-la-Chapelle. Ger. Pat. 314,782, 3.7.17.

Mixing and kneading machines. F. Aeschbach A.-G., Aarau, Switzerland. Eng. Pat. 124,743, 20.3.19. (Appl. 7022/19.) Int. Conv., 20.3.18.

Mixing and sifting powdered and granulated substances; Apparatus for —. B. Young, London. Eng. Pat. 139,597, 26.2.19. (Appl. 4765/19.)

Electric precipitators; Device for protecting insulators in —. A. F. Meston, Assignor to Research Corporation, New York. U.S. Pat. 1,332,510, 2.3.20. Appl., 6.3.18.

Furnaces. A. B. Chantraine, Marcinelle, Belgium. Eng. Pat. 18,471, 8.8.14.

SEE Fr. Pat. 475,649 of 1914; this J., 1916, 53.

[Electrically] separating suspended material from gases; Process of — and apparatus therefor. A. Mond, London. From International Precipitation Co., Los Angeles, Cal., U.S.A. Eng. Pat. 136,464, 27.5.19. (Appl. 13,336/19.)

SEE U.S. Pats. 1,329,737 and 1,329,817—8 of 1920; this J., 1920, 257 A.

Electrical separation of substances in suspension in fluids; Apparatus for —. G. Gallot, Paris, and P. Poussin, Pantin, Assignors to Soc. Purification Industrielle des Gaz, Paris. U.S. Pat. 1,332,981, 9.3.20. Appl., 21.5.18.

SEE Eng. Pat. 116,104 of 1918; this J., 1920, 196 A.

Separating [emulsions of] hydrocarbons and water; Process for —. W. A. Brown and F. G. White, Los Angeles, Cal., U.S.A. Eng. Pat. 139,417, 22.8.19. (Appl. 20,689/19.)

SEE U.S. Pat. 1,309,794 of 1919; this J., 1919, 621 A.

Filtering fluids; Devices for —. F. K. and E. F. Atkins, Harrison, N.J., U.S.A. Eng. Pat. 139,991, 8.7.19. (Appl. 17,059/19.)

SEE U.S. Pat. 1,273,127 of 1918; this J., 1918, 566 A.

Drying process and apparatus therefor. F. P. Boland, Providence, R.I., U.S.A. Eng. Pat. 140,145, 31.12.18. (Appl. 21,927/18.)

SEE U.S. Pat. 1,294,035 of 1919; this J., 1919, 282 A.

Evaporator. E. A. Barbet, Assignor to E. Barbet et Fils et Cie., Paris. U.S. Pat. 1,325,461, 16.12.19. Appl., 28.6.15.

SEE Eng. Pat. 22,844 of 1914; this J., 1916, 293.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal dust in steel mills; Explosions of —. L. D. Tracy. Chem. and Met. Eng., 1920, 22, 422—423.

THE use of pulverised coal as fuel may constitute a serious fire or explosion risk if suitable precautions are not adopted. Fans which drive the dust from the storage bins to the furnace should not be stopped before the pipe line has been thoroughly blown out, as a back draught may carry live sparks into the pipes. Contact of the coal dust with hot metal is to be avoided, and accumulation of dust in the building should be prevented, the use of naked lights during cleaning being forbidden.

—W. J. W.

Low-grade fuels; Firing with —. E. Haack. Brennerzeit., 1919, 36, 8429—8430, 8435—8436. Chem. Zentr., 1920, 91, II., 163.

CHIMNEY stacks have usually been erected to deal with high-grade fuels, and do not give sufficient draught where lower qualities are to be burned, although they suffice for the removal of the products of combustion where forced draught is employed. In new construction it is advisable to build chimneys capable of dealing with low-grade fuel. The air supply to the fuel may be assisted by jets of steam or air, the former being simpler. For inducing draught by steam, however, 5–6% of the steam generated by high-grade fuel is consumed as against 1–1.5% for compressed air.—H. J. H.

Lignite; Separation of — into different groups of substances, their characters and behaviour on dry distillation. W. Schneider. Ges. Abhandl. Kenntn. Kohle, 1919, 3, 325—357. Chem. Zentr., 1920, 91, II., 99—101.

LIGNITE from the Riebeck Montan works yielded 15% of its dry substance as "Bitumen A" on extraction with benzene at 70°–75° C. and 8% as "Bitumen B" on subsequent extraction with benzene at 250°–260° C., the residue constituting 77%. The bitumen A was further divided, by extraction with alcohol and ether, into 2% of a dark resinous substance and 13% of true montan wax. Bitumen B somewhat resembled the resin in appearance, being almost black and transparent in thin layers; in composition and solubility, however, it more closely resembled montan wax. It melted indefinitely about 75° C. The composition and properties of all these fractions are given in tables, and also the amounts and characters of the products produced by their dry distillation. Nearly one-half of the total amount of tar (including the major part of the paraffin) yielded by the original lignite was attributable to the montan wax fraction, bitumen B and the residue accounting each for about one-quarter of the total. By repeated extraction of the residue with boiling 5% sodium hydroxide solution 80% (and by a single extraction for 3 hours at 200° C. in an autoclave 84%) was obtained in solution in the form of humic acids precipitable on acidifying the solutions. The residue insoluble in alkalis contained 36% of ash and 0.47% of nitrogen, had a methyl value of 1.7, and yielded 17.2% of tar on distillation. All lignites appear to yield their major part in a soluble form on heating with alkalis, and this part does not contribute appreciably to the production of tar on distillation.—J. H. L.

Oil from compressed oil-gas. J. C. Mansukhani and J. J. Sudborough. J. Indian Inst. Sci., 1918, 2, 73—77.

Gas from a low-grade kerosene oil is compressed for use as an illuminant by the Indian railway companies. A sample of the volatile oil ("hydrocarbon") deposited during the compression yielded about 9% of olefine hydrocarbons, 15.2% of benzene, and 7% of toluene, both of the latter giving nitro compounds of good colour. No paraffin hydrocarbons were present. About 7000 galls. of this oil is produced per month in India.—C. A. M.

Petroleum oils; Rapid determination of sulphur in —. A. W. Christie and C. S. Bisson. J. Ind. Eng. Chem., 1920, 12, 171—172.

ABOUT 0.5 grm. of the oil is burnt in a calorimetric bomb, and the contents of the latter are diluted with water, filtered, and titrated with standard alkali solution. A portion of the solution, equivalent to about 1.5 mgrm. of sulphur, is diluted to 25 c.c., treated with 1 drop of dilute hydrochloric acid and 10 c.c. of 0.8% benzidine hydrochloride solution. The benzidine sulphate is collected, after 15 mins., on an asbestos filter, washed with cold water, then

dissolved in hot water and 1 c.c. of 10% sodium hydroxide solution, the solution diluted to 100 c.c., 5 c.c. of concentrated sulphuric acid added, and the benzidine titrated hot with $N/20$ permanganate solution. A slight excess of the latter should be added, then a slight excess of $N/20$ oxalic acid solution, and the titration completed with permanganate. The number of c.c. of permanganate solution required to oxidise the benzidine is multiplied by 0.041 to give mgrms. of sulphur in the portion of solution taken for the precipitation.—W. P. S.

Aromatic hydrocarbons in petrol; Methods of estimation of —. D. Florentin and H. Vandenberghe. Bull. Soc. Chim., 1920, 27, 204—209.

To 20 c.c. of the petrol, in a flask cooled in ice, nitric acid (sp. gr. 1.5) is added, drop by drop, with shaking, until a brown tint is no longer produced, and the mixture is then left for fifteen minutes. The contents of the flask are poured into a large excess of water and neutralised with sodium hydroxide. By steam distillation small amounts of impurities arising from the oxidation of impurities in the petrol are removed, and then the petrol is decanted. The aqueous solution is extracted twice with light petroleum spirit, and the extracts are added to the decanted petrol, this mixture then being made up to 100 c.c. with more light petroleum spirit. An aliquot portion is evaporated in a small glass dish *in vacuo* over sulphuric acid to constant weight (or very slight constant loss in weight) the residue being the nitro derivative. The weight of this, multiplied by 0.634, gives the weight of benzene. Alternatively an aliquot portion may be heated with titanous chloride for two hours under a reflux condenser, and the residual titanous chloride titrated with a solution of ferric salt (this J., 1920, 224 A). The second method is rapid and exact, and is preferable to the first when the petrol contains any appreciable quantity of high boiling-point fractions. The examination of a number of samples of commercial motor spirit shows that they contain appreciable quantities of benzene hydrocarbons, particularly toluene.—W. G.

Lubricating oils; Control of —. R. Dubrisay. Ann. Falsif., 1920, 13, 25—33.

The general requirements of a good lubricating oil are mentioned, and the limits given in literature and in commercial specifications for various lubricating oils are recorded. An apparatus described previously by the author (this J., 1917, 1123) may be used for determining the viscosity or fluidity of an oil (see also this J., 1918, 499 A; 1919, 4 A).

—W. P. S.

Paraffin wax; Influence of high temperatures on —. H. Burstin and W. Jakubowicz. Petroleum, 1919, 15, 189—192. Chem. Zentr., 1920, 91, 11., 213.

COMMERCIAL paraffin wax, m.p. 51.5°C ., from petroleum, when heated at 150°C . in a drying oven for 70 hours, lost 77.99% in weight and was carbonised. Another sample, m.p. 57°C ., lost 79.44% in 56 hours. Even after 3 hours in the drying oven at 125°C . the loss in weight of a normal paraffin wax was over 5%. When the access of air was prevented the loss in weight on heating at 125°C . was only 0.49% in 22 hours, and the wax remained white. The loss was still less when carbon dioxide was passed over the paraffin wax, and amounted to only 0.11% after 16 hours. In industrial work the distillation of paraffin wax should be conducted under vacuum with the exclusion of atmospheric oxygen as completely as possible.—J. F. B.

Asphalt rock. Kröhnke. See IX.

Carbon black. Neal. See XIII.

Chloroform from coke-oven gas. Dey. See XX.

PATENTS.

Coal washing plant. C. Rogerson, Hamilton, Scotland. Eng. Pat. 139,612, 4.13.19. (Appl. 5286/19.)

COMPLETE separation of the wash water from the "small" is effected by the use of perforated conveyors, one above the other, along which the coal is caused to travel in opposite directions, the coal being subsequently moved along sheets and withdrawn whilst the water flows in the transverse direction. The larger coal from the upper conveyor is transferred at one end to the lower conveyor so that small particles passing through the perforations of the upper conveyor fall on to a bed of larger coal on the lower conveyor.—A. G.

Fuel; Manufacture of — from anthracite duff. C. H. Steel, Kidwelly, Wales. Eng. Pat. 139,977, 12.6.19. (Appl. 11,848/19.)

ANTHRACITE duff is briquetted with 6—16% of pitch, $\frac{1}{2}$ —2% of lime or chalk, and one part in 450,000 of oxalic acid at a pressure of at least 5 tons per sq. in.—A. G.

Fuel; Composite —. G. Vickers and A. Dickson, Whitehaven. From W. Clayton, Toronto, Canada. Eng. Pat. 110,025, 28.10.19. (Appl. 26,395/19.)

THE fuel comprises 20% of coal, coke, or coal dust with 10% charcoal or ground peat, briquetted with a binder consisting of 25% loam, 10% clay, 15% sawdust, 5% sand, 13% linseed or other crude oil, and 2% lime.—A. G.

Lignite; Treating [briquetting] of —. W. Runge, Orange, N.J., Assignor to International Coal Products Corp., Richmond, Va. U.S. Pat. 1,334,170, 16.3.20. Appl., 30.1.19.

LIGNITE is mixed with pitch, the mixture is partially carbonised, and the product is crushed, formed into briquettes, and carbonised.

Briquettes; Method of manufacturing —. C. H. Smith, Short Hills, N.J., Assignor to International Coal Products Corp., Richmond, Va. U.S. Pat. 1,334,180, 16.3.20. Appl., 18.2.18.

FINELY powdered non-coking bituminous coal is mixed with coking coal and pitch in proportions such that the volatile matter is from 11 to 20% of the whole mixture. Briquettes are formed from the mixture and carbonised.—J. W. D.

Drying of fuels with hot gases; Process for preliminary —. A.-G. für Brennstoffvergasung, Saarbrücken. Ger. Pat. 315,621, 29.10.18.

THE hot gases prior to entering the dryer are passed through a layer of readily ignited material which removes contained oxygen. Such a material is dried lignite, which can be placed on a step grate through which the hot gases travel; or a burner with an inadequate supply of primary air may be employed so that the hot gases in traversing the flame are deprived of oxygen.—H. J. H.

Coke-oven and method of operating same. C. E. Lucke, New York. Assignor to L. and A. A. Wilputte, New Rochelle, N.Y. U.S. Pat. 1,332,909, 9.3.20. Appl., 1.3.18.

A REGENERATIVE coke-oven has each of its heating walls formed with a number of vertical flues, all of which are connected at their upper ends by a common horizontal passage. Half of the vertical flues at one end are connected by small openings at their lower ends to a single horizontal channel at the bottom, and the remaining flues are similarly connected to another horizontal channel. Air is alternately supplied to, and waste gas withdrawn from, each of the horizontal channels at the opposite side of the dividing plane of the two sets of flues from that at which the channel is connected to the flues.

—W. F. F.

Pitch; Process for vaporising and gasifying —.

W. Herrmann, Kiel. Ger. Pat. 314,996, 26.11.18.

THE pitch (soft or hard) is melted at a high temperature and atomised by means of sprays into retorts filled with or traversed by hot gases or superheated steam. The particles of pitch are partly vaporised and deposited in a semi-solid condition on the walls of the retort, where they evolve the remainder of their volatile constituents and unite with the coke already there to form a light, porous deposit which is easily detached. No fusion of the coke layer takes place, and, since the quantity of pitch present in the retort at any given time is very small, no frothing over can occur.—J. F. B.

Alum-shale; Utilisation of — as fuel in calcining furnaces.

H. G. V. Rydahl, Skövde, Sweden. Ger. Pat. 315,695, 23.10.17.

THE alum-shale is carried through the furnace on an endless travelling grate and the material to be calcined is carried on an endless travelling conveyor above the grate. The fuel conveyor may run on rollers arranged alternately at different heights, and the two conveyors should travel in opposite directions. This arrangement is designed to prevent the sintering of the alum shale and the formation of lumps.—J. F. B.

Peat; Process for gasifying —. Elektro-Osmose

A.-G. (Graf Schwerin Ges.), Berlin. Ger. Pat. 316,651, 3.11.17.

THE peat is dried by hot air, the air which has become laden with moisture in its passage through the drying room being discharged into the gas producer. In this way the moisture contained in the drying air is utilised for the gasification, and in the production of "semi-water-gas" the quantity of steam required may be reduced.—J. F. B.

Gas producers. L. Fornas, Paris. Eng. Pat.

139,578, 13.2.19. (Appl. 3503/19.) Addition to 123,323.

THE gas-producer described in the chief patent (this J., 1920, 260 A) is modified by providing arrangements for moistening hard, dry fuel.—A. G.

Gas-producers. Poetter G.m.b.H., Düsseldorf. Ger.

Pat. 317,042, 12.7.17.

A MIXTURE of gas and air is blown through the bottom of the hearth, so that the clinker is converted into a molten slag.—J. F. B.

Gas; Apparatus for manufacturing —. W. D.

Wileox, Chicago, Ill. U.S. Pat. 1,332,369, 2.3.20. Appl., 1.4.18.

TWO gas-generating chambers, each having means for the admission of fuel, the withdrawal of ash, and the introduction of air or steam at the lower portion, are separated by a secondary combustion chamber having an inlet for air and an outlet for waste gas. The gas produced in either generator is passed through the upper part of the combustion chamber, thence into the lower part of the other generator and up through the fuel bed before being withdrawn.—W. E. F. P.

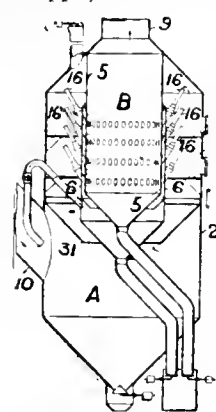
By-product condenser. A. Roberts, Evanston,

Assignor to American Coke and Chemical Co., Chicago, Ill. U.S. Pat. 1,333,631, 16.3.20. Appl., 17.4.15. Renewed 7.1.18.

A SERIES of vertical, columnar units has a gas inlet and a gas outlet manifold at the lower and upper ends respectively, and a partition provided with a valve is disposed between the lower end and the gas inlet of each column. By means of a valved spray-nozzle in alinement with the upper end of each vertical unit, a regulated quantity of precipitating medium is delivered into each column; and a connexion is provided above each of the partitions for the delivery of the fractional precipitate.—W. E. F. P.

Gases; Apparatus for cleaning —. F. R. McGee,

Steubenville, Ohio. U.S. Pat. 1,333,325, 9.3.20. Appl., 13.5.18.



GAS to be cleaned passes through the inlet, 10, into the chamber, A, and then passes upwards through the funnel, 31, into the superimposed annular chamber formed between the outer and inner walls, 2 and 6, respectively. A series of separator pipes, 16, project downwards through the double walls, 6, 5, into the inner chamber, B, and each pipe is provided with means for giving the gas a whirling motion. The gas is thus separated into a central core of clean gas which passes into the collecting chamber, B, and a peripheral layer of impure gas which passes through openings in the wall of each pipe into the narrow annular space between the walls, 5, 6. Each of the chambers is provided with collecting means for deposits at the bottom, and the clean gas is withdrawn at the outlet, 9.

—W. F. F.

Exhaust gases of internal combustion engines; Purification of —. Gasmotoren-Fabrik Deutz,

Cöln-Deutz. Ger. Pat. 315,451, 28.9.18.

THE complete separation of moisture from the exhaust gases suffices to render them innocuous without any further treatment to remove carbon dioxide and sulphur dioxide. The water may be removed by chemical means or by freezing.—H. J. H.

Acetylene gas and the like; Solvent for —. H. S.

Smith, New York, and G. O. Curme, jun., Pittsburgh, Pa., Assignors to The Prest-O-Lite Co., Inc., New York. U.S. Pat. 1,332,525, 2.3.20. Appl., 13.8.18.

A SOLVENT for acetylene gas and the like comprises acetone containing an ester of an organic acid, miscible therewith.—A. G.

Gasoline; Process for obtaining — from natural

gas. J. B. Garner, Pittsburgh, and H. C. Cooper, Clarksburg, W. Va., Assignors to Hope Natural Gas Co., Pittsburgh, Pa. U.S. Pat. 1,332,290, 2.3.20. Appl., 8.6.16.

THE gas, under pressure, is treated with an absorbent. By successive distillation under pressure the absorbent is then deprived of its lighter combustible constituents and gasoline content.

—A. E. D.

Asphalt; Manufacture of —. R. R. Rosenbaum,

Assignor to Central Commercial Co., Chicago, Ill. U.S. Pat. 1,332,359, 2.3.20. Appl., 2.4.15. Renewed 29.7.18.

PARAFFIN hydrocarbons are mixed with non-fusible or imperfectly fusible bitumen. Heat is applied to the mixture in a closed vessel, so that the gas evolved produces a high pressure, until a homogeneous melt is obtained.—A. E. D.

Petroleum reduction; Process of —. F. A.

Kormann, New York, Assignor to W. F. Hull, Brooklyn, N.Y. U.S. Pat. 1,332,849, 2.3.20. Appl., 17.7.18.

THE physical properties of petroleum or like hydrocarbon material are altered by absorbing it in a heated absorbent material, and then expelling it by the application of heat.—A. E. D.

Hydrocarbons; Treating —. J. W. Coast, jun.,

Tulsa, Okla., Assignor to The Process Co. U.S. Pat. 1,333,964, 16.3.20. Appl., 5.5.17.

OIL is cracked in a still at 50 lb. pressure and the

vapour therefrom is passed through fine orifices into a body of cooler oil contained in a reservoir. The escaping vapour from the reservoir passes through a reflux condenser before entering the main condenser. The oil continuously leaves the reservoir and passes into the still.—A. E. D.

Lubricating oils from coal tar oils; Manufacture of —. H. Klever, Karlsruhe. Ger. Pats. (a) 301,774, 2.2.15, and (n) 301,776, 27.3.15.

(a) By heating coal tar oils under pressure to about 250°–350° C., if necessary in the presence of catalysts, they are converted into viscous lubricating oils. The increase in viscosity is mainly due to the condensation of bases and phenols of high molecular weight, particularly the bases, and in a minor degree to the condensation and polymerisation of the neutral constituents, such as hydrocarbons. During the process considerable quantities of ammonia and water are formed. Suitable catalysts are fuller's earth, kieselguhr, kaolin, alumina, quartz powder, aluminium, zinc, iron, cadmium and cuprous chlorides. To obtain products of low solidifying point the tar oils are first treated for the removal of the constituents which readily crystallise, for instance, by distilling off the fractions containing these constituents or by prolonged storage at a low temperature and subsequent filtration or centrifugal extraction. (n) The process described under (a) may be carried out in the presence of small quantities of air or oxygen. Pitch oils obtained from coal tar pitch may be treated by this process.—J. F. B.

Lubricating oil and paraffin wax; Production of high-grade viscous — from high-boiling fractions of gas-producer and low-temperature retort tars. Allgem. Ges. für Chemische Industrie, Berlin. Ger. Pat. 310,653, 11.7.17.

The tar fractions of high boiling point, after removal of constituents containing oxygen by washing with aqueous caustic alkali or ethyl or methyl alcohol, are treated with liquid sulphur dioxide, and the resulting solution is separated from the solid paraffin which settles out. From a producer gas coal-tar distillate, b.p. 270°–450° C., a lubricating oil with a viscosity up to 10 at 50° C., sp. gr. 1.03, and a setting point below –20° C. is obtained.—J. F. B.

Tars, particularly oil-tar; Extraction of — and *deresination of the resulting lubricating oils.* Röchlingsche Eisen- und Stahlwerke G.m.b.H., and R. Schröder, Volklingen. Ger. Pat. 315,554, 15.8.18.

THE tar is subjected to distillation up to 200° or 250° C., and is then transferred hot from the retort into a rotating extractor in which it is treated with calcium oxide or other chemicals capable of combining with resin acids; after cooling it is treated with twice its weight of alcohol and stirred for several hours at the ordinary temperature. The extractor consists of a rotating drum in which trough-shaped vessels filled with chemicals are so arranged that the contents of the troughs cannot be emptied during rotation. The alcohol dissolves the so-called resin acids, while the paraffins and asphaltic constituents remain behind; the remainder of the resin acids is combined with calcium oxide. The alcoholic solution of the oil, when subjected to fractional distillation, yields valuable lubricating products.—J. F. B.

Lubricating and drilling machine oil; Substitute for —. K. S. Fuchs, Heppenheim. Ger. Pat. 316,028, 6.7.18.

THE solution of alkali salts of lignic acid obtained as waste liquor in the digestion of straw, maize-cob meal, etc., is precipitated by waste mineral acid or

bisulphate; the gelatinous product is heated at 70°–75° C., and the lignic acid settles out. A 25% solution of the sodium salt of this product gives a brown oily liquid, which is used as such or after emulsification with a little oil.—J. F. B.

Coke; Process and plant for utilising the steam developed in the quenching of —. J. Pintsch A.-G., Berlin, and L. Rodde, Augsburg-Oberhausen. Ger. Pat. 316,114, 2.5.18.

Gas producers or carbonising plants; Furnace grates, more particularly for use with —. J. Wells, Cairo, Egypt. Eng. Pat. 139,731, 19.9.19. (Appl. 23,086/19.)

Waste fuel; Apparatus for recuperation of —. F. Lioud, Assignor to Soc. Le Coke Industriel, St. Etienne, France. U.S. Pat. 1,331,765, 23.3.20. Appl., 15.11.19.

SEE Eng. Pat. 120,932 of 1918; this J., 1919, 672 A.

Furnace [coke-oven]; Regenerative —. E. Hurez, Petit Couronne, France. U.S. Pat. 1,333,716, 16.3.20. Appl., 19.3.17.

SEE Eng. Pat. 107,178 of 1917; this J., 1917, 861.

[Gas] retorts; Machine for charging and discharging —. J. G. W. Aldridge, London. U.S. Pat. 1,327,582, 6.1.20. Appl., 26.10.15.

SEE Eng. Pat. 22,066 of 1914; this J., 1915, 1236.

Gas manufacturing; Purifying method and apparatus for —. F. A. Umsted, Chicago, Ill. U.S. Pat. 1,332,529, 2.3.20. Appl., 1.2.18.

SEE Eng. Pat. 135,931 of 1918; this J., 1920, 98 A.

Hydrocarbons; Process of producing low-boiling —. P. Hubbard, Assignor to The Iroline Co. of America, Washington, D.C. U.S. Pat. 1,326,056, 23.12.19. Appl., 1.8.14.

SEE Eng. Pat. 3327 of 1915; this J., 1916, 828.

Producer-gas furnace. Eng. Pat. 139,957. See I.

Gas furnace. U.S. Pat. 1,331,309. See I.

Gaseous mixtures. U.S. Pat. 1,333,850. See XXIII.

IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Distillation of South Indian woods. H. E. Watson, J. J. Sudborough, K. S. D. Dose, and K. U. Rao. J. Indian Inst. Sci., 1918, 2, 107–119.

DETAILS are given of the yields of charcoal, acetic acid, methyl alcohol, and tar obtained by the distillation of 22 species of South Indian woods. Most of the woods gave lower yields than the woods commonly distilled in other countries. *Casuarina equisetifolia* (Forst.) wood, however, gave good yields of acetic acid (4.12 and 4.21%), and methyl alcohol (1.19 and 2.11%).—C. A. M.

PATENTS.

Retort ovens for the distillation of fuels and bituminous ores. E. Hauser, Madrid. Eng. Pat. 125,980, 22.4.19. (Appl. 9970/19.) Int. Conv., 20.4.18.

A VERTICAL retort of the Hawkins and Barton type (Eng. Pat. 5461 of 1890), having side walls which diverge slightly downwards, has a vertical collecting flue adjacent to one of its side walls. The retort communicates with the flue through a number of closely spaced openings inclined upwards and outwards, and the flue is subdivided by fixed transverse partitions which slope downwards and outwards from the retort. Each compartment thus

formed is provided with a valve-controlled discharge pipe through which, as required, air, inert or reducing gas, or steam may be injected at any desired zone. When the retort is constructed of metal its side walls may be extended to the surrounding setting to separate the flues for the distillation and heating gases. The retorts may be formed with internal transverse undulations on their walls to facilitate the descent of the charge.

—W. F. F.

Arc-lamp. G. M. Little, Pittsburgh, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,333,665, 16.3.20. Appl., 12.11.15.

The electrodes are enclosed in an arcing chamber, and means are provided by which, through the heat of the lamp, the surfaces exposed to the fumes of the arc are covered with a protective coating derived from an amine.—A. R. P.

Catalytic heating apparatus. Soc. Lyonnaise des Réchauds Catalytiques, Camell, Cochet, Gritte et Cie., Lyons, France. Eng. Pat. 140,001, 30.7.19. (Appl. 18,936/19.) Int. Conv. 24.4.19.

III.—TAR AND TAR PRODUCTS.

Phenols; Identification of. — II. J. A. Lyman and E. E. Reid. J. Amer. Chem. Soc., 1920, 42, 615—619. (Compare this J., 1917, 333.)

The melting points of *p*-nitrobenzyl ethers of a number of phenols are given. In the etherification of esters of salicylic acid it is advisable to employ as solvents the respective alcohols from which they are derived, and to avoid excess of alkali; otherwise interchange of the alkyl groups of the alcohol and of the ester takes place to a certain extent (compare Pardee and Reid, this J., 1920, 305A. Also J. C. S., i., 381).—J. K.

Nitro-compounds; Reduction of aromatic — by means of platinum and hydrogen. G. Cusmano. Annali Chim. Appl., 1919, 12, 123—130.

AROMATIC nitro-compounds (*p*-nitro-derivatives of phenol, anisol, and toluene; *o*-nitrotoluene, *m*-nitroaniline) are readily reduced to amines when shaken in ethereal solution with platinum black in an atmosphere of hydrogen, but even when an insufficient quantity of hydrogen for complete reduction is used, the intermediate stages of nitroso- and β -hydroxylamino-derivatives cannot be detected. This is due to the fact that under the experimental conditions the intermediate compounds are reduced more rapidly than the nitro-compounds. By introducing negative substituents into the nitro-compound (nitrobenzoic acids, nitrobenzaldehydes, dinitrobenzenes, dinitrotoluenes) or by effecting reduction in acid solution (*m*-nitroaniline), the decomposition of the intermediate hydroxylamino derivative can be retarded and its formation demonstrated. (Cf. J. C. S., i., 298.) —C. A. M.

Petrol. Florentin and Vandenberghe. See II A.

Pyrrole, indole, and carbazole. Franklin. See XX.

PATENTS.

Coal tar hydrocarbons and other like matters; Fractional distillation plant for. — W. E. Edwards, Wigan. Eng. Pat. 139,263, 28.1.19. (Appl. 2073/19.)

Tar is led from a receiver through two preheaters into two stills arranged in series. In the first preheater, the tar flows up a cylindrical vessel fitted with a worm which serves as a condenser for the vapours generated in the second still, and is then

led to the second preheater in which it flows up a number of vertical tubes heated by the pitch escaping from the second still. Light oils distil from the tar in the first still, and are conveyed to a water condenser; creosote oil and anthracene oil are removed in the second still, and as the vapours pass down the worm of the first preheater, the anthracene oil condenses first and is tapped off at a point about halfway down the worm, whilst the creosote oil, which condenses in the lower half, is drawn off at the bottom. The first still may be heated by the hot gases escaping from the furnace under the second still, and any permanent gas not condensed in the water condenser may be utilised for heating the stills.—L. A. C.

Carbazole; Manufacture or isolation of. — Burt, Boulton and Haywood, Ltd., and F. D. Miles, London. Eng. Pat. 139,931, 19.6.19. (Appl. 15,440/19.)

CRUDE carbazole, or a mixture containing carbazole, is heated with an alkali metal, or an alkali metal oxide or hydroxide, in the presence of an indifferent solvent, e.g., naphthalene, or toluene under pressure, at a temperature below the melting point of the alkali-carbazole. The solvent is subsequently removed, and the alkali-carbazole is decomposed by boiling with water.—L. A. C.

Hydrocarbon; Process for the manufacture of lower-boiling — from higher-boiling hydrocarbon [toluol from xylol]. A. E. Houlehan, Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,331,033, 16.3.20. Appl., 25.5.17.

XYLOL is subjected to the action of anhydrous aluminium chloride whereby toluol is produced and is distilled off continuously.—A. E. D.

Sulphonation and chlorination of aromatic substances. H. N. Morris and Co., Ltd., and H. N. Morris, Manchester. Eng. Pat. 140,007, 5.6.18. (Appl. 21,360/19.)

AN aromatic hydrocarbon and either sulphuric acid or liquid chlorine are led through atomisers into the mixing chamber in the apparatus described in Eng. Pat. 139,234 (this J., 1920, 291A), and the mixed liquids pass through a chamber or series of chambers maintained at the desired temperature, and, in the case of chlorination, containing a catalyst.

—L. A. C.

Sulphonation of hydrocarbons of the aromatic series. L. M. Dennis, Ithaca, N.Y. U.S. Pat. 1,332,203, 2.3.20. Appl., 6.7.17.

β -NAPHTHALENESULPHONIC acid is extracted from the mixture obtained by sulphonating naphthalene with about 75% sulphuric acid, and the solution obtained is cooled.—L. A. C.

Carbazole; Production of high-percentage. — H. Leroux, Assignor to Soc. d'Éclairage, Chauffage, et Force Motrice, Paris. U.S. Pat. 1,318,212, 7.10.19. Appl., 31.10.18.

SEE Eng. Pat. 121,455 of 1918; this J., 1920, 9 A.

Gasifying pitch. Ger. Pat. 314,996. See II A.

Lubricating oils. Ger. Pats. 301,774—5. See II A.

Lubricating oil. Ger. Pat. 310,653. See II A.

Lubricating oils. Ger. Pat. 315,554. See II A.

Rubber composition. Plastic composition. U.S. Pats. 1,334,060—1. See XIV.

IV.—COLOURING MATTERS AND DYES.

Vat dyestuffs; Benzidinoquinones and bisquinonyl-benzidines as —. K. Brass and O. Papp. Ber., 1920, 53, 446—462.

BENZIDINE condenses with toluquinone to yield benzidino-toluquinone, $\text{CH}_3\text{C}_6\text{H}_4\text{O}_2\text{NH}\cdot\text{C}_6\text{H}_4\cdot\text{C}_6\text{H}_4\text{NH}_2$, a dark violet powder which cannot be preserved in contact with air, and which dyes cotton in brown shades from a faintly alkaline vat, the colour being, however, due to polymerised products of the dye. Under similar conditions methylanilino-*p*-benzoquinone yields a mixture of much 2-benzidino-5-methylanilinobenzoquinone and little NN'-bis-5-methylanilinobenzoquinonyl-2-benzidine $[\text{CH}_3(\text{C}_6\text{H}_4)\text{N}\cdot\text{C}_6\text{H}_4\text{O}_2\text{NH}\cdot\text{C}_6\text{H}_4]_2$, which have similar tinctorial properties and dye cotton in yellowish-brown shades from a yellow vat. Di-*o*-anisidine and methylanilinobenzoquinone give 2-di-*o*-anisidine-5-methylanilinobenzoquinone, $\text{CH}_3(\text{C}_6\text{H}_4)\text{N}\cdot\text{C}_6\text{H}_4\text{O}_2\text{NH}\cdot\text{C}_6\text{H}_3(\text{OCH}_3)\cdot\text{C}_6\text{H}_3(\text{OCH}_3)\text{NH}_2$, m.p. 124°—128° C. (decomp.) after softening from 120° C., and NN'-bis-5-methylanilinobenzoquinonyl-2-dianisidine $[\text{CH}_3(\text{C}_6\text{H}_4)\text{N}\cdot\text{C}_6\text{H}_4\text{O}_2\text{NH}\cdot\text{C}_6\text{H}_3(\text{OCH}_3)]_2$, m.p. 244°—246° C., which, on cotton, yield brownish shades with a tendency towards olive. NN'-Bis- α -naphthoquinonyl-2-dianisidine $[\text{C}_{10}\text{H}_7\text{O}_2\text{NH}\cdot\text{C}_6\text{H}_3(\text{OCH}_3)]_2$, brownish-violet crystalline powder, m.p. 303°—305° C., which dyes cotton in violet shades, is prepared from α -naphthoquinone and dianisidine, whilst NN'-bis-3-chloro- α -naphthoquinonyldianisidine, red needles, m.p. 270°—272° C., giving brownish-violet shades on cotton, is similarly derived from 2,3-dichloro- α -naphthoquinone. α -Ethoxybenzidine and α -naphthoquinone yield NN'-bis- α -naphthoquinonyl-3-ethoxybenzidine, $\text{C}_{10}\text{H}_7\text{O}_2\text{NH}\cdot\text{C}_6\text{H}_4(\text{OC}_2\text{H}_5)\cdot\text{C}_6\text{H}_4\text{NH}\cdot\text{C}_{10}\text{H}_7\text{O}_2$, violet-brown needles, m.p. 279°—281° C., which gives violet shades on cotton. 2-Anilino-3,5,6-trichlorobenzoquinone is oxidised by manganese dioxide in the presence of sulphuric acid to 3-anilino-2',5',3',5',6-pentachlorodibenzoquinonylaniline, $\text{C}_6\text{O}_2\text{Cl}_3\cdot\text{N}(\text{C}_6\text{H}_4)\cdot\text{C}_6\text{O}_2\text{Cl}_3\cdot\text{NHC}_6\text{H}_5$, bluish-grey leaflets, which do not melt below 300° C., and which dye cotton in dull brown shades. (Cf. J. C. S., i., 398.)—H. W.

Hæmatoxylin; Preparation of —. P. A. Houseman and C. K. Swift. J. Ind. Eng. Chem., 1920, 12, 173—174.

CRUSHED logwood extract, free from dust, is placed in a cylindrical vessel fitted with a reflux condenser; ether vapour from a flask passes into the top of the cylinder, is condensed, and falls on to the contents of the cylinder; the ethereal extract siphons continuously back into the flask. The latter is heated electrically; a mercury manometer on the flask is in circuit with a relay which cuts off the current if pressure develops in the apparatus owing to incomplete condensation of the ether. When the extraction is finished a tap on the siphon is closed and the ether distilled from the flask into the cylinder. The residue of crude hæmatoxylin in the flask is dissolved in hot water and the solution filtered; pure hæmatoxylin crystallises out as the solution cools.

—W. P. S.

Pyrrole Blacks. A. Angeli and C. Lutri. Atti R. Accad. Lincei, 1920, 29, i., 14—22. (Compare this J., 1919, 139 A.)

IN presence of water or in alcoholic, ethereal, or moist benzene solution, pyrrole and quinone react, giving a black, amorphous compound, which closely resembles Pyrrole Blacks and melanins, and is apparently formed by the interaction of 3 (or 4) mols. of pyrrole and 5 (or 7) mols. of quinone. Under similar conditions indole does not react with pyrrole, but the latter (1 mol.) and 1,4-naphthoquinone (3 mols.) yield a compound crystallising in violet-

black needles. Isatin and pyrrole also react readily. Oxidation of pyrrole by means of hydrogen peroxide yields a Pyrrole Black and a compound, $\text{C}_8\text{H}_{10}\text{ON}_2$, which crystallises in almost colourless needles, m.p. 136° C. (Cf. J. C. S., i., 397.)—T. H. P.

Picric acid. Marqueyrol and Carré. See XXII.

PATENTS.

Disazo dyestuffs; Manufacture of new yellow —. G. B. Ellis, London. From Chemical Works, formerly Sandoz, Basle, Switzerland. Eng. Pat. 132,995, 1.5.19. (Appl. 10,887/19.)

TETRAZOTISED 4,4'-diaminodiphenyl-2,2'-dicarboxylic acid is combined with *o*-hydroxycarboxylic acids of the benzene series, e.g., salicylic acid, 4-hydroxyisophthalic acid, or *o*- or *m*-cresotic acid. The products give yellow shades when printed on cotton with chromium or chromium-aluminium mordants.

Azo dyes; Production of —. J. L. Kane, Philadelphia, Pa. U.S. Pat. 1,333,807, 16.3.20. Appl. 19.6.19.

As an improvement in the production of azo dyes, an aromatic amino is treated with a starchy material in aqueous suspension.—L. A. C.

Azo dyestuffs dyeing mordanted goods. H. Fritzsche, Assignor to Society of Chemical Industry in Basle, Switzerland. U.S. Pat. 1,325,841, 23.12.19. Appl., 14.12.18.

SEE Eng. Pat. 126,460 of 1918; this J., 1919, 457 A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

[Wool.] *Causes of the so-called "Allwörden's reaction."* P. Kraiss and P. Wentig. Neue Faserstoffe, 1919, 1, 265—266. Chem. Zentr., 1920, 91, H., 209. (See this J., 1916, 416; 1917, 706.)

ALLWÖRDEN'S reaction does not give uniform results with all kinds of wool and different sections of the same wool fibre behave differently. The chlorine water must be freshly prepared; with bromine water the swellings separate better from the fibre. The removal of suint and grease is important. Hairs containing a medullah may show the reaction very distinctly; feathers do not react. Both in the chlorine reaction which causes the formation of the swellings and in the treatment with alkali which removes the "elastium" from the wool, there is an attack on the protein matters of the wool. The substance sensitive to chlorine is a protein present in the fibrillie layer or in a cementing substance adhering to the fibrillie. At high temperatures the sensitiveness of the test to the influence of alkalis increases to an extraordinary degree, so that at the boiling temperature the traces of alkali which are dissolved from an ordinary glass vessel are sufficient to cause the absence of the reaction. The effect of boiling, however, is entirely suppressed by traces of acid, and the reaction remains positive after an acid treatment which would certainly have an injurious effect on the quality of the wool.—J. F. B.

Fibres and yarns, especially artificial silk and staple-fibre; Tensile strength of —. P. Kraiss. Neue Faserstoffe, 1919, 1, 266—268. Chem. Zentr., 1920, 91, H., 208. (See also this J., 1919, 895 A.)

STAPLE-FIBRE YARNS from viscose or cuprammonium silks show a loss of tensile strength on damping with water, also with spinning emulsions, but petroleum, amyl acetate, and phthalic acid esters impart a distinct increase in strength. Heating for a short time increases the strength in the dry condition,

but the strength in the wet condition is not improved. As regards chemical after-treatments, dyeing with chrome yellow has a favourable effect on viscose yarns, and cuprammonium yarns are improved both by chrome yellow and by treatment with tannin-gelatin. Chemical after-treatments may also mitigate the tendering of artificial silks on damping.—J. F. B.

Straw and wood pulp manufacture; Utilisation of spent liquors from —. M. Müller. *Wochenbl. Papierfab.*, 1919, 50, 2749–2750. *Chem. Zentr.*, 1920, 91, 11., 151.

STEAM is admitted to the digester freshly charged with wood in order to create a vacuum. The whole of the hot, black, spent liquor from a previous digestion is then pumped on to the prepared charge, after which the spent liquor is quickly forced out again by steam free from air. In this way the most important feature of Ungerer's process is utilised. At the end of the digestion, in the manufacture of pulp for bleaching, carbon dioxide and atmospheric oxygen must, as far as possible, be excluded from the digested pulp, for which reason blowing out the charge is preferable to extraction by Shank's process. The freshly blown-out pulp should be treated with hot fresh caustic liquor, which is drained off and used for digesting fresh wood. The destructive action of the liquor on the cellulose may be avoided by using the process described in Ger. Pat. 284,681 (this J., 1915, 1048).

—J. F. B.

Cellulose mucilage. C. G. Schwalbe and E. Becker. *Z. angew. Chem.*, 1920, 33, 57–58.

THE parchment ("pergamyn") cellulose mentioned in a previous paper (this J., 1919, 858 A; 1920, 58 A) had a high methyl value indicating that the treatment had left considerable quantities of lignin unattacked. The physical properties of a number of cellulose mucilages were investigated; that from cotton wool was almost entirely free from fibres exhibiting structure, but some of the parchment cellulose mucilages contained fibre débris. In the case of mucilage prepared from soda- or sulphite-cellulose by grinding in an edge-runner, the Schopper-Riegler sieve apparatus is useless for determining the degree to which the fibre has been converted into mucilage, but the apparatus gave more trustworthy results with mucilage prepared by prolonged beating in the hollander.—W. P. S.

Cellulose mucilage. Hygroscopic properties of paper containing mucilage. C. G. Schwalbe and E. Becker. *Z. angew. Chem.*, 1920, 33, 58–59.

PARCHMENT ("pergamyn") cellulose and parchmentised paper and similar products have a moisture content of 6–8% after exposure to the ordinary atmosphere, but after exposure for four hours in air saturated with water vapour the water content increases to 17–26%. Previous heating at 120° C. diminishes this property of absorbing water.

—W. P. S.

Lignin; Researches on —. II. *Potash fusion of the lignosulphonic acids.* M. Hönig and W. Fuchs. *Monatsh.*, 1919, 40, 341–349.

THE removal of the sulphonic groups from the barium salts previously described (this J., 1918, 502 A) by fusion with caustic potash is only complete at 250°–300° C. In each case protocatechuic acid was the sole phenolic product obtained (compare Melander, this J., 1919, 625 A), the yield corresponding to 13–19% of the purely organic portion of the original material. The sulphonic acids are therefore complicated aromatic sulphonic acids, containing the carbon-skeleton of protocatechuic acid. (*Cf.* J. C. S., i., 291.)—J. K.

Paper; Testing tearing resistance of —. C. F. Sammet. *Paper*, 1920, 25, 1053–1054.

THE Schopper tensile testing machine may be modified so as to afford a numerical value for the resistance to tearing. The lever arm is raised to a definite position up the scale, and a strip of paper, about 6 in. long and 1½ in. wide, is slit at one end to a depth of 2 in. One of the slit ends is then clamped in the upper jaw and the adjacent slit end in the lower jaw of the machine, and the strip is brought to a taut condition by adjusting the hand wheel and starting a tear in the paper of about ¼ in. The lever arm is then released from its fixed position and exerts a tearing force on the paper which decreases as the lever falls. The lever arm comes to rest at the point on the scale where the resistance of the paper balances the weight of the falling arm. The results are concordant, and practical working values are obtained from an average of five tests in each direction of the web. The tearing resistance of machine-made papers is slightly higher across the web than along the web.—J. F. B.

Glucosides. Karrer. *See* XX.

PATENTS.

Broom fibres; Process for improving —. Nessel-anbau-Ges.m.b.H., Berlin. Ger. Pat. 315,754, 10.9.18.

BROOM fibres are treated with strong caustic soda solution or substances with similar action, e.g., sodium sulphide, aluminate, and zincate. The fibres are thereby strengthened; they become curly, more elastic, and softer, and a good spinning material is obtained.—J. F. B.

Paper yarns and textiles; Process for softening —. C. Bennert, Grünau. Ger. Pat. 315,834, 6.3.18.

THE material is treated with the decomposition products of proteins, such as protalbinic and lysalbinic acids or their salts, alone or in conjunction with other suitable substances. When these substances are added to the dyebaths in which cellulose textiles are to be dyed, they promote the absorption of the dyestuff and give more brilliant shades.—J. F. B.

Straw and similar stem and bast fibres; Manufacture of a spinning and weaving fibre from —. Geraer Jute-Spinnerei und Weberei A.-G., Triebes. Ger. Pat. 316,109, 14.5.18.

THE raw material is boiled with a solution of 4–15% of sulphides or polysulphides of alkalis or alkaline earths to which caustic soda is added in the proportion of 1 to 4, until the pectin and lignin matters and the silica are dissolved; the digested mass is then resolved into separate fibres by the force of hot or boiling water directed in the manner of a waterfall upon it, being at the same time washed free from sulphides. In order to avoid hardening during drying, the mass, immediately after the treatment with water, is further treated with dilute aqueous hydrochloric acid, then thoroughly washed and dried.—J. F. B.

Fibres [peat moss]; Process for converting short, brittle — into a condition suitable for spinning. H. Deelen, Amersfoort, Holland. Ger. Pat. 316,511, 22.2.18.

PEAT or peat moss, previously freed from impurities, is treated with a dilute solution of soap or emulsion of fat in such a way that the individual fibres are penetrated by the fatty matter, and the production of a pasty, sticky mass is avoided. The impregnation may take place during the separation of the impurities by shaking or other mechanical treatment of the fibrous material, or else after it has been washed. Yarns can be spun from the treated peat or peat moss, which do not shrink under the

influence of air and moisture, and which are extremely soft and pliable, suitable for weaving in conjunction with stronger materials, such as wool, cotton, or jute.—J. F. B.

Waterproofing fabrics of all kinds. Badische Anilin- und Sodafabrik, Ludwigshafen. Ger. Pat. 303,390, 2.8.17.

THE fabrics are treated with emulsions of crude paraffin and alkaline substances. Crude paraffin, unlike the purified hydrocarbons, readily forms with alkalis emulsions which are stable in the cold. The creosote in the crude paraffin exerts a disinfectant action.—J. H. L.

Paper pulp; Dyeing — with insoluble artificial colouring matters. Farbw. vorm. Meister, Lucius, und Brünig, Höchst. Ger. Pat. 316,259, 5.8.17.

THE paper pulp is intimately mixed with the colouring matters converted into a soluble form, or with the soluble components from which the colouring matters are formed, in the presence of a small amount of water. The process is conducted not in the hollander, but in edge-runners, pulping machines, and kneading machines or other appliances which ensure the intimate mixing of the pulp with only small quantities of water.—J. F. B.

Strawboard, paper, and the like containers; Impregnating or saturating — and subsequently draining the same. J. Spicer and Sons, Ltd., London. From A. E. Shirlow, Glen Huntly, Victoria. Eng. Pats. 139,533 and 139,534, 1.11.18. (Appls. 17,902 and 17,906/18.)

Paper-pulp; Separator for —. J. White, Edinburgh. U.S. Pat. 1,333,287, 9.3.20. Appl., 22.3.19.

SEE Eng. Pat. 131,418 of 1918; this J., 1919, 759 A.

Parchmentised paper, vulcanised fibre, and the like; Manufacture of —. R. H. Clayton, Manchester, J. Huebner, Cheadle Hulme, and H. E. Williams, Assignors to The Manchester Oxide Co., Manchester. U.S. Pat. 1,333,465, 9.3.20. Appl., 10.6.19.

SEE Eng. Pat. 124,979 of 1918; this J., 1919, 358 A.

Parchment-paper; Process of manufacturing —. W. Dagnall, Hampton Wick. U.S. Pat. 1,334,843, 23.3.20. Appl., 4.2.19.

SEE Eng. Pat. 123,594 of 1918; this J., 1919, 283 A.

Lubricating oil. Ger. Pat. 316,028. See IIIA.

Heat insulating material. Ger. Pats. 304,239—40. See IX.

Plastic material. U.S. Pat. 1,331,519. See XI.

Saccharifying plant material. Ger. Pat. 305,690. See XVIII.

Fodder from wood. Ger. Pat. 305,090. See XIXA.

bottom plate. Liquid can be forced in either direction through the fibre chamber. (u) A fibre chamber of which the size can be varied is formed of non-perforated sides and movable foraminous top and bottom plates, spaced outwards from which are two other foraminous plates. By means of a pump liquid can be circulated in either direction through the device and through a reserve tank.

[*Aniline*] *Black; Production of — upon textile fibres.* E. A. Fourncaux, Manchester. Reissue 14,824, 23.3.20, of U.S. Pat. 1,277,056, 27.8.18. Appl., 14.8.19.

SEE Eng. Pat. 116,562 of 1917; this J., 1918, 463 A.

Softening paper yarns. Ger. Pat. 315,834. See V.

Dyeing paper pulp. Ger. Pat. 316,259. See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric acid containing oxides of nitrogen; Rapid decolorisation of strong —. K. Rosenstand-Wöldike. Chem.-Zeit., 1920, 44, 255.

THE addition of peroxides of lead or barium results in the formation of hydrogen peroxide, which oxidises the nitrogen oxides to nitric acid. The lead or barium formed is precipitated as sulphate, which is insoluble in the cold acid. So small an amount as 0.001—0.01% of hydrogen peroxide will effectively decolorise ordinary acid, whilst 1% suffices for an almost black material.—A. E. D.

Sulphuric acid; Gravimetric determination of —. L. W. Winkler. Z. angew. Chem., 1920, 33, 59—60.

Dry barium sulphate evolves a small quantity of sulphuric anhydride when ignited. When sulphuric acid is precipitated from a potassium sulphate solution having an acidity of about N/100, the weight of precipitated barium sulphate (about 0.3 gm.) dried at 130° C. should be multiplied by 1.0045, or that of ignited barium sulphate by 1.0099, in order to obtain the correct weight.—W. P. S.

Nitrogen oxides; Improvements in the industrial production of — in arc furnaces. F. Gros. Comptes rend., 1920, 170, 811—813.

THERE is considerable loss of energy in the fixation of atmospheric nitrogen in electric arc furnaces owing to the humidity of the atmosphere and its relatively low oxygen content. Yields at least 50% higher per kilowatt-hour can be obtained by using dry gases and artificially increasing the oxygen content up to 50%. The nitrogen peroxide can be collected by cooling the issuing gases to low temperatures. The gases circulate in a closed circuit, thus remaining dry, dry oxygen and nitrogen being introduced as required. The nitrogen peroxide collected as above may be converted into nitric acid of any strength required. The heat recoverable from the gases issuing from the furnaces is more than equivalent to the energy necessary for working the cooling apparatus and for the manufacture of the necessary oxygen.—W. G.

Nitrogen; Theoretical study of fixation of — by the electric arc. C. P. Steinmetz. Chem. and Met. Eng., 1920, 22, 299—304, 353—357, 411—416, 455—462.

THE theoretical factors involved in the fixation of nitrogen by the electric arc method have been extensively studied by the author. His investigations include a consideration of the reaction velocities and thermal equilibrium, tables of thermal nitrification constants, reaction periods and velocities in air being given. The influence of the rate of cooling

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Dyeing machine. H. M. Dudley, Philadelphia, Pa. U.S. Pats. (A) 1,332,188 and (B) 1,332,489, 2.3.20. Appl., 28.9 and 26.10.17.

(A) WITHIN a dyeing chamber are arranged a series of fibre chambers each composed of non-perforated sides, a foraminous bottom plate, and a foraminous top plate which can be moved within the chamber and held in any desired position with respect to the

is illustrated by curves. The employment of multiple arcs, and the cooling of the arc by adiabatic expansion and molecular diffusion, are discussed, as well as the effect of the arc in dissociating N_2 , O_2 , and NO . Finally, the principles of the arc and the characteristics of the various types have been studied and compared.—W. J. W.

Ammonium sulphate; Caking of —. C. G. Atwater and J. F. W. Schulze. *Chem. and Met. Eng.*, 1920, 22, 373—374.

THE caking of certain samples of ammonium sulphate was found to be due to the presence of pyridine sulphate, which causes the material to absorb moisture on damp days, subsequent loss of this on dry days producing a caking effect. Phenols do not cause the phenomenon. By passing dry ammonia gas, preferably preheated, through the sample, or by treatment with very dilute ammonia liquor, pyridine is liberated and the absorption of moisture and caking become inappreciable. (*Cf* this J., 1920, 101 r.)—W. J. W.

Ammonium nitrate; The formaldehyde method for determining —. J. T. Grissom. *J. Ind. Eng. Chem.*, 1920, 12, 172—173.

NEUTRAL 20% formaldehyde solution is added to the ammonium nitrate solution, the mixture heated to 60° C., and titrated with $N/3$ sodium hydroxide solution, using phenolphthalein as indicator. The reaction proceeds according to the equation: $6CH_2O + 4NH_4NO_3 + 4NaOH = C_6H_{12}N_4 + 4NaNO_3 + 10H_2O$. The results are consistently about 0.25% too low.—W. P. S.

Ammonium chloride; Phenomena observed during the melting, freezing, and boiling of mixtures of — and other chlorides. K. Hachmeister. *Z. anorg. Chem.*, 1919, 109, 145—186.

THE inversion temperature of ammonium chloride lies at 174° C. The following melting points are given: cuprous chloride 425° C., lithium chloride 605° C., zinc chloride 283° C., cadmium chloride 568° C., ferric chloride 303° C., thallous chloride 427° C., and lead chloride 501° C. In addition to binary compounds of ammonium chloride with the above named chlorides, already known, the following also exist: $CdCl_2 \cdot NH_4Cl$; $2ZnCl_2 \cdot NH_4Cl$, and $FeCl_3 \cdot NH_4Cl$. The compound $FeCl_3 \cdot NH_4Cl$ possesses a definite boiling point, 386° C. (*Cf* J.C.S., May.)—J. F. S.

Caustic pot settings. F. G. Wheeler. *Chem. and Met. Eng.*, 1920, 22, 372—373.

FOR securing economical working and long life of plant in caustic soda manufacture, a high temperature must be maintained in the furnace, and the ash-pit should hold a small amount of water so that steam passes with the air through the grates and keeps them clinkered. A combustion chamber behind the grate enables the soot to be burned before the hot gases are chilled by the pot, and so reduces smoke. Baffles are placed in the flue to keep the gases close to the pot before escaping up the stack. The life of the pots is increased by protecting them from any blow-torch effect of the flames by means of a guard wall thin enough not to hold the heat or reflect it into the furnace. The pots themselves should be so arranged as to facilitate turning them periodically.—W. J. W.

Nitrates; Effect of chlorides on the nitrometer determination of —. M. T. Sanders. *J. Ind. Eng. Chem.*, 1920, 12, 169—170.

IN the determination of nitric acid in sodium nitrate by means of the nitrometer, it is not possible to obtain results accurate within 0.1% if the nitrate contains more than 15% of its weight of sodium chloride.—W. P. S.

Copper sulphate; Rapid determination of — in works liquors. E. C. Carron. *Ann. Chim. Analyt.*, 1920, 2, 69—71.

THE amount of copper sulphate in the solutions at various stages of the manufacture may be determined with an accuracy within 1% by a simple process of centrifuging. The liquors must not contain more than 20—25 grms. of acid per litre, and the temperature of the mother liquors after centrifuging must not be below 22° C. The average contraction of the liquors after taking the sample is about 4%, whilst the copper sulphate crystals separated by centrifuging have sp. gr. 1.86: Fifty c.c. of hot liquor is measured into each of two copper tubes, which are placed for 15 to 20 mins. in a cold water bath, so as to lower the temperature from 90° to about 35—40° C., and then centrifuged for 3 to 4 mins. The mother liquors from the tubes are then drained into a graduated burette. Their volume deducted from 96 gives the number of c.c. occupied by the crystallised copper sulphate, and the latter value multiplied by 1.86 gives the weight of that salt. The amount of copper sulphate remaining in solution in the mother liquor may be found by determining the temperature of the liquid and referring to a table. At 10° C. the liquor contains 317.6 grms.; at 15° C., 337.1; at 20° C., 356.7; at 25° C., 378.1; and at 30° C., 401.6 grms. per litre.—C. A. M.

Catalysis. VII. Catalysis in heterogeneous systems. N. R. Dhar. *Proc. K. Akad. Wetensch.*, 1920, 22, 570—575.

THE velocity of solution of anhydrous ferric sulphate in water can be increased by the presence of reducing agents such as stannous chloride or ferrous sulphate. When 20% nitric acid in excess acts on copper at 18° C. so that the whole of the copper is dissolved, the action is accelerated by both ferrous and ferric salts as well as by a number of other compounds. Oxidising agents, as hydrogen peroxide, chromic acid, etc., retard the action of the acid. There is a small group of substances which in very small concentration exert a slight accelerating effect, whilst in larger concentrations they have a retarding effect. It is shown that nitrous acid is formed by the action of nitric acid on ferrous salts.—W. G.

Lead chromates. M. Gröger. *Z. anorg. Chem.*, 1919, 109, 226—234.

NORMAL lead chromate is prepared by adding 20 c.c. of $N/1$ potassium chromate to 19 c.c. of $N/1$ lead acetate. If 40 c.c. of $N/1$ lead acetate is treated with 10 c.c. of potassium chromate and the mixture evaporated to dryness on a water bath and washed with water the basic compound, PbO , $PbCrO_4$, is produced. By rubbing 2 grms. of normal lead chromate with 30 c.c. of $N/1$ potassium acetate, evaporating to dryness on a water bath, and washing with water, a lead potassium chromate, $2PbCrO_4 \cdot K_2CrO_4 \cdot H_2O$, is obtained.—J. F. S.

Lead cyanide. N. M. Gupta. *J. Ind. Inst. Sci.*, 1914, 1, 47—48.

THE precipitate formed by the interaction of cyanides and lead salts in aqueous solution is a basic salt, and varies in composition with the concentration of the solutions employed. On boiling a 5% solution of hydrogen cyanide with the precipitated cyanide under a reflux condenser, then filtering the liquid, and allowing it to evaporate in a desiccator, deep yellow needle-shaped crystals of lead cyanide, $Pb(CN)_2$, separated. Lead cyanide is unattacked by concentrated nitric and sulphuric acids in the cold, but on adding water to a crystal the insoluble oxycyanide is formed.—G. F. M.

Magnesium nitride; Behaviour of — towards carbon monoxide and carbon dioxide. F. Fichter and C. Schölly. *Helv. Chim. Acta*, 1920, 3, 298—304.

THE reactions between freshly prepared magnesium nitride and carbon monoxide and carbon dioxide respectively at 1250° C. are expressed by the equations: $\text{— Mg}_3\text{N}_2 + 3\text{CO} = 3\text{MgO} + \text{N}_2 + 3\text{C}$; $\text{— Mg}_3\text{N}_2 + 3\text{CO} = 3\text{MgO} + 3\text{CO} + \text{N}_2$. Carbon monoxide causes no change at 750° C., and reacts less readily than the dioxide. The reactions probably follow upon the dissociation of the nitride, which is almost complete at 1500° C. Aluminium nitride, which dissociates much less readily (Fichter and Oesterheld, this J., 1915, 518), is oxidised by carbon dioxide at 1300° C. to the extent of only 89%. (*Cf.* J.C.S., May.)—J. K.

Gadolinium. Methods of separation of the terbium and ytterbium earths. R. J. Meyer and U. Müller. *Z. anorg. Chem.*, 1919, 109, 1—30.

GADOLINIUM is best obtained from euxenite, samarskite, or monazite residues. When the mixture contains little cerium earths and much yttrium earths the gadolinium is separated with the cerium earths in one operation by the potassium double sulphate method. If the mixture contains much cerium earths the major portion of these is removed by the double nitrate method. In either case a fraction very much richer in gadolinium is obtained. This is converted into bromates and the yttrium earths removed by fractionation; the ytterbium earths first pass into the mother liquors, then follow the erbium earths, and last of all yttrium. It is essential that the yttrium should be entirely removed at this point, for it is hardly possible to separate it at any of the succeeding stages. The main fraction contains samarium, gadolinium, neodymium, terbium, and europium; it is converted into acetate and fractionated, the fractionation being combined with the fractionation of the double bismuth nitrates. The cerium earths are rapidly separated, and a brown mixture of oxides, containing gadolinium and terbium, is obtained. These elements are rapidly separated by fractional precipitation with ammonia. It is possible, by the above method, to obtain pure gadolinium oxide in a few months. (*Cf.* J.C.S., May.)—J. F. S.

Carbonyl chloride. E. Paternò and A. Mazzucchelli. *Gazz. Chim. Ital.*, 1920, 50, 30—53.

THE density of liquid carbonyl chloride between -15.4° and $+59.9^\circ$ C. is $1.4264 - 0.002326 t$, t being in degrees centigrade. The critical temperature is $187^\circ \pm 2^\circ$ C., critical density 0.5135, critical pressure about 51.5 atm., b.p. 8.02° C. at 760 mm. The chloride decomposes in contact with pure water, but its decomposition products, carbon dioxide and hydrochloric acid, retard further action, which is thus dependent on diffusion; acids in general also delay the interaction of water and carbonyl chloride. Besides the ordinary reaction between carbon tetrachloride and fuming sulphuric acid, $\text{CCl}_4 + 2\text{SO}_3 = \text{S}_2\text{O}_7 + \text{COCl}_2$, in presence of excess of the hot fuming acid the reaction, $\text{CCl}_4 + 4\text{SO}_3 = 2\text{S}_2\text{O}_7 + \text{CO}_2$, also takes place. At -15° C. liquid carbonyl chloride absorbs chlorine, giving a liquid containing 28.3% of free chlorine. Carbonyl chloride is absorbed in considerable proportions by heavy lubricating oils, from which it is expelled rapidly and almost completely by a current of air. (*Cf.* J.C.S., May.)—T. H. P.

Liquefied and compressed gases; Purification of — for laboratory purposes. L. Moser. *Z. anorg. Chem.*, 1920, 110, 125—142.

CARBON dioxide, in steel cylinders, is generally 98—99% pure, and may contain small quantities of

oxygen, nitrogen, and carbon monoxide, rarely traces of hydrogen sulphide and sulphur dioxide. It can be completely freed from oxygen by passing at a rate of 1—6 litres per hour through a paste of chromium acetate and then through a 15% solution of titanous chloride acidified with hydrochloric acid, or by passing over red hot copper at 4—10 litres per hour. Copper has no reducing action on carbon dioxide. To free it from traces of carbon monoxide the gas may be passed over asbestos carrying finely divided cuprous oxide at $700^\circ - 800^\circ$ C. Hydrogen sulphide can be removed by passing the gas over pumice stone soaked in copper sulphate solution, and sulphur dioxide by means of acidified 2N permanganate solution. Commercial nitrogen, prepared by the Linde process, has only to be freed from traces of oxygen. The best wet absorbent is copper gauze in ammonium carbonate solution, the gas being subsequently passed through concentrated sulphuric acid to absorb ammonia. It can finally be passed over copper-asbestos heated to $700^\circ - 800^\circ$ C. to absorb the last traces of oxygen. Hydrogen, if prepared electrolytically, is practically free from impurities, but if prepared by the Linde process from water-gas it may contain air, carbon dioxide, carbon monoxide, and sometimes arsine. In many cases the hydrogen is contaminated with iron pentacarbonyl, formed by the action of carbon monoxide on the steel cylinder under pressure. Since it is practically impossible, by chemical means, to free the hydrogen from this impurity or its decomposition products, such samples are unsuitable for laboratory use. Commercial oxygen, prepared by the Linde process, contains only traces of nitrogen, which cannot be removed by chemical means, and a trace of carbon dioxide which can be removed by soda-lime. Electrolytic oxygen may contain up to 4% of hydrogen, which can be removed by passing the gas over heated platinised asbestos and then through sulphuric acid to remove the water formed. Commercial chlorine, which is generally prepared electrolytically, is purified by liquefying the gas from the cylinder in a tube immersed in a carbon dioxide-ether mixture, allowing impurities such as air and carbon monoxide to escape. Sulphur dioxide can be purified in the same way as chlorine, but the gas as supplied is pure enough for most purposes. Commercial ammonia is about 98% pure, the remainder consisting of water and organic substances, such as pyridine, methylamine, pyrrole, etc., which are difficult to remove. It can be dried by means of lime or soda-lime.

—E. H. R.

Nitroglycerin waste acid. Dodds. *See* XXII.

PATENTS.

Sulphuric and haloid acids; Production of —. H. Tobler, Hackensack, N.J., Assignor to American Bromine Co., Maywood, N.J. U.S. Pat. 1,332,581, 2.3.20. Appl. 9.1.19.

SULPHUR dioxide and a halogen are introduced into, and caused to combine in, a stream consisting of a mixture of the acids with water, the haloid acid being then separated by the action of sulphur dioxide alone.—W. J. W.

Nitric acid; Process for producing —. T. F. Banigan, Kenvil, N.J., Assignor to Hercules Powder Co., Wilmington, Del. U.S. Pat. 1,332,195, 2.3.20. Appl. 12.3.19.

A STREAM of a mixture of pulverised nitre-cake and sodium nitrate is subjected to a simultaneous tumbling and progressive movement, the supply end of the stream being kept at a low temperature, and the discharge end at a high temperature. The nitric acid produced is caused to flow in a contrary direction to the stream.—W. J. W.

Ammonia; Synthetic production of —. F. J. Metzger, New York. U.S. Pats. (a) 1,313,314, (b) 1,313,315, and (c) 1,313,316, 19.8.19. Appl., 12.6.18.

(A) High efficiency is attained in the catalytic production of ammonia from nitrogen and hydrogen by periodically changing the temperature during the reaction period in order to restore the catalyst. (B) Synthetic ammonia is produced by bringing a mixture of hydrogen and nitrogen into intimate contact with a fluid catalytic composition consisting of a finely divided catalyst in an organic liquid vehicle, *e.g.*, a finely ground mixture of cerium or cerium alloy and paraffin. (C) An unsaturated organic liquid, *e.g.*, cottonseed oil, is used as or in addition to the organic liquid vehicle specified under (B). The unsaturated substance acts as a "promoter" of catalytic activity.—L. A. C.

Ammonia; Apparatus for the production of synthetic —. R. F. Gardiner, Clarendon, Va. U.S. Pat. 1,333,404, 9.3.20. Appl., 28.5.19.

The apparatus consists of an electrolytic cell of the suction type, packed with a diaphragm of "filtros," "fruit pit," and cotton, saturated with an alkaline solution. (See also U.S. Pat. 1,328,082; this J., 1920, 231 A.)—W. E. F. P.

Ammonia; Process of recovering — by means of sodium bisulphate. G. N. Vis, Paris. U.S. Pat. 1,332,419, 2.3.20. Appl., 25.10.18.

SODIUM bisulphate is treated with ammonia to produce crystallised sodium ammonium sulphate, and the latter is heated with ammonium sulphate solution. The insoluble residue (sodium sulphate) is then removed, and the solution is cooled to produce crystals of ammonium sulphate.—W. E. F. P.

Ammonia coolers; Process and apparatus for the working of —. A. Klönne. Ger. Pat. 314,362, 8.12.17.

FLUCTUATIONS of pressure between the ammonia cooler and the receptacle for condensed liquid, which is connected to the former through an overflow pipé, are equalised, without loss of gas, by means of a device parallel to the overflow pipe. This device consists of an outer cylinder communicating by a pipe with the gas inlet to the cooler, and an inner cylinder communicating by a pipe with the lower part of the cooler and with the overflow pipe. Perforations around the base of the inner cylinder, which are normally covered by a water-seal, enable gas to pass from the outer to the inner cylinder in the event of a pressure difference sufficient to uncover the perforations. Thus whenever the pressure of gas at the inlet to the cooler exceeds a certain value, gas passes directly through the cylinders to the base of the cooler and the overflow pipe.

—J. H. L.

Ammonium bisulphate; Process for producing — from ammonium sulphate. Badische Anilin- und Sodafabrik, Ludwigshafen. Ger. Pat. 315,622, 16.9.17.

NORMAL ammonium sulphate, which can be readily prepared by interaction of ammonium carbonate with natural sulphates, is heated directly by flame gases, preferably rich in steam, and the fused ammonium bisulphate is withdrawn from the hot reaction zone as rapidly as possible.—J. H. L.

Cyanogen compounds; Process of manufacturing —. H. Foersterling, H. Philipp, and R. N. Sargent, Perth Amboy, N.J., Assignors to The Roessler and Hasslacher Chemical Co., New York. U.S. Pat. 1,332,439, 2.3.20. Appl., 29.10.13. Renewed 14.1.18.

A VOLATILISABLE metal and nitrogen are brought into contact with excess of charcoal and heated to

about the boiling point of the metal. When the commencement of the reaction is denoted by an appreciable rise in temperature, the extraneous heat is discontinued, and the combination of the elements is allowed to proceed by means of the heat of reaction, most of the cyanogen compound produced being absorbed by the excess of charcoal.—W. J. W.

Alumina; Method for the production of —. B. F. Halvorsen, Christiania. U.S. Pat. 1,333,020, 9.3.20. Appl., 30.10.19.

ALUMINA is produced from solutions of aluminium compounds by heating with ammonia under pressure.—W. J. W.

Hydrosulphite liquors; Preserving — from decomposition. P. MacIntyre, Clinton, Mass. U.S. Pat. 1,333,029, 9.3.20. Appl., 13.8.19.

THE decomposition of hydrosulphite liquors is prevented by keeping them at a sufficiently low temperature and excluding oxidising influences.

—W. J. W.

Titanium compounds from titaniferous material; Process of extracting — and the product obtained thereby. G. Jebesen, Assignor to Titan Co. A./S., Christiania. U.S. Pat. 1,333,819, 16.3.20. Appl., 12.10.17.

FERROTITANIFEROUS material is treated with sulphuric acid, and the resulting slime is heated to produce a solid mass, which is lixiviated to obtain a solution of titanium and iron compounds. The quantity of acid employed is less than that required to form normal salts with the base-forming material present in the resulting solution.—W. E. F. P.

Magnesium carbonate; Process for producing light basic —. "Lipsia" Chem. Fabrik, Mügeln. Ger. Pat. 301,733, 20.2.17.

A SOLUTION of a magnesium salt (*e.g.*, waste salts from the potash industry) is treated with ammonium magnesium carbonate at a temperature not above 55° C., and the magnesium carbonate produced is separated and converted into the basic carbonate by heating with water. A very light normal carbonate is obtained which on boiling with water yields a basic carbonate about twice as voluminous as itself.—J. H. L.

Soda alum; Process for producing —. R. Linkmeyer, Barby. Ger. Pat. 303,981, 21.6.17. Addition to 301,641 (this J., 1920, 64 A).

IN the process of the original patent, water is added to or evaporated from the melt as required, in order to maintain approximately equimolecular proportions of water and bisulphate during the reaction period. The reaction temperature is thereby kept within the required limits, and the melt acquires the right physical characters.—J. H. L.

Nitrous gases; Process for absorbing — in stages. H. Pauling, Berlin-Lichterfelde. Ger. Pat. 306,353, 13.12.17.

THE gases, *e.g.*, as formed by combustion of air, are subjected to counter-current absorption with the absorbent alternately at higher and lower temperatures. The absorbent may be circulated so as to return once in every cycle to a point of minimum temperature; it may be passed over two towers and conducted over a heater in passing from the first to the second tower and over a cooler in passing from the second to the first, so that the gases give up heat to the absorbent in the first tower and withdraw heat therefrom in the second. This arrangement promotes the oxidation of nitric oxide at the higher temperature, and the absorption of the nitrous gases at the lower one.—J. H. L.

Soda; Process for the production of ammonia —. R. Schad, Darmstadt. Ger. Pat. 315,549, 26.4.17.

BEFORE the separation of the bicarbonate from its mother liquor sufficient water is added to dissolve the whole of the ammonium chloride present. The washing and drying of the bicarbonate are thereby facilitated.—J. H. L.

Mercuric oxide; Electrolytic production of — from mercury. Consortium für elektrochem. Industrie G.m.b.H. Ger. Pat. 315,656, 31.3.18.

THE alkaline electrolytic liquor is treated with organic substances possessing the character of protective colloids, e.g., glue, starch, waste liquors from the treatment of wood and cellulose, caramel, dextrin, etc. The current efficiency is thereby increased almost to the theoretical, and the mercuric oxide obtained deposits slowly and can easily be separated, by sedimentation, from metallic mercury.—J. H. L.

Sulphur retort. F. W. Beecraft and A. L. Genter, Salt Lake City, Utah, Assignors to United Filters Corporation, New York. U.S. Pat. 1,332,537, 2.3.20. Appl., 25.2.19.

A HORIZONTAL, rotary cage for containing sulphur ore is enclosed in a casing having a stationary head and a shell portion which is movable lineally with respect to the head.—W. E. F. P.

Sulphur; Process for obtaining — by heating gypsum with reducing agents. F. Weeren, Neukölln. Ger. Pat. 301,712, 30.9.15.

SULPHUR is produced by heating gypsum with reducing agents in presence of silicious fluxes in a blast furnace, the gypsum and the silicious fluxes being introduced into the furnace in lumps. A freely flowing slag is obtained free from sulphuric acid, calcium sulphate, or calcium sulphide.

—J. H. L.

Hydrogen; Apparatus for producing — from steam. K. Zöller, Charlottenburg. Ger. Pat. 300,761, 6.3.17.

IN the production of hydrogen from steam by alternate oxidation and reduction of iron ore or other contact agent, the latter is contained in two or more columns so connected that first one and then the other receives the reducing gases, the excess of which, mixed with air, serves to preheat the alternate column, whilst the steam is conducted in the reverse direction. The contact material is heated directly, the usual superheating being omitted.

—J. H. L.

Hydrogen; Process for producing — from hydrocarbons and steam. Badische Anilin- und Soda-Fabrik, Ludwigshafen. Ger. Pat. 306,301, 21.8.14. Addition to 296,866.

IN the production of hydrogen from hydrocarbons and steam at high temperatures by means of nickel as catalyst, the steam is partially or entirely replaced by carbon dioxide. The reaction, $\text{CH}_4 + \text{CO}_2 = 2\text{CO} + 2\text{H}_2$, takes place to an extent which increases with rise in temperature and in the concentration of carbon dioxide. A second reaction, however, $\text{CH}_4 + 3\text{CO}_2 = 4\text{CO} + 2\text{H}_2\text{O}$, is usually the predominant one, but the products can be converted into carbon dioxide and hydrogen by subsequent treatment with a suitable catalyst at lower temperatures.—J. H. L.

Chlorides; Preparation of anhydrous metallic —. P. L. Hulin, Grenoble, France. Eng. Pat. 140,114, 7.5.18. (Appl. 7661/18.) Addition to 128,327.

SEE U.S. Pat. 1,304,567 of 1919; this J., 1919, 630 A.

Barium hydroxide; Process for preparing — from barium sulphide. E. A. Barnes, Allentown, Pa., U.S.A. Eng. Pat. 140,297, 18.7.19. (Appl. 17,991/19.)

SEE U.S. Pat. 1,316,133 of 1919; this J., 1919, 819 A.

Ammonia; Synthetic production of —. G. Claude, Assignor to L'Air Liquide (Soc. Anon. pour l'Étude et l'Exploit. des Proc. G. Claude), Paris. U.S. Pat. 1,332,460, 2.3.20. Appl., 14.3.18.

SEE Eng. Pat. 130,086 of 1918; this J., 1919, 718 A.

Ammonium nitrate; Manufacture of —. J. R. Partington, Lostock Gralam, and G. J. Jones, London. U.S. Pat. 1,306,924, 17.6.19. Appl., 10.12.18.

SEE Eng. Pat. 134,562 of 1918; this J., 1920, 266 A.

Ammonium nitrate; Production of —. J. R. Partington, G. J. Jones, and T. K. Brownson, London. U.S. Pat. 1,330,136, 10.2.20. Appl., 10.12.18.

SEE Eng. Pat. 136,190 of 1918; this J., 1920, 266 A.

Titanium and iron salts; Solution of — and process of preparing same. H. Olsen, Christiania, and T. R. Förland, Haugesund, Assignors to Titan Co. A./S., Christiania, Norway. U.S. Pat. 1,333,849, 16.3.20. Appl., 12.10.17.

SEE Ger. Pat. 310,304 of 1917; this J., 1919, 363 A.

Potassium salts; Extracting — from potash-bearing minerals. A. C. Auden, London. U.S. Pat. 1,334,940, 23.3.20. Appl., 28.11.19.

SEE Eng. Pat. 132,855 of 1918; this J., 1919, 861 A.

Liquefying air; Apparatus for —. J. F. Place, Glen Ridge, N.J. U.S. Pat. 1,326,961, 6.1.20. Appl., 14.2.16.

SEE Eng. Pat. 105,106 of 1916; this J., 1917, 548.

Liquefaction of air. Ger. Pat. 315,511. See I.

Heat insulating material. Ger. Pats. 304,239—40. See IX.

Potassium compounds. Eng. Pat. 130,963. See XVI.

VIII.—GLASS; CERAMICS.

Striae in optical glass; Comparison tests for — by the Brashear converging light, direct view, Bureau of Standards' tank immersion, and short range projection methods. L. E. Dodd. J. Amer. Ceram. Soc., 1919, 2, 977—1006.

THE author determined the number of striae in 117 samples of glass by each of the four methods mentioned in the title and found that, when the samples have polished ends, the short range projection method is the best for regular use. A beam of light is projected from a small lamp of special shape, giving a line of light 2.4 mm. long and 0.5 mm. wide, through the glass to be examined on to a sheet of plate glass with an opal backing. Shadows of any striae in the specimen are projected on to the screen.—A. B. S.

Cristobalite; Latent heat of fusion of —. E. W. Washburn. J. Amer. Ceram. Soc., 1919, 2, 1007—1008.

THE latent heat of fusion of cristobalite in binary mixtures of silica and alumina, very rich in silica, calculated from the lowering of the freezing point, is found to be 6950 cal. per mol. if the solute is sillimanite, or 6650 cal. per mol. if it is alumina. The weighted average of these results is 6800 cal. per mol. or 110 cal. per gram. If the

solute consists of associated molecules the calculated latent heat of fusion will be too large. (Cf. J. C. S., May.)—A. B. S.

Enamel-lined apparatus; Manufacture of —. E. P. Poste. J. Amer. Ceram. Soc., 1919, 2, 944—976.

A DETAILED, illustrated description of one of the customary methods of enamelling steel and cast iron, including recipes for enamels, method of fritting, grinding, applying and burning the enamel. The use of the microscope in studying the effect of over- or under-firing is recommended.

—A. B. S.

PATENTS.

Kiln [; Ceramic —]. R. C. Purdy, Worcester, Mass., Assignor to Norton Co. U.S. Pat. 1,332,471, 2.3.20. Appl., 15.6.18.

A CIRCULAR kiln with several inlet flues beneath the floor has fire-boxes opening into the flues, a baffle wall or bag wherein the gases from the fire-boxes and flues are mixed, a conduit from the mixing chamber to the exterior of the heating chamber of the kiln, and means for removing the gases from the latter.—A. B. S.

Tunnel kiln. G. W. Booth, Islington, Ontario. U.S. Pat. 1,333,099, 9.3.20. Appl., 17.5.19.

A TUNNEL kiln has, in its walls, an air space extending longitudinally through the kiln. At one end of the firing zone is a furnace and at the other end a chimney stack both communicating with the interior of the kiln. A flue in the lower part of the tunnel communicates at one end with the stack and with the aforesaid air space at the other end.—A. B. S.

Carboys or large glass containers; Making —. H. M. Brown, Assignor to Spring Stopper Co., Brooklyn, N.Y. U.S. Pats. 1,334,647—8, 23.3.20. Appl., 2.12.18 and 28.2.19.

IX.—BUILDING MATERIALS.

Cement; Effect of calcium sulphate on —. J. C. Witt. Philippine J. Sci., 1919, 14, 221—232.

A KNOWN amount of gypsum was added either to the finished cement or to clinker, the mixture was ground in a small ball mill, analysed to determine the SO_2 content and subjected to the usual physical tests. With the particular cement tested, for laboratory samples containing up to 2.5% SO_2 , the time of setting was practically independent of the SO_2 content; but when the clinker was ground in the usual way in the cement plant a quick or "flash" set was obtained if the SO_2 content was below 1.5%, an irregular or erratic set for a SO_2 content between 1.5 and 1.8%, and a normal slow setting for a SO_2 content between 1.8 and 2.1%, the higher limit being the maximum allowed by the U.S. Government specifications. The effect of exposure of the cement to air was very variable, and was independent of the SO_2 content. Experiments on the addition of lime up to 2% to cement with SO_2 content varying from 1.5 to 2.0% showed that the setting time increases with the proportion of lime, but the effect is independent of the SO_2 content.—W. F. F.

Asphalt rock; Synthetic — with special reference to Zimmer's process. Kröhnke. Chem.-Zeit., 1920, 44, 253—255.

NATURAL asphalt stone can be compressed to a hard and compact mass, whereas the artificial products obtained by mixing bitumen with finely-powdered mineral matter (limestone) fail in this respect. Natural asphalt stone heated to 120° C. crumbles to a brown powder which can be stamped to a hard mass of higher density. If it be heated to a higher temperature than 150° C. it loses this property and gives a "dead" product which is poor and sandy

in texture. Zimmer considers that the mineral matter in rock asphalt is chemically united with the bitumen through the medium of salts of sulphonated acids which have resulted in the course of the production of petroleum. The properties of Zimmer's asphalt (U.S. Pats. 1,237,852 and 1,319,918; this J., 1917, 1095; 1919, 948 A) are compared with those of Swiss, Abruzzi, and Sicilian products, and it is considered that the artificial material, on account of its higher dropping point and lower solidifying point, is superior to the natural substance. The composition and properties of a number of artificial rock asphalts are tabulated.—A. E. D.

Wood; Effect of viscosity on the penetration of creosote into —. E. Bateman. Chem. and Met. Eng., 1920, 22, 359—360.

A DEFINITE relationship exists between the absolute viscosity of creosote and its penetration into wood. For longleaf pine and noble fir with a time of treatment of 2 hrs. and a pressure of 75 lb. per sq. in., for the longitudinal penetration, $yx=k$; for tangential penetration, $y\sqrt{x^3}=k_1$; and for radial penetration, $yx^2=k_2$; x being the penetration, y the absolute viscosity, and k_1 , k_2 , and k_3 being constants. In a large number of experiments the maximum value of xy was found to be 0.6, the minimum value 0.09, and the average value 0.3. Change of temperature of the oil has no other effect than that of changing the viscosity, nor has the presence of free carbon in tar creosote any other effect than is to be expected from increased viscosity. Factors affecting the penetration are viscosity, time of treatment, pressure during the application, and variations in the wood, but the influence of changes in the last three is not definitely known.—W. J. W.

PATENTS.

Woods; Process for hardening [fireproofing] and preserving permeable soft —. A. Lambert, Rome. Eng. Pat. 139,974, 11.6.19. (Appl. 14,789/19.)

PERMEABLE soft woods are impregnated under pressure, with an ammoniacal solution of copper and zinc salts, borax, potassium chromate, and Portland cement, with or without salicylic acid and sodium fluoride.—A. B. S.

Lumber; Process for the drying and curing of —. O. E. Jacobs, Kalamazoo, Mich. U.S. Pat. 1,333,848, 16.3.20. Appl., 15.9.16.

LUMBER, enclosed in a suitable vessel, is heated to about 100° F. (38° C.) to air-dry the surface, and then treated with live steam at about 20 lb. pressure until it has become evenly heated throughout to a temperature not exceeding 250° F. (121° C.). Steam is then blown off and the chamber is subjected to the action of a vacuum, which is continued, with the lumber maintained at 100°—120° F. (38°—49° C.), until the latter is dried.

—W. E. F. P.

Heat-insulating material and process of making the same. R. B. Macdonald, Oakland, Cal., Assignor to W. H. Winkley, Los Angeles, Cal. U.S. Pat. 1,332,348, 2.3.20. Appl., 1.7.18.

A HEAT-INSULATING material is formed from a mixture of heat-insulating earth, alkali, starch, and water, a portion of the alkali being combined with the earth to form a silicate, and the material having a large number of air-cells formed by the drying of the starch.—A. B. S.

Heat-insulating material and process of making the same. W. H. Winkley, Los Angeles, Cal. U.S. Pat. 1,332,370, 2.3.20. Appl., 14.8.19.

THE material contains heat-insulating earth, sodium carbonate, hydrated lime, starch, and water.

—W. E. F. P.

Insulation; Process for utilising the final liquors of the potash industry for the production of materials for heat —. *Process for producing [heat] insulating materials from basic magnesium carbonate and fibres.* "Lipsia" Chem. Fabrik, Mügeln. Ger. Pats. (a) 304,239 and (n) 304,240, 8.7.15. Additions to 303,310.

(a) The magnesia contained in the final liquors of the potash industry is precipitated as carbonate, $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$, on suitable fibrous tissue. On drying, the carbonate is converted into basic carbonate, and a very light, porous product of low thermal conductivity is obtained. (n) Hydrated magnesium carbonate is first produced and then converted into basic carbonate and precipitated on fibrous tissue.

—J. H. L.

Cements and similar products; Manufacture of —. M. L. Boillot and J. Daudignac, Moutiers, France. U.S. Pat. 1,332,422, 2.3.20. Appl., 28.6.18.

A MIXTURE of the finely-divided constituent materials is melted in an electric furnace, and the molten mass is kept in motion by the action of the electric arc until discharged.—W. E. F. P.

Magnesium oxychloride cement. J. B. Shaw and G. A. Bole, Alfred, N.Y. U.S. Pat. 1,333,510, 9.3.20. Appl., 24.2.19.

MAGNESIAN limestone is calcined under such conditions as will form magnesia without decomposing the calcium carbonate and is then mixed with a mixture of chlorides containing more than 50% of calcium chloride.—A. B. S.

Material for deadening sound and other purposes and method of making same. H. B. MacFarland, Chicago, Ill., and R. J. Shoemaker, Topeka, Kans., Assignors to E. H. Gold, Chicago, Ill. U.S. Pat. 1,333,624, 16.3.20. Appl., 3.1.16.

A COHERENT, felted body of the fibres of *Zostera marina* is treated superficially with an acid, whereby a hard and adherent film is produced on the surface of the mass.—W. E. F. P.

Portland cement; Manufacture of —. W. J. Mellersh-Jackson, London. From F. Krupp A.G. Grusonwerk, Magdeburg-Buckau, Germany. Eng. Pat. 16,530, 10.7.14.

SEE Ger. Pat. 304,080 of 1911; this J., 1918, 374 A.

Mortar-forming material from anhydrite; Manufacture of a —. F. Hartner, Bad Homburg, Germany. Eng. Pat. 117,605, 17.5.18. (Appl. 8305/18.) Int. Conv., 18.7.17.

SEE Ger. Pat. 312,239 of 1917; this J., 1919, 905 A.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Nitrogen in steel; Effects produced by —. G. F. Comstock and W. E. Ruder. Chem. and Met. Eng., 1920, 22, 399—405.

AN outline is given of published researches on nitrogen in steel, together with photo-micrographs showing the structures produced. Experiments were conducted to determine the effect of heat treatment, especially the rate of cooling, on the micro-structure of nitrogenised pure iron. Five samples were cut from a $\frac{1}{4}$ in. swaged bar of pure, vacuum-fused, electrolytic iron which had been heated for 3 hours to 700° C. in a stream of ammonia gas. Four of these were heated for 15 mins. to 600°, 700°, 800°, and 950° C. respectively, and then quenched with water, after which they were again heated for 15 mins. at the same temperatures and allowed to cool with the furnace. The fifth sample was heated to 1000° C. and slowly cooled. In the first case, the

nitride needles were absorbed by the ferrite and held in solution in the quenched sample, appearing as pearlitic patches and intergranular material in the slowly cooled sample. In sample 2, quenched, the grains were larger, and the nitrogen only appeared at the boundaries. The presence of needles in the slowly cooled sample indicates that a minimum temperature of 700° C. with slow cooling is necessary for their appearance. The third sample showed similar results to sample 2, except for lighter and wider grain boundaries. At 950° C. (sample 4) all the nitride was absorbed and retained in solution on quenching, and after slow cooling the grain was smaller and the nitride appeared as needles at the boundaries. Diffusion inwards of the outside nitride band had also taken place. The fifth sample showed needles and widened grain boundaries and rapid diffusion inwards of the outside nitride band. The experiments show that nitrogen occurs in various forms, dependent on treatment, and may not be visible at all, even in highly nitrogenised steel. The dark pearlitic patches do not necessarily denote carbon, but are a function of the nitrogen present and the rate of cooling. The needles are not Neumann's lines or slip bands caused by cutting, as they neither disappear on heating above A_c , nor appear in the quenched as well as the slowly-cooled samples. These lines appear to be due to a nitride of iron, soluble at 700° C. in ferrite, retained in solution by quenching but reappearing at the boundaries on slow cooling.—W. J. W.

Iron objects; Cooling of highly heated —. F. Riedel. Stahl u. Eisen, 1920, 40, 1—9.

FORMULÆ have been devised for calculating the rate of cooling of highly heated iron objects by radiation and by conduction. Results of experiments on heat radiation were in fair agreement with the calculated results. The temperature distribution in the interior of the iron bodies during cooling is also discussed, and the results of some experiments are given.—J. W. D.

Gold and silver; Electro-deposition of — from cyanide solutions. S. B. Christy. U.S. Bureau of Mines. Bull 150. 1919. Pp. 171.

THE paper, which embodies the results of twenty years' investigations on the electro-deposition of gold and silver from dilute cyanide solutions such as are obtained in the leaching of ores, contains a review of all the methods that have been proposed, more especially those which the author himself has worked out (see U.S. Pats 613,096 of 1900; 756,328 of 1904; and 883,170 of 1908; this J., 1901, 494; 1908, 454). Clear solutions and rapid circulation are necessary for good results. A large cathode area and an excess of potassium cyanide above that required to form the double cyanides are necessary for rapid and complete precipitation, and, if the voltage is kept low and the circulation rapid, a considerable amount of cyanide is regenerated. The current should be stopped at the point at which the amperage becomes constant. The use of pervious electrodes (fragments of carbon packed in boxes) both as anodes and cathodes has been adopted. A complete description of the construction and working of a model plant is given. The cell consists of an oblong wooden box containing a simple pervious cathode at one end and a simple pervious anode at the other. The intervening space is filled up with a series of compound pervious electrodes (U.S. Pat. 883,170; this J., 1908, 454), $\frac{1}{4}$ in. apart with a potential of 3.5 volts between each. The electrodes are all made up of "Excelsior" charcoal (carbonised "Excelsior fibre") packed in wooden frames, both sides of which are covered with cheesecloth. The solution is circulated through the box by means of a small centrifugal pump; the speed of circulation is determined to a large extent by the current gap, a $\frac{1}{4}$ in. gap giving

three times the capacity that a $\frac{1}{2}$ in. gap gives. In the former case, for example, for a 96% precipitation a maximum capacity of 144 tons per 25 hours for a 1-ton box was obtained.—A. R. P.

Zinc; Reactions of the metallurgy of —. M. Lemarchands. *Comptes rend.*, 1920, 170, 805—807.

In an atmosphere of nitrogen zinc oxide is readily reduced directly by carbon without the intermediate formation of carbon monoxide. The amount of carbon entering into the reaction is intermediate between the amounts necessary if carbon monoxide or carbon dioxide is respectively the gaseous product. In explanation of this the author states that the reduction of zinc oxide by carbon monoxide takes place with a velocity superior to the reduction of carbon dioxide by carbon and commences at a lower temperature, i.e., 350° C. as against 400° C. for the latter.—W. G.

Electrolytic zinc production; Method for analytical control of —. H. F. Bradley. *Chem. and Met. Eng.*, 1920, 22, 651—65.

A DESCRIPTION is given of methods for analysing leaching and electrolyte solutions, concentrates, calcine and leach residues, zinc dust precipitate, and anode scale from cells.

Roasting; Physical chemistry of —. II. *Equilibria in the system lead-sulphur-oxygen*. W. Reinders. *Z. anorg. Chem.*, 1919, 109, 52—62.

THE results of Schenck and Albers (this J., 1919, 370 A) are criticised and shown to be untrustworthy and in some cases, particularly the quintuple points in the system lead-sulphur-oxygen, impossible.—J. F. S.

Coal dust in steel mills. Tracy. See IIA.

Catalysis. Dhar. See VII.

Films of metalloids and metals. Braly. See XXIII.

PATENTS.

Aluminium; Solder for —. F. F. Boisselier, Colombes, France. *Eng. Pat.* 124,715, 30.1.19. (Appl. 2288/19.) *Int. Conv.*, 26.3.18.

AN alloy of the composition Al 28%, Zn 36.5—37.5%, Sn 33%, Cu 1.5—2.5%.—J. W. D.

Solder. C. W. Hill, Edgewood, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U. S. Pat. 1,333,619, 16.3.20. *Appl.*, 22.7.18.

A SOLDER consisting of 6—10% cadmium and 94—90% lead, and containing a small but appreciable amount of zinc.—A. R. P.

Solder. G. P. Luckey, Wilksburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U. S. Pat. 1,333,666, 16.3.20. *Appl.*, 13.3.18.

A SOLDER consisting of an alloy of 90—94% lead and 10—6% cadmium.—A. R. P.

Electroplating. C. A. Bolton, Blagden Hill, Somerset. *Eng. Pat.* 133,967, 17.1.19. (Appl. 1271/19.)

COPPER is deposited electrolytically on aluminium or its alloys by immersing the article in strong nitric acid, then quickly "flashing" it electrolytically in a bath consisting of an acid solution of cupro-nickel sulphate or nitrate, and finally plating in an acid bath of copper sulphate or nitrate.—B. N.

Aluminium or its alloys; Preparing for electroplating —. H. D. Cunningham, Brighton. *Eng. Pat.* 139,967, 4.6.19. (Appl. 14,111/19.)

THE articles are scoured with an abrasive such as

pumice, or brickdust moistened with water at 80° F. (27° C.), washed, then either subjected to a second scouring with abrasive material moistened with a solution of copper sulphate at 90° F. (32° C.) to produce a thin coating of copper, or placed in an acidulated bath of copper sulphate, and subsequently a thicker coating of copper is deposited by electrolysis using a low voltage.—J. W. D.

Metal; Method of preparing — to be galvanised or coated. H. G. Harmeling, Paducah, Ky., Assignor to J. L. Fay, St. Louis, Mo. U. S. Pat. 1,334,092, 16.3.20. *Appl.* 29.7.18.

THE metal is heated and subjected to the action of hot acetic acid solution.—J. W. D.

Metals; Process for coating substances having greatly developed surface, with heavy — or insoluble salts thereof. H. Bechhold, Frankfurt. *Ger. Pat.* 309,187, 11.5.18.

THE substance to be coated is allowed to absorb a soluble salt of the heavy metal, from aqueous solution, and the salt is then converted into an insoluble compound or reduced to metal. For example, 100 grms. of bolus is digested with 1 litre of 0.4% copper sulphate solution; the absorbed salt is converted into hydroxide by means of sodium hydroxide, and then warmed. The oxide layer thus produced may be reduced by means of hydrogen or hydrazine. A deposit of silver on charcoal may be similarly obtained, by means of formaldehyde.—J. H. L.

Ore concentration. E. C. R. Marks, London. From Luckenbach Processes, Inc., San Francisco, Cal., U. S. A. *Eng. Pat.* 139,535, 4.11.18. (Appl. 18,030/18.)

THE ore is brought in contact with a mineral selective agent, e.g., a mixture of rubber solution and petroleum grease (see also *Eng. Pats.* 133,277 and 133,498; this J., 1919, 909 A), supported on a travelling belt, rotating cylinder, or rotating table, or by a matted or tangled mass of material such as wood shavings or mineral wool. The valuable mineral constituents adhere to the selective agent and may be subsequently separated by treatment with hot water, centrifuging, or the like.—J. W. D.

Furnaces; Tilting crucible or melting pot —. Soc. des Alliages et Bronzes Forgeables, Paris. *Eng. Pat.* 140,015, 26.9.19. (Appl. 23,746/19.) *Int. Conv.* 21.7.19.

THE axis of the trunnions on which the tilting takes place is tangential to the edge of the mouth of the crucible at the point or very close to the point over which the molten metal flows on pouring.—J. W. D.

Carbonizing [case-hardening] furnace. J. H. L. De Bats, Zelenople, Pa., Assignor to New Process Metals Corporation, Wilmington, Del. U. S. Pat. 1,333,796, 16.3.20. *Appl.*, 19.8.18.

A CARBONISING furnace consists of a hermetically closed rotating drum, to receive the carbonising material and the metal to be carbonised, and an interior flue, which is arranged along the axis of the drum, and is heated by means of an injector burner.—A. R. P.

Blast furnace. C. H. Mace, Denver, Colo., Assignor to A. T. Carozza, Baltimore, Md. U. S. Pat. 1,334,310, 23.3.20. *Appl.*, 28.9.17.

THE tuyères between the shaft and crucible are diametrically opposed, rectangular in section, with straight parallel sides. The continuation of the sides form connecting surfaces between opposite tuyères.—W. F. F.

Molten metal; Removing impurities from —. W. White, Washington, D.C. U.S. Pat. 1,332,585, 2.3.20. Appl., 1.11.18.

GASES and other impurities are removed from molten metal contained in a crucible, by imparting to the metal a rotary motion by means of a paddle, whereby the surface of the metal is displaced into a vertical position around the sides of the crucible, and the sound metal is forced towards the exterior of the mass. The rotary movement is then slowly reduced so as to allow the metal to fall back gradually to its normal position, and the portions of the charge containing the impurities are separated from the sound metal.—A. R. P.

Tungsten crucible and method of making and using same. J. H. Robertson and D. Jankower, New York. U.S. Pat. 1,333,036, 9.3.20. Appl., 14.7.19.

A CRUCIBLE is made from a disc of sheet tungsten which is laid over the top of a die electrode; the other electrode, in the form of a plunger, is brought down to make contact with the disc, and, when the latter is hot and in a pliable condition, presses it into the die, thereby forming the crucible. Alternating current is used and a regulating rheostat is included in the circuit.—A. R. P.

Copper-lead smelting process. W. McA. Johnson, Hartford, Conn. U.S. Pat. 1,333,720, 16.3.20. Appl., 24.10.14.

AN acid and a basic ore mixture are heated to a high temperature (1200° C.) in separate rotary kilns and then mixed and smelted by means of internally developed heat in a closed container, i.e., an electric furnace. The charge is tapped into, and allowed to settle in, two electrically heated settling hearths arranged in series, and products (lead, matte, and slag) are tapped as usual.—A. R. P.

[Zinc] refining process; Ebullient —. W. McA. Johnson, Hartford, Conn. U.S. Pat. 1,333,721, 16.3.20. Appl., 12.2.16.

IMPURE spelter from an electric furnace or retort plant is melted in a settling furnace and the top layer is tapped off into an electric distilling furnace. The vapours are passed through a layer of zinc oxide to remove from the spelter any volatile impurity having a greater affinity for oxygen than zinc. The last fraction of the metal in the distilling furnace is returned to the settling furnace at the end of the day's run and the heavy lead-iron alloy, which sinks to the bottom of the latter, is tapped once a week and returned to the plant.—A. R. P.

Alloy. F. A. Fahrenwald, Cleveland, Ohio. U.S. Pat. 1,333,965, 16.3.20. Appl., 25.6.17.

A STRONG, hard, and light alloy contains aluminium and less than 65% of beryllium (glucinum).

—A. E. D.

Magnesium alloy. W. R. Veazey, Cleveland, Ohio, Assignor to The Dow Chemical Co., Midland, Mich. U.S. Pat. 1,334,193, 16.3.20. Appl., 10.10.18.

AN alloy of magnesium with relatively small proportions of manganese and copper.—J. W. D.

Sodium; Method of making metallic —. A. W. Smith and W. R. Veazey, Cleveland, Ohio, Assignors to The Dow Chemical Co., Midland, Mich. U.S. Pat. 1,334,179, 16.3.20. Appl., 12.10.18.

METALLIC sodium is produced by electrolysis of a fused mixture of sodium carbonate and sodium chloride in such proportions that the mixture has a lower melting point than either of its constituents.

—J. W. D.

Melting furnaces and the like; Lids for —. Akt.-Ges. Brown, Boveri & Co., Baden, Switzerland. Eng. Pat. 131,887, 25.8.19. (Appl. 20,858/19.) Int. Conv., 23.8.18.

Furnaces for heat treatment of metals. D. M. Cunningham, Armadale, Scotland, and Stein and Atkinson, Ltd., London. Eng. Pat. 140,238, 23.4.19. (Appl. 10,132/19.)

Separating minerals from ore-bearing materials; Machine for —. R. P. Park, South Melbourne, Victoria. Eng. Pat. 139,661, 14.4.19. (Appl. 9510/19.)

Steel; Basic Bessemer process for production of —. W. Goutermann, Siegen, Germany. Eng. Pat. 115,027, 4.4.18. (Appl. 5811/18.) Int. Conv., 4.4.17.

SEE Ger. Pat. 302,768 of 1917; this J., 1918, 377 A.

Iron; Manufacture of —. G. J. Stock, Assignor to H. H. Blackburn, Bradford. U.S. Pat. 1,333,821, 16.3.20. Appl., 11.10.17.

SEE Eng. Pat. 130,343 of 1916; this J., 1919, 727 A.

Rust-proofing of iron and steel articles. J. A. Thompson, Smethwick, and G. H. Alexander, Birmingham. U.S. Pat. 1,318,027, 7.10.19. Appl., 8.11.18.

SEE Eng. Pat. 129,831 of 1918; this J., 1919, 727 A.

Sulphide ores; Process of treating —. M. Sem, Assignor to Det Norske Aktieselskab for Elektro-kemisk Industri, Norsk Industri-Hypotekbank, Christiania. U.S. Pat. 1,317,179, 30.9.19. Appl., 15.6.16.

SEE Ger. Pat. 313,122 of 1915; this J., 1919, 869 A.

Electric furnace for heating, annealing, and melting metallic material. M. Fossati, Assignor to Soc. Anon. Stabilimenti Biak, Turin, Italy. U.S. Pat. 1,322,416, 18.11.19. Appl., 6.3.19.

SEE Eng. Pat. 124,438 of 1919; this J., 1920, 29 A.

Coating with tin and tin alloys articles made of aluminium or aluminium alloys. M. N. Salvati (G. Beer and A. Tedesco, Exors.), Assignors to Soc. Anon. Stabilimenti Biak, Turin, Italy. U.S. Pat. 1,332,307, 2.3.20. Appl., 15.11.18.

SEE Eng. Pat. 134,315 of 1918; this J., 1920, 29 A.

Composite or compound metal; Manufacture of plates and large sheets of —. E. Martin, Assignor to Comp. Belge du Bi-Metal, Brussels. U.S. Pat. 1,332,674, 2.3.20. Appl., 13.2.19.

SEE Eng. Pat. 128,855 of 1919; this J., 1919, 642 A.

Copper; Recovery of — from its ores and solutions. H. L. Sulman and H. F. K. Picard, London. U.S. Pat. 1,333,688, 16.3.20. Appl., 27.12.13.

SEE Eng. Pats. 1152, 9171, and 16,017 of 1913; this J., 1914, 488.

XI.—ELECTRO-CHEMISTRY.

Nitrogen fixation. Steinmetz. See VII.

Electrical purification of air. D'Arsonval and others. See XIXb.

PATENTS.

Electrolysers; Mono-polar electrode —. G. G. Hepburn, Manchester. Eng. Pat. 139,296, 22.2.19. (Appl. 4472/19.)

A MONO-POLAR electrode electrolyser of the filter press type comprises a number of units, consisting of anodes, cathodes, frames, and diaphragms, each unit being electrically independent of the others, having independent liquid connections, and insulated from adjacent units.—B. N.

Batteries; Primary —. *Electric battery*. O. E. Ruhoff, Assignor to French Battery and Carbon Co., Madison, Wis. U.S. Pats. (A) 1,331,213, 17.2.20, and (B) 1,331,877, 24.2.20. Appl., (A) 9.11.18, (B) 5.7.18.

(A) A MATERIAL for electric batteries is made by adding a manganese compound to a suspension prepared by stirring deloculated conductive material with water, thoroughly agitating the mixture, adding sal ammoniac (ammonium chloride), or similar salt, again agitating the mixture and filtering. The material is dried, compacted, and the product ground, mixed with more carbonaceous conductive material and sal ammoniac. (B) A battery, containing carbon, powdered depolarising material, and zinc, is provided with a porous partition, which is inserted between the depolarising material and the zinc. The partition is soaked during the process of manufacture with a 1–5% solution of potassium bichromate or other salt of chromic acid, which will act on the zinc to form a product which protects it from the depolarising material when the battery is at rest, but does not affect the efficiency of the battery when in action.—B. N.

Electrolytic cell. E. A. Le Sueur, Ottawa, Canada. U.S. Pat. 1,331,571, 24.2.20. Appl., 25.1.19.

THE anode is provided with a conducting stem protected by a conducting sheath of resistant metal, and sheets of resistant metal are clamped against opposite sides of the sheath and extend from it in a lateral and downward direction.—B. N.

Plastic material practically incombustible, strong, and possessing insulating properties. D. Motta, Assignor to E. Rugiu and A. Villain, Genoa, Italy. U.S. Pat. 1,331,519, 24.2.20. Appl., 23.7.19.

A PLASTIC and elastic insulating material is prepared from paper paste without size and equal parts by volume of calcium carbonate and magnesium silicate, the weight of the combined carbonate and silicate being substantially the same as the paper paste.—B. N.

Coumarone-resin insulating material. A. A. Wells, Caldwell, N.J., Assignor to Ellis-Foster Co. U.S. Pat. 1,332,860, 2.3.20. Appl., 9.4.17.

A MOULDED electric insulator containing coumarone resin as an essential constituent.—A. de W.

Insulating liquids for transformers and switchboards; Incombustible or difficultly combustible —. H. Grossmann, Ober-Urdorf, Switzerland. Ger. Pat. 315,402, 17.2.14. Int. Conv., 31.1.14.

A NORMAL transformer oil is mixed with a more volatile incombustible insulating liquid, such as tetrachloroethane or epichlorhydrin, and a non-conducting organic base, such as aniline or dimethylaniline, which will neutralise acid decomposition products.—H. J. H.

Insulating or inflammable liquids; Process for electrically heating —. Allgem. Elektrizitäts-Ges., Berlin. Ger. Pat. 316,012, 25.10.18.

A CONDUCTING liquid carrying current is passed through a coil immersed in the liquid to be treated. The current is led in by electrodes. The heating liquid should have a high boiling point, and by maintaining it under increased pressure with the aid of a suitable safety-valve still higher temperatures can be attained.—H. J. H.

Tungsten or platinum wire coils; Process for removing metallic cores from —. Elektrische Glühlampenfabrik "Watt" A.-G., Vienna. Ger. Pat. 315,667, 18.2.19. Int. Conv., 14.3.18.

THE core is removed by an electrolytic process which leaves the coil untouched. The coil may be made

one pole of a cell, the other pole being carbon or another metal, or the two components of the coil may themselves be joined in the bath to form an electrolytic couple, whereby the core passes into solution.—H. J. H.

Electric muffle furnaces [; Doors for tilting —]. A. Imbery, Halifax. Eng. Pat. 140,138, 4.6.19. (Appl. 20,375/18.)

Electric resistance furnace. J. Simon and P. F. Sarron, Lyon, France. U.S. Pat. 1,334,809, 23.3.20. Appl., 13.11.18.

SEE Eng. Pat. 132,080 of 1918; this J., 1919, 779 A.

Electrical precipitation. U.S. Pat. 1,331,225. See I.

Electric precipitators. Ger. Pat. 314,626. See I.

Electrical precipitator. Ger. Pat. 314,947. See I.

Electrical precipitation. Ger. Pat. 315,534. See I.

Oxidation and reduction of gaseous compounds. U.S. Pat. 1,332,730. See I.

Ammonia. U.S. Pat. 1,333,404. See VII.

Mercuric oxide. Ger. Pat. 315,656. See VII.

Cements. U.S. Pat. 1,332,422. See IX.

Sterilising liquids. U.S. Pat. 1,333,633. See XIXb.

XII.—FATS; OILS; WAXES.

Oils; Splitting of — by means of castor seed lipase. J. J. Sudborough, H. E. Watson, and P. S. Varma. J. Indian Inst. Sci., 1919, 2, 241–265.

NICLOUX's method of enzymic hydrolysis (this J., 1904, 327, 942) was found the most suitable for the treatment of oils on a large scale. A lipase preparation from decorticated castor seeds is more active than an equivalent quantity of enzyme from undecorticated seeds. The activity of castor seed lipase begins to diminish after three days from the date of preparation. By the use of manganous sulphate as activator (0.2 part per 100 parts of oil and 4 parts of crushed castor seed) 90 to 95% of hydrolysis is effected in 48 hours. Samples of concentrated crude fermentation glycerin prepared by enzymic hydrolysis gave analytical results equal to those of a Twitchell crude glycerin, but yielded an inferior distillate and left a larger residue.

—C. A. M.

Oil splitting with germinating and resting castor seeds. K. G. Laiwala. J. Indian Inst. Sci., 1919, 2, 266–267.

THE results obtained by hydrolysing various oils by means of crushed meal from resting and germinating castor seeds showed that, on the whole, the former seeds possess greater lipolytic activity.

—C. A. M.

Castor and soya beans; Lipolytic activity of —. A. W. Barton. J. Amer. Chem. Soc., 1920, 42, 620–632.

FROM a study of the hydrolysis of lard, olive oil, and ethyl butyrate by the lipases of castor and soya beans it is deduced that the lipase of castor beans is more intense in its action than that of soya beans. The range of acidity in which action on lard and olive oil took place was practically the same with both enzymes and independent of the substrate. The degree of activity for a given acidity was practically the same for olive oil and for lard. The activity towards ethyl butyrate was less, and the range of acidity in which action took place was

much higher and narrower. It is concluded that soya and castor beans contain the same lipase or lipases, and that both contain more than one lipolytic enzyme.—J. K.

Chrysalis oil; Characters of — L. Desvergues. Ann. Chim. Analyt., 1920, 2, 80—82.

Two samples of silkworm chrysalis oil obtained from an oil works in the Rhône valley differed widely, one being a dark-brown substance resembling vaseline, and the other a pale limpid oil. The latter had the following characters: Sp. gr. at 15° C., 0.915; solidif. pt., -10° C.; n_D^{20} = 1.4665; acid value, 8.5; saponif. value, 186.1; iodine value, 83.9; unsaponifiable matter 1.3%; mol. equiv. of fatty acids, 268.6; m.pt. of fatty acids, 26.0° C.; m.pt. of saturated fatty acids, 50.0° C.; and iodine value of unsaturated fatty acids, 95.1. The semi-solid product had: Sp. gr. at 15° C. 0.961; solidif. pt., 23.5° C.; acid value, 113.0; saponif. value, 184.8; iodine value, 86.7; unsaponif. matter, 4.8%; mol. equiv. of fatty acids, 263.2; m. pt. of fatty acids, 33.3° C.; m.pt. of saturated fatty acids, 50.0° C.; and iodine value of unsaturated fatty acids, 139.6. The unsaponifiable matter from the oil contained only a small quantity of cholesterol, whereas that from the semi-solid product contained a considerable quantity. The different results given by the two samples are to be attributed to differences in the methods of extraction.—C. A. M.

Fats and oils; Determination of the iodine values of — by Winkler's bromate method. J. V. Lakhani and J. J. Sudborough. J. Indian Inst. Sci., 1916, 1, 173—179.

WINKLER's method (this J., 1914, 872) gives trustworthy results with ordinary oils and fats, provided that the addition takes place in the absence of light. In presence of diffused daylight substitution of bromine takes place to an extent which increases with the duration of contact. Correct iodine values are obtained with acids such as crotonic and cinnamic acids with α -ethylene linkages within 30 mins. if light is excluded, but the values obtained with the esters of such acids are much too low and not concordant. The use of finely powdered bone charcoal caused a considerable increase in the rate of the absorption of bromine, the iodine value of ethyl cinnamate being raised thereby from about 18.6 to 121, and that of ethyl crotonate from 24.5 to 170.3.—C. A. M.

Phloroglucinol reagent; Method of preparing — for the Kreis [rancidity] test. H. W. Dixon. J. Ind. Eng. Chem., 1920, 12, 174.

ONE HUNDRED grms. of sodium hydroxide is dissolved in 40 c.c. of water and cooled to a temperature at which a few crystals of resorcinol dropped into the liquid do not become brown; 15 grms. of resorcinol is then stirred into the liquid, the vessel is covered to exclude air and the mixture heated on a sand-bath for 2—3 hrs. until a dark chocolate-coloured melt is obtained. After cooling the mass is dissolved in 500 c.c. of water, the solution cooled in ice, acidified with hydrochloric acid, and extracted with successive 100 c.c. portions of ether. The united ethereal extracts are treated with animal charcoal and filtered, the filtrate forming a stock solution for the Kreis test (this J., 1904, 1001; Kerr, this J., 1918, 475 A).—W. P. S.

Emulsions. T. R. Briggs. J. Phys. Chem., 1920, 24, 120—126.

INTERMITTENT shaking is much more efficient for preparing emulsions than continuous shaking. The time required for complete emulsification depends on the interval of rest between the individual shakes. Emulsions are readily made by rolling or rotating cylinders containing oil and soap solution. (Cf. J.C.S., May.)—J. F. S.

Emulsions. T. R. Briggs, F. R. Du Cassé, and L. H. Clark. J. Phys. Chem., 1920, 24, 147—160.

EMULSIONS of oil and water may be made by grinding the two together in a mortar in the presence of a finely divided solid which is easily wetted. The finely divided solid serves to increase the area of the interface between oil and water and is more effective if suspended in the oil before adding the water. In the "Continental" method of preparing emulsions gum arabic acts both as finely divided solid and as emulsifying colloid. Emulsions of water in oil may be made in a mortar by using finely divided solids easily wetted by oil. (Cf. J.C.S., May.)—J. F. S.

Proso millet oil. Dunbar and Binnewies. See XIXA.

PATENTS.

Oils and fats; Process for recovering — from soap-free waste water. Naaml. Vennoots. A. Jurgens' Vereenigde Fabrieken, Oss, Holland. Eng. Pat. 124,220, 12.3.19. (Appl. 6142/19.) Int. Conv., 9.3.18.

THE water is treated with small quantities of an electrolyte, e.g., 0.01% to 0.02% of sodium chloride, alum, or sulphuric acid, to break down the emulsion.—A. de W.

Washing and degreasing plant; [Beater-arms for] benzine — W. Irwin, Copenhall. Eng. Pat. 140,191, 4.3.19. (Appl. 5327/19.)

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

White lead; Modification of the Thompson method for the determination of acetic acid in — L. McMaster and A. E. Goldstein. J. Ind. Eng. Chem., 1920, 12, 170—171.

THE Thompson method (this J., 1905, 487) may be shortened by distilling with phosphoric acid as described until the mixture has a small bulk and then continuing the distillation with steam until about 600 c.c. of distillate is obtained. This is treated with 0.5 c.c. of phosphoric acid and distilled under a pressure of about 150 mm.; during the distillation air is admitted through a fine capillary reaching into the liquid. When 10 c.c. of the distillate requires no more than one drop of $N/10$ sodium hydroxide for neutralisation the distillation is stopped and the total distillate titrated. In the case of white lead which has been ground with oil it is almost impossible to extract the whole of the oil, and the distillate obtained in the Thompson method contains free fatty acids; the second distillation under reduced pressure yields a distillate free from fatty acids, and consequently the results obtained for acetic acid are lower than those found by the Thompson method.—W. P. S.

Lead chromates. L. Bock. Farben-Zeit., 1920, 25, 761.

THE lead chromates show many examples of change of colour with alteration of dispersity, e.g., many products of tones varying from orange to red can be brought to the same tone by grinding, whilst pale pigments can be obtained from the normal chromate, after washing, by continued standing under water. The sulphur or canary tones of the chrome yellows obtained by simultaneous precipitation of the chromate and sulphate of lead depend on their high degree of dispersity. The pigments obtained by addition of highly dispersed lead sulphate to or even precipitation of lead sulphate on to ordinary chrome yellow are not appreciably lighter in tone than the ordinary pigment, whilst their purity and opacity are inferior, simulating that of "reduced" chrome yellows.—A. de W.

Pigments; Fineness and texture of —. H. A. Gardner. Circ. 90, Paint Manufacturers' Assoc., U.S.A., and Nat. Varnish Manufacturers' Assoc. Mar., 1920. 6 pp.

THE pigment under examination is rubbed with the finger across the grooves of an Edison disc phonograph record, and the surface is then examined with the microscope. Most pigments consist of coarse particles covered with fine ones, and when examined by the method described the coarser particles are retained in the grooves with their facets arranged parallel to the plane of the record. The microscopic observation of pigments mounted on a record is best done by viewing obliquely. The number of particles in a given weight of pigment may be estimated by preparing a suspension of 1 grm. of the pigment in a litre of water containing a very little gelatin, and counting the particles in a few drops of the suspension contained in the cell of a haemocytometer.—A. de W.

Carbon black in natural gas; Apparatus for estimating —. R. O. Neal. Chem. and Met. Eng. 1920, 22, 358—359.

AN apparatus for determining the quantity of carbon black obtainable from gases of unknown composition comprises a central portion, hood, and hopper, of sheet iron, the hopper having a sliding door to enable the carbon black to be removed from a depositing plate by means of a scraper. Below the depositing plate, at an adjustable distance, is fixed the gas pipe with lava tip burners, each capable of burning 4 cb. ft. of gas per hr., and the gas supply is connected to a meter and is controlled by a needle-valve, a U-tube for determining the pressure being inserted between the meter and the burners. The apparatus may also be used without a meter by previously calibrating it against a meter and observing different pressures on the manometer tube and recording the time. The test should last at least one hour, using 15—20 cb. ft. of gas. The carbon black should be removed at intervals of 10 mins., and at the end of the test it is removed by a brush and the total weighed.—W. J. W.

Oleo-resin from Hardwickia pinnata. K. S. Iyer and J. J. Sudborough. J. Ind. Inst. Sci., 1918, 2, 29—35.

THE oleo-resin obtained from *Hardwickia pinnata* is a dark-brown viscid liquid soluble in alcohol, ether, light petroleum, acetic acid, and turpentine. It closely resembles copaiba balsam, as the following constants show: Sp. gr. at 15° C., 1.008; acid value, 90.2; saponif. value, 112.9; acetyl value, 79.6; volatile oil, 42.7%. The volatile oil, separated by distillation with superheated steam, is colourless, and has a pungent, bitter taste. It boils mainly between 248° and 253° C., and its physical constants closely resemble those of caryophyllene, the nitroschloride of which was actually isolated from the oil and identified. The residual resin is brittle, is completely soluble in 90% alcohol, and yields a good quality spirit or oil varnish.—G. F. M.

Oleo-resin from Dipterocarpus indicus. J. C. Mansukhani and J. J. Sudborough. J. Ind. Inst. Sci., 1918, 2, 37—45.

THE oleo-resin of *Dipterocarpus indicus* (Dhuma tree) is intermediate between the oleo-resin of *Hardwickia pinnata* (preceding abstract), and that of other *Dipterocarpus* sp. which yield the so-called garjan (gurjun) balsam. The following constants are given for the oleo-resin: Acid value, 12.6; saponif. value, 16.3; acetyl value, 48.2, total volatile oil, 67.8%. The volatile oil, isolated by steam distillation, boiled mainly at 250°—254° C. The nitroschloride was obtained from the oil in somewhat greater yield (10—11%) than that obtained from the *Hardwickia* oil, and was identified as

α -caryophyllene nitroschloride. The residual resin is harder than the *Hardwickia* resin, and gives both spirit and oil varnishes of good quality. The volatile oil apparently possesses therapeutic properties similar to copaiba oil.—G. F. M.

Varnishes; Solvent media and their effect on [synthetic resin] —. H. Wolff. Farben-Zeit., 1920, 25, 668—671.

THE solubility of synthetic resins (formolites) of different makes and even of different deliveries of the same make varies very considerably, especially in mixtures containing benzene and its homologues. This is shown by actual determinations of the benzene-carrying power of different specimens when dissolved in various solvents, e.g., ethyl and methyl acetates, methyl formate, and acetone. The viscosity curves of solutions of synthetic resins in acetone-benzene mixtures show that with increasing proportion of benzene to acetone there is a change from molecular to colloidal dispersed systems. In some cases precipitation or even gel-formation results from increasing addition of benzene. The persistent softness of the films left when some formolite varnishes are allowed to dry spontaneously is attributable to the selective evaporation of the solvent in the film, the solvent of higher boiling-point and weaker solvent power being retained to a proportionately greater degree as evaporation proceeds, so that a surface layer of relatively impermeable gel of the resin in the indifferent solvent is formed, which inhibits further evaporation in the under layers. The solvents chosen for use in formolite varnishes should therefore be of such nature as to permit of a large addition of the poorer of the solvents without precipitation of the resin, or the more active solvent should possess the lowest rate of evaporation in the mixture. Alternatively a component, e.g., pyridine, may be added which diminishes the selective evaporation of the active and non-active solvents.—A. de W.

PATENTS.

Linoleum; Producing — and product thereof. G. W. Priest, Assignor to E. I. du Pont de Nemours & Co., Wilmington, Del. U.S. Pats. (A) 1,334,049 and (B) 1,334,050, 16.3.20. Appl., 8.3.17.

(A) AN oil or (B) an oxidised oil is mixed with a polymerised product of the coumarone or indene group at a temperature not above 100° C., and the mixture is applied to a fabric layer.—A. de W.

Plastic rubber-like masses; Manufacture of —. P. B. Ribot, Schwabach. Ger. Pat. 315,847, 11.7.18.

THE liquor which settles out in the saponification of resins is purified by sedimentation or filtration, then treated in a stirring vessel with dilute sulphuric or hydrochloric acid in a finely-divided state, while heating at about 100° C. The resulting mass is slightly acid, insoluble in water, soluble in alcohol, ether, and caustic alkali; it is plastic when warm and brittle in the cold.—J. F. B.

[Resinous] polymerised compounds from unsaturated hydrocarbons; Production of —. H. V. Dunham, Brattleboro, Vermont, U.S.A. Eng. Pat. 138,046, 8.9.19. (Appl. 22,095/19.)

SEE U.S. Pat. 1,324,649 of 1919; this J., 1920, 164 A. Reference is directed to Eng. Pats. 7282 of 1914 and 109,077; this J., 1915, 346; 1917, 1089.)

Paint; Waterproof —. S. Kuroki, Tokyo, Japan. U.S. Pat. 1,333,321, 9.3.20. Appl., 6.5.19.

SEE Eng. Pat. 135,132 of 1919; this J., 1920, 35 A.

Coumarone-resin insulator. U.S. Pat. 1,332,860. See XI.

XIV.—INDIA-RUBBER; GUTTA-PRECHA.

Rubber goods; Determination of cellulose in —. S. W. Epstein and R. L. Moore. *Indiarubber J.*, 1920, 59, 559—566.

ONE half gram of the rubber is digested with 25 c.c. of freshly distilled cresol (b. pt. 195° C.) for 4 hours at 160°—185° C., then cooled and 200 c.c. of light petroleum spirit (b. pt. 45°—50° C.) added with constant agitation. After allowing to settle and decanting the liquid through a Gooch crucible containing a pad of acid-treated, ignited asbestos, the residue is washed thrice with petroleum spirit, five times with hot benzene, and once or twice with acetone; it is then treated with hot hydrochloric acid (10%), transferred completely to the crucible, washed 10 times with hydrochloric acid, then with water until free from chlorides, and treated with acetone and subsequently with a mixture of acetone and carbon bisulphide (1:1) until the extracts are colourless. The residue is washed with alcohol, dried for 90 min. at 105° C., transferred to a weighing bottle and weighed. The extracted material is digested in a beaker with 15 c.c. of acetic anhydride and 0.5 c.c. of sulphuric acid for 30 min. at 75° C., cooled, treated with 25 c.c. of 90% acetic acid, filtered slowly through a pad of treated asbestos in a Gooch crucible, washed repeatedly with hot 90% acetic acid and then 5 times with acetone. The crucible is placed in a weighing bottle, dried for 2 hours at 150° C., and then weighed. The loss in weight on acetylation represents the cellulose. The presence of leather does not interfere with the method, but in such case it is desirable to digest with cresol at 120° C. for 16 hours. The acetylation process indicates 95% of any wood present, 90% of any jute, 21% of cork, and 70% of any leather.

—D. F. T.

PATENTS.

[*Rubber*] *latex; Process for treating — and product obtained thereby. Rubber composition and process for the production thereof.* E. M. Slocum, Medan, Sumatra, Assignor to General Rubber Co. U.S. Pats. (A) 1,332,925 and (B) 1,332,926, 9.3.20. Appl., 30.1 and 23.2.17.

(A) RUBBER-PRODUCING and similar latex is coagulated under conditions restraining expansion, so that the latex is subjected to a gradually increasing pressure during its coagulation. (B) A vulcanisable plastic material is mixed with rubber seed pulp.

—D. F. T.

Rubber product and composition. Plastic composition. J. M. Weiss, New York, Assignor to The Barrett Co. U.S. Pats. (A) 1,334,060 and (B) 1,334,061, 16.3.20. Appl., 3.7.19.

(A) A COMPOSITION is prepared containing, dissolved in a common solvent, more than 50% of rubber and 10—30% of a semi-solid to solid bitumen obtainable by the destructive distillation of coal-tar pitch. (B) A plastic composition contains a semi-solid to solid bitumen derivable from coal tar by destructive distillation, together with other material not liquefiable by heat.—D. F. T.

Rubber-like mass. Ger. Pat. 315,847. See XIII.

Leather substitute. U.S. Pat. 1,332,320. See XV.

XV.—LEATHER; BONE; HORN; GLUE.

Molybdenum value [of tannins]; Simplified method for the determination of the —. R. Lauffmann. *Ledertechn. Rundsch.*, 1919, 11, 93—91. *Chem. Zentr.*, 1920, 91, II., 220. (See also this J., 1913, 153.)

TEN c.c. of filtered tannin solution of about four

times the usual strength for analysis is mixed with 10 c.c. of a reagent composed of equal vols. of 10% ammonium molybdate and 15% ammonium chloride solutions and filtered. Ten c.c. of the filtrate is evaporated to dryness and dried in the water oven to constant weight. At the same time, 10 c.c. of the tannin solution and 10 c.c. of the above reagent are evaporated together in a basin and dried to constant weight. The weight found by the evaporation of the filtrate is doubled and the product is subtracted from the dry weight of the unfiltered mixture, the difference giving the weight of the precipitate. This is calculated as a percentage of the total dissolved solids of the tannin extract. The following molybdenum values are given: Oak bark extract 65.5—81.3; oak wood extract 112.0—152.1; spruce bark extract 23.6—36.7; chestnut extract 148.2—191.3; quebracho extract, solid, not sulphited 2.0—33.8; the same, fluid, sulphited 0.0—23.4; mangrove extract 117.9; mimosa bark extract 67.9.—J. F. B.

PATENTS.

Leather; Manufacture of —. W. C. Blatz, Wilmington, Del., Assignor to J. B. Blatz, Philadelphia, Pa. U.S. Pat. 1,334,216, 16.3.20. Appl., 5.2.17. Renewed 24.4.19.

ALUM-CHROME tanned skins or hides are impregnated with an insoluble barium salt to produce glazed white washable leather.—D. W.

Substitute for leather, and similar purposes; Composition of matter for use as a —. G. Christenson, Jamaica, N.Y., Assignor to H. W. Johns-Manville Co. U.S. Pat. 1,332,320, 2.3.20. Appl., 27.3.18.

RUBBER, finely-ground sponge, and a considerable proportion of an inert filler are intimately mixed together to form a dense, non-elastic composition. —D. W.

Leather substitute; Manufacture of a —. D. Waller, Hamburg. Ger. Pat. 315,434, 15.1.18. Addition to 300,952.

INSTEAD of zinc chloride, calcium chloride, magnesium chloride or other hygroscopic salts are employed. Millboard is dipped into a 30% solution of the salt, then dried and treated with a warm fatty oil and finally with a solution of rosin.—J. F. B.

Horny masses from casein; Manufacture of —. R. Weiss, Hamburg. Eng. Pat. 115,439, 3.5.18. (Appl. 7479/18.) Int. Conv., 28.4.17.

SEE Ger. Pat. 313,881 of 1917; this J., 1920, 36 A.

XVI.—SOILS; FERTILISERS.

Lime; Absorption of — by soils. F. J. Warth and M. P. Saw. *Mem. Dep. Agric. India*, 1919, 5, 157—172.

EXPERIMENTS on the absorption by different types of soil of calcium carbonate from solutions of calcium bicarbonate of different concentrations in the presence of varying amounts of carbon dioxide showed that carbon dioxide plays an important part in the equilibrium attained. A new method of determining the lime requirement of a soil may be based on the experimental results, but is too difficult and involved for ordinary laboratory work. —W. G.

Soil; Retention of certain acidic to basic constituents of the — affected by ammonium sulphate and sodium nitrate. L. P. Howard. *Soil Sci.*, 1919, 8, 313—321.

THE "acidity" in a soil caused by long continued use of ammonium sulphate is a result of a change

in the ratio of acids to bases. Iron and aluminium take the places normally occupied by the stronger bases, calcium and magnesium, and the salts of the weaker bases undergo hydrolysis and give rise to a definite hydrogen ion concentration in the soil solution. (*Cf.* J. C. S., i., 416).—W. G.

Potassium-bearing minerals as a source of potassium for plant growth. E. de Turk. Soil Sci., 1919, 8, 269—301.

TRIALS were made with orthoclase, microcline, leucite, alunite, and lepidolite on a very loose peaty soil quite free from sand and from roots or other residues of recently grown plants. In the pot experiments, where crops were grown, the minerals were added at a rate equivalent to 2 tons per acre, and one ton of ground limestone per acre was also added. Lepidolite was detrimental to the growth of buckwheat under these conditions, whilst the other finely-ground minerals gave an increase in yield of 21—35%. The additional application of organic manures or soluble sodium or magnesium salts did not increase the yield of crop or the availability of the potash in the minerals used. Except in the case of lepidolite, the solubility of the minerals used was low. This high solubility of lepidolite probably accounts for its effect in stunting the crop, some toxic element, such as lithium, possibly being dissolved from the mineral. The solubility of all the minerals was increased by the presence of ammonium chloride or of decomposing alfalfa (lucerne). The low availability of the potash in dune sand is due to the fact that most of it is contained in the larger particles, which offer a relatively small surface to the solvent action of the water. The increase in solubility produced by grinding such sands is not sufficient to be of practical value.—W. G.

Iron-aluminium phosphates; Utilisation of —. G. H. Stanley. J. S. Afr. Assoc. Anal. Chem., 1920, 3, 13—21.

SAMPLES of iron-aluminium phosphate from a large deposit at Saldanha Bay, S. Africa, had the following composition:— P_2O_5 , 5.68—32.68; Fe_2O_3 , 2.60—11.96; Al_2O_3 , 2.22—17.29; and SiO_2 and insoluble matter, 15.67—77.05%. The material is therefore of less value than Redonda phosphate. By heating the Saldanha phosphate rock with limestone and small coal in an iron vessel with perforated false bottom, whilst blowing air through the heated mass, it could be converted into a material resembling a low-grade basic slag which could be produced and sold at about 7s. per unit of P_2O_5 . By this process about 75% of the phosphoric anhydride is converted into a citric-soluble form.

—C. A. M.

Plants: Influence of organic compounds on the development of —. IV. G. Ciamician and C. Ravenna. Atti R. Accad. Lincei, 1920, 29, i., 7—13. (Compare this J., 1919, 297 A.)

FURTHER experiments have been carried out with compounds belonging to a number of different chemical classes. (*Cf.* J. C. S., i., 408).—T. H. P.

Nitrogen [in fertilisers]; Use of potassium permanganate in determining — by the Kjeldahl method. W. Frear, W. Thomas, and H. D. Edmiston. J. Assoc. Off. Agric. Chem., 1919, 3, 220—224.

THE addition of potassium permanganate at the end of the acid digestion period caused a considerable loss of nitrogen in the case of a wide range of fertilisers. The loss depended somewhat on the amount of permanganate added, but chiefly on the time of addition. If the addition was delayed for 2 mins. after the flame had been removed there was no loss.—W. P. S.

PATENTS.

Potassium compounds; Process for extracting — from rocks containing potassium and simultaneously producing nitrogen-containing fertilisers. C. Rossi, Legnana, Italy. Eng. Pat. 130,963, 12.5.19. (Appl. 11,913/19.) Int. Conv., 9.8.18.

FINELY-GROUND potash-bearing rocks such as leucite, volcanic ash, felspar, orthoclase, granite, etc., are mixed with calcium carbide and heated in a stream of nitrogen. Calcium cyanamide is formed together with a complex nitrogenous fertiliser produced by the interaction of the free carbon and the lime present and their combination with the nitrogen, whilst the potassium compounds are volatilised and may be completely recovered. The addition of coal to the rocks increases their absorption of nitrogen. A complex fertiliser, consisting of a mixture of potassium compounds with the nitrogenous product, may be obtained by keeping the temperature of reaction below the volatilising temperature of the potassium compounds.—W. J. W.

Fertilisers containing nitrogen and phosphoric acid. Norsk Hydro-Elektrisk Kvaelfabrikations-selskab, Christiania. Eng. Pat. 132,496, 25.7.19. (Appl. 18,536/19.) Int. Conv., 13.9.18.

A MIXTURE of an insoluble phosphate and a urea salt, e.g., urea nitrate, is either employed direct as a fertiliser, in which case the moisture in the soil causes reaction between the two compounds with formation of soluble phosphates, or the reaction is first effected by moistening the mixture, and the product is dried for subsequent use as a fertiliser. (Reference is directed to Eng. Pat. 17,507 of 1902; this J., 1902, 1462).—L. A. C.

XVII.—SUGARS; STARCHES; GUMS.

Dextrose; Detection and characterisation of — in plants by a new biochemical method. E. Bourquelot and M. Bridel. Comptes rend., 1920, 170, 631—635.

THE dry plant material is extracted with boiling 80% alcohol, the solution is evaporated to dryness, resinous materials are removed by extraction with ethyl acetate, and the residue is dissolved in a little water and the solution evaporated to dryness under reduced pressure. The residue is dissolved in 70% methyl alcohol, and the reducing power and rotation of the solution are measured. Emulsin is added and the solution is left to stand until no further change in its rotation occurs. The reducing power and rotation are again measured, and from their changes the amount of dextrose present may be calculated; emulsin converts 82.6% of the dextrose present in 70% methyl alcohol into β -methylglucoside even in the presence of other sugars.—W. G.

Dextrose; Determination of — in the presence of lactose. E. Hildt. Ann. Chim. Analyt., 1920, 2, 78—80.

LACTOSE may be quantitatively hydrolysed by means of Twitchell's sulpho-aromatic compounds (see this J., 1919, 51 A). A suitable catalyst is prepared by dissolving 180 grms. of sodium benzenesulphonate, free from iron, in water containing 49 grms. of pure sulphuric acid, and diluting the solution to a litre. A quantity of a solution (e.g., defecated urine), containing not more than 1 gm. of sugar, is mixed with 20 c.c. of the Twitchell reagent, made up to 100 c.c., shaken, and filtered, and 50 c.c. of the filtrate hydrolysed for 6 hours at 95°—98° C. The remainder of the solution is used for the titration of reducing sugars by means of Fehling's solution diluted with 4 vols. of water, and treated with 5 or 6 drops of sodium hydroxide solution. The

hydrolysed portion, after cooling and dilution to 50 c.c., is titrated in the same way. If n and n' be the number of c.c. required to decolorise 10 c.c. of Fehling's solution before and after hydrolysis respectively, the amounts of hydrated lactose (x) and anhydrous dextrose (y) in grms. per 100 c.c. are given by the equations: $x = 3.55/n' - 3.55/n$, and $y = 1.458/n' + 3.34/n$.—C. A. M.

Glucosides. Karrer. See XX.

PATENTS.

Caramel; Process for producing —. A. Daniel, Charlottenburg, Germany. Eng. Pat. 137,339, 13.11.17. (Appl. 16,678/17.)

CARBOHYDRATES such as starch, sugars, molasses, etc., are heated with (glacial) acetic acid and dehydrating agents such as acetic anhydride, sodium acetate, alkalis, or mineral acids, under atmospheric or higher pressures, or with sodium acetate alone under atmospheric pressure, the reaction itself in this case liberating a certain quantity of acetic acid. The caramel produced yields clear solutions in water and is suitable for spirituous liquors.—J. H. L.

Saccharifying plant material. Ger. Pat. 305,690. See XVIII.

Fodder from wood. Ger. Pat. 305,090. See XIXA.

XVIII.—FERMENTATION INDUSTRIES.

Malting and brewing. P. Petit. Brasserie et Malterie, 1919, 9, 241—245, 257—261.

By restricting germination the malting loss is reduced, but the short-grown malt obtained is imperfectly modified in respect of proteins, which remain to a large extent insoluble. They can be partially dissolved by pre-mashing, but are liable to precipitation by a slight change of reaction of the wort. The conversion of these imperfectly dissolved proteins into completely soluble degradation products by "protein rest" is incomplete, because the short-grown malt is deficient in proteolytic power. Hence the increased yield of extract attained by means of pre-mashing and the "protein rest" consists largely of proteins in a state of unstable solution. Their contribution to flavour and head-formation is of doubtful value, and they are certainly liable to cause difficulties in clarification and protein haze in the finished beer.—J. H. L.

Alcoholic fermentation; Chemistry of —. C. Neuberg and E. Reinfurth. Ber., 1920, 53, 462—469.

In reply to Zerner (this J., 1920, 276 A) it is pointed out that the impossibility of effecting a quantitative conversion of sugar into glycerol and acetaldehyde by fermentation in the presence of sodium sulphite is to be attributed to the gradually increasing alkalinity of the solution and not to the slowness of reaction between aldehyde and sulphite and, further, that Zerner's conclusion that the formation of pyruvic acid cannot be the preliminary step in the production of acetaldehyde is based on faulty experiment. (Cf. J. C. S., i., 406).—H. W.

Pyruvic acid; Biochemical production of —. A. Fernbach and M. Schoen. Comptes rend., 1920, 170, 764—766.

FURTHER evidence is given that in a mineral nutrient solution pyruvic acid is formed during alcoholic fermentation by yeast, providing the medium is kept neutral by the presence of an excess of calcium carbonate.—W. G.

Wine; Bacterial decomposition of tartaric acid and glycerol in —. H. Müller-Thurgau and A. Osterwalder. Landw. Jahrb. der Schweiz, 1919. Chem. Zentr., 1920, 91, II., 90—91.

A NUMBER of Swiss red wines developed, some weeks or months after the decomposition of the malic acid was complete, maladies due to bacterial decomposition of tartaric acid and glycerol in some cases, and of glycerol alone in others. These maladies were associated with evolution of carbon dioxide and increase of volatile acidity (acetic with a small proportion of propionic acid); very frequently the wines lost their red colour to some extent and became brownish, and in a few cases they acquired a bitter flavour. Access of air aggravated the trouble. In some cases the tartaric acid was eventually decomposed completely, but about 0.2—0.3 % of glycerol remained in all cases. From a wine in which both tartaric acid and glycerol underwent decomposition, two new organisms were isolated, viz., *Bact. tartarophthorum* and *B. tartarophthorum* var. α . Both decompose tartaric acid energetically, but whilst the former also decomposes glycerol the latter does so to only a slight extent. Acetic acid and carbon dioxide are produced from tartaric acid, and acetic, propionic, and lactic acids from glycerol. Both bacteria are facultative anaerobes and form non-motile and non-sporulating rods 0.8—1 μ thick. They decompose malic acid readily and produce mannitol from levulose. High acidity and high tannin-content have an inhibitive influence on the maladies described, whilst high temperatures promote them. Suitable preventive measures are storage at low temperatures, use of sulphur dioxide and metabisulphite, and pasteurisation; but in the case of wines similar to those investigated these measures should not be applied until after the decomposition of the malic acid is complete.—J. H. L.

Methyl alcohol in spirits; Detection of —. P. Hasse. Pharm. Zentr., 1920, 61, 177—182.

THE sample is distilled and 0.5 c.c. of the distillate (containing not more than 0.025 c.c. of alcohol) is mixed with 1 c.c. of 5 % potassium permanganate solution, 2.5 c.c. of dilute sulphuric acid (sulphuric acid 19 grms., water 200 c.c.) and, after standing 2 mins., the mixture is decolorised by the addition of 1 c.c. of 10 % oxalic acid solution. To 0.5 c.c. of this mixture are then added 1 drop of peptone solution (=2.5 mgrms. of peptone) and 1 c.c. of sulphuric acid containing iron (0.05 gm. of iron alum dissolved in 1 c.c. of water and added to 300 grms. of sulphuric acid). A deep blue colour is obtained if the spirit contains 1 μ of methyl alcohol; a red-blue colour is produced by 0.3 % of the alcohol. Pure ethyl alcohol gives a yellowish-red coloration with the test. If an indication of the presence of methyl alcohol is obtained, it should be confirmed by the morphine and magenta-sulphurous acid tests.—W. P. S.

Oralic acid. Bau. See XX.

PATENTS.

(A—C) *Yeast; Manufacture of pressed* —. (D—I) *Manufacture of yeast with little or no production of alcohol.* Verein der Spiritus-Fabrikanten in Deutschland, Berlin. Ger. Pats. (A) 300,663, 25.2.15; (B) 303,251, 16.3.15; (C) 303,252, 20.8.15; (D) 300,662, 17.3.15; (E) 303,221, 1.4.15; (F) 303,222, 20.3.15; (G) 303,253, 8.5.15; (H) 304,241, 13.4.15; and (I) 301,242, 16.4.15.

(A) YEAST is grown, by the ordinary aeration process, in solutions containing not more than 2 % of refined sugar or raw beet sugar, together with not less than 15 parts of mineral nutrient salts per 100 parts of sugar. The yeast produced has a normal character and colour. (B) The mineral acid liberated

during the fermentation is neutralised by addition of suitable alkaline mineral salts, *e.g.*, carbonates. When molasses is used the basic substances therein neutralise a part of the free acid. The process may be applied also to fermentations carried out in presence of mineral salts for other purposes than the production of yeast. (c) Sodium salts may be substituted for potassium salts in the nutrient liquid. The "soda yeast" obtained possesses a working power equal to that of a good bakers' yeast. (d) Yeast is pitched in highly diluted wort, and stronger wort is added slowly and continuously during the growth of the yeast, whereby, owing to the continuous consumption of nutrient substances, the concentration of the wort remains throughout such as to ensure the best and most rapid assimilation of the nutriment supplied. (e) Wort containing yeast is continuously drawn off from the lower part of the vat as fast as fresh nutrient solution is added. The yeast is separated by centrifugal means from the wort thus drawn off. (f) Sugar and mineral salts are employed for the preparation of the wort. The mineral salts may amount to half the weight of the sugar used. In using ammonium sulphate as source of nitrogen the acidity (free sulphuric acid) increases very rapidly, and is neutralised by means of alkalis or alkaline salts. (g) Antiseptics assimilable by yeast, *e.g.*, formaldehyde and formic acid, are added to the nutrient solution. The proportions required in the solution, which is added continuously to the vat (0.01–0.1% of formaldehyde and 0.03–0.3% of formic acid), are too small to injure the yeast. (h) In the process claimed in (e) so much yeast is pitched at the commencement as can be grown in the quantity of liquid then present. (i) In the process claimed in (f) a solution of free alkalis or alkaline carbonates is added continuously to the wort, to furnish mineral nutriment to the yeast, and a faintly alkaline reaction is maintained in the wort during yeast growth.—J. H. L.

Cellulose; Process for saccharifying plant material containing —. C. G. Schwalbe, Eberswalde. Ger. Pat. 305,690, 4.5.16.

THE moist material, containing less than 50% of water, is subjected to the action of a gaseous acid such as hydrochloric, hydrofluoric, or sulphurous acid, until it is swollen but not dissolved, the heat produced being eliminated by cooling, if necessary below the ordinary temperature. The gaseous acid is then removed and the gelatinous material is saccharified by dilute acid under atmospheric or higher pressure. From 100 parts of wood dry substance it is possible to obtain 70 parts of soluble matter containing nearly the theoretical yield of dextrose and pentoses.—J. H. L.

Caramel. Eng. Pat. 137,339. See XVII.

Alcohol in beverages. U.S. Pat. 1,333,905. See XXIII.

XIXA.—FOODS.

Milk; Action of colloids in the reductase test for —. E. Eichwald. Z. Unters. Nahr. Genussm., 1919, 38, 359–361.

THE colloids (proteins and fat) present in milk appear to have an influence on the Methylene Blue test for reductase in milk; the finer the state of division of the colloids the slower is the rate at which the colour is destroyed.—W. P. S.

Milk; Analysis of —. [Influence of formaldehyde, etc.] C. Porcher. Ann. Falsif. 1920, 13, 35–37.

THE addition of 1 drop of 40% formaldehyde solution to 50 c.c. of milk did not affect the determina-

tion of the lactose, but when 5 drops were added the amount of lactose found increased from 5.00 to 5.31%.—W. P. S.

Proso millet; analysis of its oil. A characteristic alcohol. B. A. Dunbar and E. R. Binnewies. J. Amer. Chem. Soc., 1920, 42, 658–666.

MEAL from the hulled grain of the proso millet (*Panicum miliacum*) contained 2.86% ash, 12.80% moisture, 6.25% crude fibre, 15.86% crude protein, 5.07% oil (ether extract), 57.16% nitrogen-free extractives, and 59.65% starch. The oil, which was extracted by petroleum spirit equally as well as by ether, when kept out of contact with the air gradually deposited thin, pearly-white plates of "prosol," $C_{44}H_{86}O_2$, m. pt. 279° C., soluble in most organic solvents, but sparingly in cold ether or alcohol. The oil separated from prosol had sp. gr. 0.9228 at 25° C.; $n_D^{20}=1.4745$; an acetyl value corresponding to 10.9% alcohols and hydroxy acids; Hübl iodine value, 92.3; Reichert-Meißl value, 2.5; glycerin content, 3.31%; free fatty acids equivalent to 11.9% oleic acid; phytosterol, 0.63%; and unsaponifiable matter, 2.52%. It showed 3.18% increase in weight after oxygenation by Livache's process, and is therefore a semi-drying oil, ranking with rapeseed oil. The soluble acids corresponded to 0.36% formic acid, a trace of acetic acid being present. Insoluble fatty acids amounted to 89.8% of the oil, 76.5% being unsaturated and consisting of about 72% palmitic acid and a residue of carnaubic and daturic acids. The saturated fatty acids contained 51.6% oleic acid, 20.4% linolic acid, and 23.7% isolinolic acid.—J. K.

Humin formed by the acid hydrolysis of proteins; Origin of the —. IV. Hydrolysis in the presence of aldehydes. III. Comparative hydrolysis of fibrin and gelatin in the presence of various aldehydes. G. E. Holm and R. A. Gortner. J. Amer. Chem. Soc., 1920, 42, 632–640.

THE results of experiments on the hydrolysis of fibrin and gelatin in presence of benzaldehyde, butyraldehyde, isobutyraldehyde, acetaldehyde, and formaldehyde respectively confirm the previous conclusion (this J., 1918, 18 A) that in a normal protein hydrolysis the formation of black acid-insoluble humin is dependent upon the presence of tryptophane in the protein molecule.—J. K.

Arginine nitrogen. Holm. See XXIII.

PATENTS.

Cheese; Process of making Emmenthal or Swiss —. C. F. Doane, Eureka, Cal. U.S. Pat. 1,334,693, 23.3.20. Appl., 23.5.19.

MILK is heated to 134°–155° F. (57°–68° C.), then cooled, and rennet added to form a curd. The curd is heated to give the necessary firmness and to expel moisture or whey.—W. F. F.

Fodder from wood; Process and apparatus for producing —. H. Mühlenbein, Cöthen. Ger. Pat. 305,090, 18.11.16.

THE wood passes continuously through a series of vessels in which it is comminuted, then subjected to known chemical treatment with alkali and with oxidising agents such as chlorine, and then separated from the liquid, mixed with other feeding-stuffs, dried and, if necessary, ground. The digesting apparatus consists of two or more vessels, capable of being heated, and fitted with stirring mechanism, perforated feed, inlet and screened outlet for the chemical agents, and a steam outlet hood. The process can be applied also to the preparation of cellulose for paper-making, and the digesting apparatus may be used in place of a diffusion battery in producing sugar juice and fodder slices from beets, and in similar cases.—J. H. L.

Fodders; Process for the better utilisation of the nutritive value of inferior —. Kriegsausschuss für Ersatzfutter G.m.b.H., Berlin. Ger. Pat. 306,464, 18.1.16.

STRAW, hay, grasses, twigs, wood waste, etc., in a comminuted or ground state are subjected to the action of a solution of calcium saccharate at the ordinary temperature, or below or above 100° C., under atmospheric or higher pressures, and dried. —J. H. L.

Sugar beets; Process for producing a fodder and foodstuff from —, especially for use as adjunct in bread-making and the like. F. von Langen, Elsdorf. Ger. Pat. 310,028, 22.7.16.

BEETS are sliced or pulped and extracted by known methods. The residue is dried to a moisture-content of less than 10%, and the juice, without any purification, is evaporated under reduced pressure to a massecuite of 80–95% dry substance. The dried beet residue, in a finely granular or mealy form, is mixed with the hot massecuite so as to obtain a loose dry product containing at least the same proportion of sugar to other dry substance as occurs in beets. —J. H. L.

Cooling foods, beverages, and the like; Means for —. A. L. Price, Chatswood, N.S.W. Eng. Pat. 139,895. (Appls. 6550, 17.3.19, and 20,473, 20.8.19.)

XIXB.—WATER PURIFICATION; SANITATION.

Sewage; Purification of — by activated sludges. R. Cambier. Comptes rend., 1920, 170, 681–681.

THERE is very rapid disappearance of ammoniacal nitrogen during the first hour from sewage in contact with activated sludge at the ordinary temperature without a corresponding increase of nitric nitrogen, which only shows a marked increase after the first hour. Temperature has a marked effect on the purification. At 0° C. purification is complete, but nitrification is retarded; maximum activity is reached at 20°–25° C. At 37° C. there is no formation of nitric nitrogen, and the sewage is not modified in appearance. Nitrous nitrogen is only found in appreciable quantities at temperatures above 30° C. Both the ammonia and colloidal matter of the sewage are for the most part absorbed by the sludge at the moment of contact. —W. G.

Activated [sewage] sludge; Formation of —. F. Diénert. Comptes rend., 1920, 170, 762–763.

IN the preparation of activated sludge air is bubbled for a certain time through sewage, which is then allowed to settle, the water drawn off, fresh sewage added, and the air again bubbled through, this being repeated daily until, during a period of bubbling of air for five consecutive hours 20 mgrms. of ammoniacal nitrogen per litre of sewage is converted into nitrous or nitric nitrogen. This period of activation varies with the sewage or water used, the volume of air, and the temperature of the water. The change from inactivity of the organisms to activity is manifested quite suddenly. —W. G.

Air; Electrical purification of —. D'Arsonval, Bordas, and Touplain. Comptes rend., 1920, 170, 636–638.

By means of a high-tension electric discharge perpendicular to the direction of the current of air, the air may be purified from solid or liquid particles or bacteria. Using a copper tube at zero potential and an axial wire at a negative potential of 25,000 to 30,000 volts, the air passing at the rate of 127 cb. m. per hr., the issuing air was absolutely sterile, and 95–97% of the solid particles was

removed. Sterilisation is probably effected by ozone produced by the high-tension discharge.

—W. G.

Hypochlorous acid; Strength of — in solution, with particular reference to *eusol*. J. McCrae. Pharm. J., 1920, 191, 273.

IN Hart's method of determining the strength of *eusol* (this J., 1920, 79A) an error is made in assuming that 2 atoms of available chlorine correspond to 1 mol. of calcium hypochlorite; actually half of the available chlorine is derived from the chloride residue. Hence the amounts of bleaching powder given by Hart must be doubled if a solution containing 0.45% of hypochlorous acid is to be obtained. —C. A. M.

Picric acid; Toxicity of —. F. Koelsch. Z. ges. Schiess- u. Sprengstoffw., 1920, 15, 63–65.

PICRIC acid, under the conditions obtaining in munition works, is a relatively harmless substance, and, with the exception of certain supersensitive persons, the workers are but little affected by it; acute or chronic poisoning was never observed by the author. In the case of workers who exhibit sensitiveness towards picric acid, the symptoms noticed are irritation of the eyes, nose, throat, and mucous membrane, cardialgia, etc., but these effects may be lessened by paying attention to personal cleanliness. In a few instances it may be necessary to remove a person from the picric acid plant to other work, but special hygienic regulations do not seem to be required. —W. P. S.

PATENTS.

Sterilising liquids; Apparatus for —. H. B. Rudd, Akron, Ohio, assignor to The Electropure Dairy Co., Chicago, Ill. U.S. Pat. 1,333,633, 16.3.20. Appl., 22.9.19.

AN upright receptacle is provided on opposite sides with electrodes which extend at an angle from the top to the bottom and form water-jacket chambers between themselves and the wall of the receptacle. The intake and discharge pipes of the apparatus communicate with each of the water-jacket chambers, and the discharge of liquid from the receptacle is controlled by thermodynamic valves operable at predetermined temperatures. —W. E. F. P.

Sewage and other effluents; [Mixing] apparatus for use in treatment of — [by the activated sludge process]. L. Whyte, Wilmslow, and H. Fothergill, Cheadle Hulme. Eng. Pat. 104,189, 28.2.19. (Appl. 5028/19.)

Water; Softening —. E. Edser and S. Tucker, London, Assignors to Minerals Separation North American Corporation, New York. U.S. Pat. 1,333,393, 9.3.20. Appl., 3.7.18.

SEE Eng. Pat. 118,668 of 1917; this J., 1918, 670 A.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Strychnine; Test for —. H. E. Buc. J. Assoc. Off. Agric. Chem., 1919, 3, 193.

TO 0.5 c.c. of the strychnine salt solution are added 1 grm. of amalgamated zinc and 0.5 c.c. of hydrochloric acid. After 15 mins. the liquid is decanted and treated with 0.02% potassium ferrieyanide solution, added drop by drop. A pink to rose-red coloration develops. Large amounts of some alkaloids and other organic substances interfere, but in their absence the test will detect 0.001 mgrm. of strychnine. —W. P. S.

Opium assay. D. B. Dott. Pharm. J., 1920, 194, 302.

A MODIFICATION of the British official process is

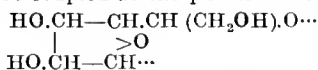
described which includes certain features taken from pharmacopœias and an improvement on the Japanese method of purifying the morphine-lime solution.—W. P. S.

Glucosides. V. *Synthesis of β-gaultherin, of methyl β-tetra-acetylglucosidoanthranilate, and of β-glucosidoresorcylic acid methyl ether.* P. Karrer and H. Weidmann. *Helv. Chim. Acta*, 1920, 3, 252—257.

By methylation of β-glucosidosalicylic acid with diazomethane, its methyl ester is obtained. Whilst structurally identical with natural gaultherin, it differs from the latter in that it is hydrolysed by emulsin, but not by gaultherase. Gaultherin is therefore the α-stereoisomeride. Mauthner's product (this J., 1913, 748 A) is identical with the tetra-acetyl derivative of the above ester, and is therefore not tetra-acetylgaultherin. (*Cf.* J.C.S., i., 395.)—J. K.

Glucosides. VI. *Configuration of glucosides.* P. Karrer. *Helv. Chim. Acta*, 1920, 3, 258—260.

PICTET has shown that levoglucosan is produced by the distillation of certain β-glucosides, but not from α-glucose (this J., 1918, 49 A; 1920, 40 A). A considerable amount is, however, obtainable from β-glucose, and the reaction is therefore conditional upon the presence of the β-configuration. Starch and cellulose, which answer to the reaction, probably contain β-glucosidic linkages, and it is possible that they contain the levoglucosan grouping. The structure of the trimethylglucose obtained by Denham and Woodhouse from cellulose (this J., 1917, 607) suggests that the glucose residues in cellulose are coupled at the points indicated:—



(*Cf.* J.C.S., i., 370.)—J. K.

Capsaicin, the pungent principle of capsicum; Constitution of —. II. E. K. Nelson. *J. Amer. Chem. Soc.*, 1920, 42, 597—599.

THE conception of capsaicin as vanillyldeceonoyl-amide (this J., 1919, 654 A; *cf.* Lapworth and Royle, this J., 1919, 843 A) is confirmed by the regeneration of capsaicin from vanillylamine and deconoyl chloride, the latter being obtained from the acid produced from capsaicin. (*Cf.* J.C.S., i., 380.)—J. K.

Peru balsam; Physical and chemical constants of —. E. C. Merrill. *J. Assoc. Off. Agric. Chem.*, 1919, 3, 194—197.

THE Hanus method is unreliable for determining the iodine value of the cinnamein separated from Peru balsam; the time of contact (30 mins.) usually employed is too short, at least 3 hrs. being required. There is no marked difference in viscosity between true and artificial Peru balsam. Cinnamein from artificial balsam has a slight optical rotation (+0.35° for 2 c.c. of cinnamein dissolved in 10 c.c. of benzene, and observed in a 100 mm. tube), and n_D^{25} less than 1.57, whilst cinnamein from genuine balsam is optically inactive, and n_D^{25} above 1.57.—W. P. S.

Truxillic acids. A. W. K. de Jong. *Proc. K. Akad. Wetensch.*, 1920, 22, 509—511.

THE isomeric truxillic acids may best be separated from cinnamic acid by sublimation at 130° C. in a current of air, and from one another first by the varying solubility of their calcium salts, and then through their barium salts. If the mixed truxillic acids are dissolved in just sufficient N/10-potassium hydroxide, and to the solution 1.5 grms. of anhydrous calcium chloride is added for each 10 c.c., the β-, δ-, and ε-acids are precipitated as

calcium salts. These are collected and converted into the barium salts, which may then be separated by their differing solubility in water. To the filtrate from the mixed calcium salts 8.5 grms. of calcium chloride is added for each 10 c.c., the calcium salt of β-cocaic acid being thereby precipitated. This is filtered off, and from the filtrate, by addition of hydrochloric acid and extraction with ether, α- and γ-truxillic acids are obtained. These may readily be separated, since the γ-acid is soluble in hot water, but the α-acid is not. (*Cf.* J.C.S., i., 389.)—W. G.

Pyrrole, indole, and carbazole; Metallic salts of —. E. C. Franklin. *J. Phys. Chem.*, 1920, 24, 81—99.

SODIUM, potassium, magnesium, and calcium salts of pyrrole, indole, and carbazole are formed by the action of the metals, and sodium, potassium, and silver salts by the action of the metal amide on the bases in liquid ammonia solution at low temperatures. The salts are all unstable, and hydrolysed vigorously by water. (*Cf.* J.C.S., i., 396.)—J. F. S.

Silver-salvarsan. A. Binz, H. Bauer, and A. Hallstein. *Ber.*, 1920, 53, 416—428.

ULTRA-FILTRATION experiments, absence of the Tyndall phenomenon, and the appearance under the ultra-microscope show that silver-salvarsan does not contain colloidal silver or silver oxide. In the formation of silver-salvarsan and its derivatives, two molecules of silver nitrate react with one molecule of the salvarsan compound, as is shown by the isolation of dichlorosalvarsan di-silver chloride $[\text{AgCl.NH}_2\text{.C}_6\text{H}_4\text{(OH).Cl.As}]_2$ in the solid state. Silver-salvarsan contains silver oxide in complex combination, but the exact mode of attachment of the metallic atoms to the salvarsan molecule remains undecided. (*Cf.* J.C.S., i., 401.)—H. W.

Oxalyl chloride and bromide; Use of — for producing acid chlorides, acid bromides, or acid anhydrides. III. R. Adams and L. H. Ulich. *J. Amer. Chem. Soc.*, 1920, 42, 599—611.

WHEN the method of preparing acid anhydrides from 1 mol. of oxalyl chloride and 2 mols. of acid (this J., 1918, 221 A) is applied to *m*- or *p*-nitro, 3,5-dinitro-, or 2,4,6-trinitrobenzoic acids, mixed anhydrides are produced, which are very stable towards excess of oxalyl chloride, but decompose on melting. Aliphatic acids give less favourable results than aromatic acids, some acid being unchanged and some acid chloride being formed in addition to the anhydride. Acid chlorides may be prepared smoothly and in good yield by boiling the anhydrides, including those of the above nitro-compounds, with 1.5—2.5 mols. of oxalyl chloride alone or in presence of benzene for two hours; except in the case of the nitrobenzoic acids, the anhydrides may be replaced by the acids themselves. A method of preparing anhydrides and chlorides which gives still better results and has not the above limitations, consists in the treatment of the sodium salts with 1 or 1.5 mols. respectively of oxalyl chloride. Oxalyl bromide behaves in a similar manner to the chloride, and gives better yields of acid bromides than phosphorus pentabromide. (*Cf.* J.C.S., i., 386.)—J. K.

Ethylene and sulphur chloride; Mechanism of the reaction between —. J. B. Conant, E. B. Hartshorn, and G. O. Richardson. *J. Amer. Chem. Soc.*, 1920, 42, 585—595.

THE formation of ββ'-dichlorodiethyl sulphide by the action of sulphur monochloride on ethylene at 40°—60° C. can be best represented by the equations: (1) $\text{S}_2\text{Cl}_2 \rightleftharpoons \text{S} + \text{SCl}_2$; (2) $\text{C}_2\text{H}_4 + \text{SCl}_2 \rightarrow \text{CH}_2\text{Cl.SCl}$; (3) $\text{C}_2\text{H}_4 + \text{CH}_2\text{ClCH}_2\text{SCl} \rightarrow (\text{CH}_2\text{ClCH}_2)_2\text{S}$. Reaction (3) becomes appreciable only when the

absorption of ethylene reaches about 30% of the total, but towards the end it predominates. An almost pure compound of formula $\text{CH}_2\text{Cl.CH}_2\text{Cl.SCl}$ can be obtained by shaking sulphur dichloride with excess of ethylene at 0°C . The product prepared in a similar manner from sulphur monochloride has similar properties, but contains small amounts of unchanged monochloride and dichlorodiethyl sulphide. When the crude dichlorodiethyl sulphide is distilled under reduced pressure, there is a residue, which, when freed from a small amount of elementary sulphur, corresponds approximately in composition to the formula $(\text{CH}_2\text{Cl.CH}_2)_2\text{S}_2$. Correspondingly, only about 50% of the theoretical amount of sulphur is produced in the reaction (it is not precipitated, but is in colloidal solution, from which it separates slowly at the ordinary temperature and rapidly at 100°C ., leaving the freezing point of the disulphide unchanged); in addition, the amount of ethylene absorbed is only 95% of that theoretically required for its complete conversion into the sulphide and only about 80% of the theoretical quantity of sulphide is actually produced. (*Cf.* J.C.S., i., 363.)—J. K.

Nitriles; New method of formation of — by catalysis. A. Mailhe. *Comptes rend.*, 1920, 170, 813–815.

Is the method previously described for the catalytic preparation of aliphatic or aromatic nitriles (this J., 1918, 74 A) by passing the vapours of the corresponding esters along with ammonia over alumina or thoria at 500°C ., the ammonia may be replaced by various primary amines.—W. G.

Oxalic acid; Determination of —. Part 2. A. Bau. *Woch. Braun.*, 1919, 36, 293–297, 301–305, 309–312, 319–321, 327–329, 337–339, 345–347, 359–360. *Chem. Zentr.* 1920, 91, II., 160–161.

ORDINARY acetic acid may contain glyoxylic acid, which is oxidised by air to oxalic acid. It is advisable therefore to keep the calcium acetate reagent (this J., 1918, 524A, 671A) in loosely stoppered bottles and to filter it before use. In the qualitative testing for traces of oxalic acid in materials containing starch great care is necessary to remove influences which destroy or mask the oxalic acid. Light and animal charcoal must be avoided, and to prevent oxidation of ferrous salts an atmosphere of carbon dioxide is desirable. Sulphates and magnesium salts mask the determination of oxalic acid. 0.02% of oxalic acid was found in barley and rye, in peas and soya beans 0.005–0.01%, whereas oats, rye and wheat flour, and white beans gave negative results. In a Japanese beer 28.64 mg. of oxalic acid per litre was found.—H. J. II.

Chloroform from coke-oven gas. A. J. Dey. *Pharm. J.*, 1920, 104, 302.

A SAMPLE of chloroform prepared from alcohol produced from coke-oven gas (see Bury, this J., 1920, 94A) complied with all the requirements of the B.P. except that, on evaporation, it yielded a characteristic odour. The latter could be removed by treatment with sulphuric acid, and was probably due to the presence of an unsaturated hydrocarbon.—W. P. S.

Ether; Action of — on metallic mercury. D. B. Dott. *Pharm. J.*, 1920, 104, 302.

PURE ether has no action on mercury, but ether containing peroxide reacts producing a dark-coloured film on the surface of the metal. When the ether contains much peroxide (the so-called ozonic ether) a certain amount of yellow mercuric oxide may be formed. The value of ether in aiding the complete mixing of mercury and calcium carbonate in making "grey powder" is due to the

presence of an oxidising impurity in the ether; pure ether would be useless for the purpose.

—W. P. S.

Phenols. Lyman and Reid. *See* III.

Reduction of nitro-compounds. Cusmano. *See* III.

Iodine values. Lakhani and Sudborough. *See* XII.

Organic chloro-compounds. Guyot and Simon. *See* XXIII.

Kjeldahl method. Phelps and Daudt. *See* XXIII.

PATENTS.

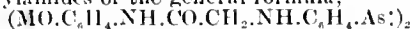
Ergot; Manufacture of a highly active preparation of —. Chem. Fabr. vorm. Sandoz, Basle, Switzerland. Eng. Pat. 125,396, 9.4.19. (Appl. 9006/19.) Int. Conv., 9.4.18.

POWDERED ergot is treated with a dilute aqueous acid such as oxalic acid, or solutions of acid salts, so as to render the basic constituents insoluble in the solvents subsequently used for the extraction of the fatty substances. The cellular tissue remaining after extraction of the fat is then mixed with fresh solvent to which an alkaline reagent such as barium hydroxide is added gradually until the acid is neutralised; the alkaloids are thus liberated and pass into the solvent from which they may be separated by evaporation under reduced pressure.

—D. F. T.

Arsenical compounds; Aromatic —. The Rockefeller Institute for Medical Research, Assignees of W. A. Jacobs, W. H. Brown, M. Heidelberger, and L. Pearce, New York, U.S.A. Eng. Pat. 128,181, 21.10.18. (Appl. 17,173/18.) Int. Conv., 13.6.18. Addition to 120,385 (see U.S. Pat. 1,280,123; this J., 1919, 268 A).

The sodium or potassium salts of the previously described (*loc. cit.*) N-(arsenoaryl)-bis- α -aminoacylarylamides of the general formula,



where M is the alkali metal, are equally valuable for treatment of trypanosomal or spirochetal infections and, being soluble in water, are particularly suitable for practical therapeutic use. The salts are prepared by dissolving in water equivalent quantities of the aromatic arsenic compound and the alkali hydroxide, and either evaporating to dryness under reduced pressure or precipitating the salt by the addition of a water-miscible liquid such as acetone or alcohol. The sodium salt forms a yellow powder readily soluble in water.

—G. F. M.

Gallic acid; Preparation of —. E. C. R. Marks, London. From Nitritfabrik Akt.-Ges., Koepenick, Germany. Eng. Pat. 139,419, 29.8.19. (Appl. 21,280/19.)

GALLIC acid is produced by heating to boiling solutions or suspensions of tannin with at least such quantities of metallic compounds, preferably the carbonates or hydroxides of the alkaline earths, that all the hydrogen atoms of the carboxyl and hydroxyl groups originally present and those formed by hydrolysis are substituted by the metal, and subsequently decomposing the gallates thus formed by the addition of an acid. It is advantageous to use the metallic compounds in some excess in order to assist in the coagulation of albuminous matter.

—G. F. M.

Mercuric iodide preparation; Manufacture of a —. J. Verfürth, Munich. Ger. Pat. 315,658, 9.7.16.

FATS, resins, and lipoids are added to solutions of red mercuric iodide in solvents which dissolve these substances, and the product may then be precipi-

tated by water. The precipitated mercury iodide does not turn into red mercuric iodide even on drying; it is soluble in many organic solvents such as ethyl alcohol, methyl alcohol, acetone, benzene, ethyl acetate, and ether, and can be rubbed down to yellow ointments.—J. F. B.

Hog-cholera virus; Artificial cultivation of the —. F. Proeschner, Sioux City, Iowa. U.S. Pat. 1,334,318, 23.3.20. Appl., 27.1.19.

Inulin; Process for obtaining — from plants. A. Daniel, Charlottenburg, Germany. Eng. Pat. 109,813, 18.9.17. (Appl. 13,418/17.)

SEE Ger. Pat. 313,986 of 1916; this J., 1920, 37 A.

Methyl alcohol; Manufacture of —. J. A. Christensen, Copenhagen. Eng. Pat. 125,946, 2.4.19. (Appl. 8320/19.) Int. Conv., 20.4.18.

SEE U.S. Pat. 1,302,011 of 1919; this J., 1919, 877 A.

Beta-halogen-ethylaminobenzoic esters and their derivatives. β -Alkylamino-ethylaminobenzoic alkyl esters and their derivatives. J. Altwegg and J. Landrion, Lyon, Assignors to Soc. Chim. des Usines du Rhône (anc. Gilliard, P. Monnet, et Cartier), Paris. U.S. Pats. 1,334,641—2, 23.3.20. Appl., 13.6.19.

SEE Eng. Pats. 128,553—4 of 1919; this J., 1920, 43 A.

Tobacco-leaves or leaves of like plants; Fluid for treating —. S. Hagino, Tokyo, Japan. U.S. Pat. 1,334,752, 23.3.20. Appl., 18.3.19.

SEE Eng. Pat. 137,683 of 1919; this J., 1920, 280 A.

XXII.—EXPLOSIVES; MATCHES.

Nitroglycerin waste acid mixtures; Formation of a solid phase in —. H. H. Dodds. J. S. Afr. Assoc. Anal. Chem., 1920, 3, 22—24.

FROM a sample of waste acid from nitroglycerin manufacture, kept at -3° to $+5^{\circ}$ C. in long closed glass tubes, there separated after about 5 days large transparent hexagonal crystals which continued to grow until about 15% of the acid had crystallised. In experiments with other waste acids of similar composition the crystals could not be obtained, although the original crystals continued to grow in any acid mixture of approximately the same composition up to the above-mentioned limit of 15%. The crystals had the composition:— H_2SO_4 , 79.40; HNO_3 , 3.47; HNO_2 , 0.32; H_2O (by diff.), 15.41; $\text{C}_{12}\text{H}_{11}(\text{NO}_3)_3$, 1.40%; total acidity (as HNO_3) 105.57%. Sp. gr. 1.7636 at 15° C. By recrystallising them twice at -1° C. their sulphuric acid content was raised to 82.18, and the nitric acid lowered to 1.43%, so that they gradually approached the theoretical composition of sulphuric acid monohydrate (84.48% H_2SO_4), with which also they agreed in appearance, m. pt. and sp. gr. The crystals, however, would not grow, but slowly dissolved in cooled mixtures of sulphuric acid and water (80—90% H_2SO_4) free from nitric acid. Since the crystals contain much less nitric acid than the liquid waste acid (e.g., 9.33%), a possible alternative method of denitration is suggested.—C. A. M.

Picric acid; Influence of the sulphonation of the phenol on the yield of —. M. Marquoyrol and P. Carré. Sealed note, 13.7.17. Bull. Soc. Chim., 1920, 27, 195—199.

THE yield of picric acid is much higher from phenoldisulphonic acid than from the monosulphonic acid, the difference in yield increasing with rise in the nitration temperature. In the sulphonation of the phenol the best yield of the disulphonic acid is obtained by heating 1 part of phenol with 5 parts of 93% sulphuric acid for $\frac{1}{2}$ —1

hour at 100° C. In practice the same result is attained by previously heating the sulphuric acid to 40° — 50° C. before adding the molten phenol at 50° — 60° C.—W. G.

Phenoltrisulphonic acid and its transformation into picric acid. M. Marquoyrol and P. Carré. Sealed note, 26.4.18. Bull. Soc. Chim., 1920, 27, 199—204.

By the sulphonation of phenol with ten times its weight of oleum containing 20% SO_3 , a mixture of the disulphonic and trisulphonic acids is obtained containing 80% of the trisulphonic acid. This latter acid, on nitration, gives a yield of picric acid equal to 90% of theory. Thus in the nitration of the phenolsulphonic acids the yield of picric acid increases from the mono- up to the trisulphonic acid, the accumulation of sulphonic acid groups increasing the resistance to oxidation of the molecule of phenol.—W. G.

Toxicity of picric acid. Koelsch. See XIXB.

PATENTS.

Explosive bodies; Process for producing compressed — from hexanitrodiphenylamine. O. von Schroeter, Oberlössnitz-Radebeul. Ger. Pat. 315,305, 4.2.09.

HEXANITRODIPHENYLAMINE is mixed with trinitrotoluene before compression. From a mixture of 80 parts of the former and 20 parts of the latter, bodies of sp. gr. 1.70—1.72 can be produced, which do not crack or become powdery.—J. H. L.

Matches; Manufacture of the igniting composition for — and matches manufactured by this method. O. J. Mellgren, Göteborg, Sweden. Eng. Pat. 140,026, 31.10.19. (Appl. 26,827/19.)

THE finely-divided bark of pine trees or other coniferous trees is used as a filling material and binding agent in match-head compositions.—W. J. W.

Match heads; Process of treating —. W. A. Fairburn, Great Barrington, Mass., Assignor to The Diamond Match Co., Chicago, Ill. U.S. Pat. 1,332,438, 2.3.20. Appl., 9.11.18.

MATCH-HEADS containing a gelatinous material and a chromium compound capable of interacting to produce a non-hygroscopic material are subjected after formation to the action of intense artificial light.—W. J. W.

Explosive materials; Manufacture of —. P. Landrin, Lyon, France. U.S. Pat. 1,334,462, 23.3.20. Appl., 31.1.18.

SEE Eng. Pat. 130,093 of 1918; this J., 1919, 742 A.

XXIII.—ANALYSIS.

Evaporator; Laboratory vacuum film —. H. E. Watson. J. Indian Inst. Sci., 1919, 2, 209—212.

A SMALL apparatus of the "Kestner Climbing Film Evaporator" type has been devised for laboratory purposes, and can be used either under atmospheric or reduced pressure. It comprises an evaporating tube of copper or tin, 6 ft. long and $\frac{1}{2}$ in. in external diameter; a steam jacket with upper and lower cocks; and a separator of tinned copper, the steam exit tube from which is connected to a condenser and receiver, the latter being attached by a side tube to a vacuum pump and manometer. The apparatus will evaporate 2 to 4 litres of water per hour under a pressure of 100 to 200 mm. of mercury, the steam being at atmospheric pressure.—C. A. M.

Nephelometer. C. Chéneveau and R. Audubert. Comptes rend., 1920, 170, 728—731.

THE absorption of light by the turbid medium is compensated by the displacement of a prism of

neutral-tinted glass of very slight angle. The adjustment is made so that the light transmitted by the medium and this prism fall side by side on a monochromatic green screen, and the prism is adjusted until the screen is uniformly illuminated. It is shown that the total mass, M , of the particles in suspension in the medium is a linear function of the displacement of the prism, and thus the value of M may be read off directly on a scale. (Cf. J.C.S., May.)—W. G.

Melting point; Influence of state of division on —.
F. Meissner. Z. anorg. Chem., 1920, 110, 169—186.

EXPERIMENTS with very fine powders of salol failed to show that, in a fine state of division, the melting point is lower than when the substance is in a coarser condition. In other experiments the substance was heated between a hot plate, in which a uniform temperature gradient was maintained, and a cylindrical lens, so that between the plate and lens there was a lamella of varying thickness. At that part of the plate where the temperature corresponded with the melting point a boundary line was formed between solid and liquid substance. The form of this line, in the cases of azobenzene, tristearin, and myristic acid, showed that the melting point of the thinnest lamella, 0.8μ , was distinctly lower than the normal melting point, 0.355°C . lower in the case of azobenzene and slightly less in the other cases. (Cf. J.C.S., May.)—E. H. R.

Films produced by metalloids and metals capable of being volatilised by heat; Rapid process for collecting and characterising —. A. Braly. Comptes rend., 1920, 170, 661—663.

A SIMPLE, portable apparatus for use in the field in applying the blowpipe test to minerals consists essentially of two sheets of mica mounted on some refractory material and capable of easy adjustment. On one sheet the mineral is heated in the blowpipe flame and the film is deposited on the other. By regulating the flame temperature successive films from the different metals or metalloids present in the mineral may be obtained. The usual tests for characterising the different elements present may be applied to the films.—W. G.

Manganese; Volumetric estimation of —. P. Nicolardot, A. Réglade, and M. Geloso. Comptes rend., 1920, 170, 803—810.

IS using von Knorre's method for the volumetric estimation of manganese (this J., 1902, 72), by precipitation with ammonium persulphate as the dioxide, re-solution of the precipitate in an excess of a reducing solution and back titration with a standard solution of potassium permanganate, the theoretical factor is 1 grm. $\text{Fe} = 0.4917$ grm. Mn. In practice a higher factor is necessary, owing to slight variation in the composition of the precipitate. The variations are due mainly to the presence of iron. In the absence of iron the factor found was 0.498, and in the presence of 40% of iron 0.4929. When the solution in which the precipitation takes place contains more than 10% of sulphuric acid the precipitation of the manganese is incomplete.

—W. G.

Molybdenum; Gravimetric determination of — as sulphide. J. Sterba-Böhm and J. Vostrebal. Z. anorg. Chem., 1920, 110, 81—103.

MOLYBDENUM can be precipitated quantitatively as MoS_3 by means of hydrogen sulphide in presence of formic acid if it is present entirely as molybdate; any trace of reduced molybdenum must be oxidised by means of nitric acid. A quantity of solution containing 0.1—0.3 grm. MoO_3 is diluted with water to 200—300 c.c., and formic acid is added to give a 5% solution of free

acid. If only traces of electrolytes are present in the solution, it is advisable to add a small quantity of pure potassium chloride, up to 0.75% calculated on the total volume of the solution. Directly the formic acid has been added a rapid stream of clean hydrogen sulphide (free from acid) is passed through the solution, in the cold for the first half-hour and then for 1—1½ hours at 40° — 50°C . Alternatively the hydrogen sulphide may be passed for 2 hours into a solution of the molybdate made alkaline with ammonia or potassium hydroxide, the formic acid then added, and the solution heated for one hour on the water bath. The precipitated molybdenum sulphide is washed by decantation, collected in a Gooch crucible, and dried in a current of carbon dioxide, first at 100°C . and then at 250°C . It is then cooled in the gas current and weighed in a weighing bottle. When these conditions are observed the product has the exact composition MoS_3 .—E. H. R.

Combustion of organic chloro-compounds by chromic-sulphuric acid mixture. J. Guyot and L. J. Simon. Comptes rend., 1920, 170, 734—736.

CHLORO-SUBSTITUTED esters may be estimated by oxidation with a mixture of chromic and sulphuric acids if an excess of chromic acid is used and an amalgamated copper coil is interposed between the reaction vessel and the gas burette where the carbon is measured so as to retain the chlorine. Methyl chlorosulphonate and chloroformate gave theoretical results, but in the case of the corresponding ethyl esters the ethyl group was not oxidised. Whereas acetic acid is not oxidised by this method, its chloro derivatives are completely oxidised.—W. G.

Nitrogen; The Kjeldahl method for the determination of —. I. K. Phelps and H. W. Daudt. J. Assoc. Off. Agric. Chem., 1919, 3, 218—220.

Numerous experiments were made with the object of rendering the Kjeldahl method applicable to the determination of nitrogen in all organic substances. It was found possible to determine nitrogen in the following "refractory" substances when 0.7 grm. of mercuric oxide, 10 grms. of potassium sulphate, and 25 c.c. of sulphuric acid were used for 0.2—0.4 grm. of substance, the digestion being prolonged for 2½ hrs.:—Isatin, atropine, cocaine, nicotinic acid, hydroxyquinoline, caffeine, amarine, quinoxaline hydrochloride, 2-methyl-1-quinazolinone, etc. Low results were obtained when sodium sulphate was used in place of potassium sulphate.—W. P. S.

Arginine nitrogen; Modification of the apparatus for the determination of — by Van Slyke's method. G. E. Holm. J., Amer. Chem. Soc., 1920, 42, 611—612. (See this J., 1911, 1135.)

By fitting a Kjeldahl flask with a cork carrying a tap funnel and the elongated water-jacketed tube of a Kjeldahl still-head, loss of material by bumping is prevented, and the necessity for transference of the solution after digestion with alkali is obviated.

—J. K.

Sulphur in petroleum. Christie and Bisson. See IIa.

Petrol. Florentin and Vandenberghe. See IIa.

Phenols. Lyman and Reid. See III.

Allwürden's reaction of wool. Kraus and Waentig. See V.

Sulphuric acid. Winkler. See VII.

Ammonium nitrate. Grissom. See VII.

Nitrates. Sanders. See VII.

Copper sulphate in works liquors. Carron. See VII.

Liquefied and compressed gases. Moser. See VII.

Analytical control of electrolytic zinc production. Bradley. See X.

Iodine values. Lakhani and Sudborough. See XII.
Phloroglucinol reagent. Dixon. See XII.
White lead. McMaster and Goldstein. See XIII.
Carbon black. Neal. See XIII.

Cellulose in rubber goods. Epstein and Moore. See XIV.

Molybdenum-value of tannins. Lauffmann. See XV.

Nitrogen in fertilisers. Frear and others. See XVI.

Lactose and dextrose. Hildt. See XVII.

Methyl alcohol in spirits. Hasse. See XVIII.

Reductase test for milk. Eichwald. See XIXa.

Milk analysis. Porcher. See XIXa.

Baking powder. Grünhut. See XIXa.

Strychnine. Buc. See XX.

Opium assay. Dott. See XX.

Oxalic acid. Ban. See XX.

PATENTS.

Gaseous mixtures; Means and method for analysing —. A. M. Kennedy, Cloverdale, Ala. U.S. Pat. 1,333,850, 16.3.20. Appl., 3.7.19.

A TEST stream of the gaseous mixture, containing an added quantity of an agent capable of combining chemically with abnormal constituents, is brought into contact with a catalyst, any chemical combination being indicated by a rise in temperature of the catalyst.—W. E. F. P.

Alcohol in beverages and the like; Process of and apparatus for estimating —. W. M. Dehn, Seattle, Wash. U.S. Pat. 1,333,905, 16.3.20. Appl., 16.10.19

A TUBULAR receiver extends partly within a distilling flask and has an outlet at the upper part outside the flask. The distillate is conducted from the flask to the bottom of the receiver through an inverted U-tube.—W. E. F. P.

Hardness of materials; Apparatus for testing the [ball] —. L. Johnson and H. Brearley, Sheffield, Eng. Pat. 140,165, 8.2.19. (Appl. 3171/19.)

Tungsten crucible. U.S. Pat. 1,333,036. See X.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Gallon and Whitfield. Distillation apparatus. 11,258. Apr. 23.

Garrow and Nielsen. Production of catalysts and oxygen carriers. 10,681. Apr. 17.

Lloyd. Furnaces. 10,422 and 10,433. Apr. 14.

Losey. Disintegrating fused materials. 10,947. Apr. 20.

Miller. Furnaces, ovens, kilns, etc. 10,526. Apr. 15.

Pettis. Decanting and agitating apparatus. 10,277. Apr. 13.

Schmidt. Recovering volatile solvents. 10,582. Apr. 15. (Ger., 7.8.15.)

Thäberg. 10,565. See II.

Vanneau. Grinding or crushing apparatus. 10,387. Apr. 14. (Fr., 9.11.17.)

Walker. Drying methods. 10,618. Apr. 16.

Walter. Purification of feed water for steam generators etc. 10,762. Apr. 17.

COMPLETE SPECIFICATIONS ACCEPTED.

16,229 (1918). Fadden. Furnaces for producing mineral distillates of definite composition. (141,089.) Apr. 21.

353 (1919). Schuck. See XII.
 392 and 393 (1919). Fidler and Maxwell. Continuous kilns and dryers of tunnel and car type. (141,124—5.) Apr. 21.

5121 (1919). Brian. Vertical drying-kiln. (141,468.) Apr. 28.

12,103 (1919). Plumbridge. Recovery of volatile solvents. (141,210.) Apr. 21.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Brownlee. Lubricating oils. 10,738. Apr. 17. (U.S., 13.5.18.)

Craven and Ormandy. Production of mixed fuels consisting of solids and liquids. 10,532. Apr. 15.

Fay, and United Oil and Coal Corporation. Storage and use of colloidal fuel. 11,234. Apr. 22.

Fay, and United Oil and Coal Corporation. Fuel for furnaces of steam generators etc. 11,235. Apr. 22.

Griffin. Enrichment of producer gas. 10,496. Apr. 15.

Howgate. Retorts for manufacture of coal-gas etc. 11,000. Apr. 20.

Jackson (International Gasoline Process Corporation). Apparatus for distillation of oil. 10,446. Apr. 14.

Kopp (Wurmback and Hoeppermann.) Production of liquid fuel for explosive motors etc. 10,634. Apr. 16.

Macdonald. Apparatus for cracking oils. 11,100. Apr. 21.

Marks (Soc. Franco-Belge de Fours à Coke). Coke ovens. 10,186. Apr. 12.

Meade. Manufacture of coal gas. 10,971. Apr. 20.

Smith. Purifying mineral oils. 10,390. Apr. 14.

Thäberg. Apparatus for filtering and pressing peat etc. 10,565. Apr. 15.

Trent. Method of cleaning coal. 10,572. Apr. 15. (U.S., 9.7.19.)

Trent. Treating carbonaceous etc. material. 10,573. Apr. 15. (U.S., 16.9.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

10,352 (1918). Strache. Gasification of carboniferous substances. (117,083.) Apr. 21.

18,039 and 19,134 (1918). Wade (Le Petit). Production of liquid fuels. (141,091.) Apr. 21.

18,270 (1918). Bataafsche Petroleum Maatsch., and Brey. Rectification of hydrocarbons. (123,719.) Apr. 21.

19,677 (1918). Marks. See VII.

9491 (1919). Stafford. Destructive distillation. wood, woody fibre, etc. (141,417.) Apr. 28.

6523 (1919). Dibdin. Revivifying spent oxide. (141,172.) Apr. 21.

9491 (1919). Stafford. Destructive distillation. (141,505.) Apr. 28.

13,374 (1919). Andrews and Averill. Treating hydrocarbons for the production of lighter hydrocarbons. (141,223.) Apr. 21.

16,383 (1919). Dempster and Beard. Purifiers employed in gas-works. (141,572.) Apr. 28.

22,094 (1919). Hall Motor Fuel, Ltd., and McCrea. Desulphurising sulphur-bearing hydrocarbons. (141,272.) Apr. 21.

III.—TAR AND TAR PRODUCTS.

APPLICATION.

Tindale. Distillation of coal tar and products derived therefrom. 10,924. Apr. 20.

COMPLETE SPECIFICATIONS ACCEPTED.

18,270 (1918). Bataafsche Petroleum Maats. See II.
1338 (1919). South Metropolitan Gas Co., and Stanier. See XX.

IV.—COLOURING MATTERS AND DYES.

COMPLETE SPECIFICATIONS ACCEPTED.

17,225 (1917). Bayer and Co. Manufacture of monoazo dyestuffs. (133,012.) Apr. 21.
20,534 (1918). Imray (Soc. Chem. Ind. in Basle). Mordant dyeing disazo-dyestuffs, and their application in dyeing and printing. (141,401.) Apr. 28.
30,839 (1919). Imray (Soc. Chem. Ind. in Basle). Manufacture of monoazo dyestuffs. (141,643.) Apr. 28.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Bronnert. Manufacture of viscose silk. 10,195. Apr. 12.
Bronnert. Manufacture of artificial silk. 11,362. Apr. 23.
Eastern Manufacturing Co. Finishing paper. 11,073. Apr. 21. (U.S., 9,12,16.)
Tattersall. Separating fatty etc. matters from effluents due to washing wool etc. 10,468. Apr. 15.
White. Manufacture of fibrous material from zoophytes etc. 11,415. Apr. 21.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Brandwood and Brandwood. Apparatus for dyeing yarns. 11,065. Apr. 21.
Calico Printers' Assoc., and Rouse. Printing cotton fabrics. 10,130. Apr. 12.
Calico Printers' Assoc., and Warr. Production of patterns etc. on fabrics. 10,286. Apr. 13.
Duncan. Dyeing and waterproofing material. 10,791. Apr. 19.
Nägelin. Apparatus for treating textile goods in hank form by circulating liquid. 10,429. Apr. 14. (Switz., 4,10,19.)

COMPLETE SPECIFICATIONS ACCEPTED.

20,116 (1918). Lord. Apparatus for treating hanks of yarn with liquids. (141,098.) Apr. 21.
20,534 (1918). Imray (Soc. Chem. Ind. in Basle). See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Bird. 10,282. See XVII.
Clayton. Sulphur-burning furnaces etc. 11,343. Apr. 23.
Dent, Golding, and United Alkali Co. Production of sulphur compounds. 10,855. Apr. 19.
Dior. Sulphuric acid chambers. 10,386. Apr. 14.
Freeth and Munro. Purification of rock-salt brine etc. 10,608. Apr. 16.
Garrow and Nielsen. 10,681. See I.
Mehner. Manufacture of cyanogen compounds. 11,116. Apr. 21. (Ger., 9,10,14.)
Soc. l'Air Liquide. Production of hydrogen peroxide. 10,751. Apr. 17. (Fr., 11,5,14.)
Wade (Lindsay Light Co.). Recovery of thorium. 11,115. Apr. 21.
Watson. Removal of arsenic from hydrochloric acid. 10,909. Apr. 20.

COMPLETE SPECIFICATIONS ACCEPTED.

19,677 (1918). Marks (Soc. Franco-Belge de Fours à Coko). Production of sulphate of ammonia from distillation and like gases. (141,093.) Apr. 21.
365 (1919). Armour Fertilizer Works. Production of aluminium nitride. (122,829.) Apr. 28.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Clarke, Girtin, and Rhodin. Bleaching sand. 10,860. Apr. 19.
Ferguson. Manufacture of sheet glass. 11,323. Apr. 23.
Norway and Scholz. Enamel-ware. 10,261. Apr. 13.
Rollason. Manufacture of refractory materials for furnace linings. 11,002. Apr. 21.
Travers. Glass-melting pots and blocks for tank furnaces. 10,201. Apr. 12.
Virginia Plate Glass Corporation. Manufacture of glass. 10,579. Apr. 15. (U.S., 17,4,19.)

COMPLETE SPECIFICATIONS ACCEPTED.

927 (1919). Carborundum Co. Manufacture of refractory articles. (133,014.) Apr. 28.
24,884 (1919). Atkinson, Stoin and Atkinson, and Moorshead. Glass furnaces. (141,617.) Apr. 28.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Fabr. Aktiebolaget Kronsten. 11,042. See XI.
Grubenholz-Imprägnierung Ges. Impregnating wood. 10,436. Apr. 14. (Ger., 29,11,13.)
Haring, Voit, and Holzbearbeitungsges. Process of drying wood. 10,435. Apr. 14. (Ger., 28,3,19.)
Milner and Robinson. Manufacture of bricks, blocks, etc., from blast-furnace slag. 10,302. Apr. 13.

COMPLETE SPECIFICATIONS ACCEPTED.

10,937 (1918). Hoare. Production of building material. (141,390.) Apr. 28.
180 (1919). Badder, Burrows, and Allender. Waterproof cements, concretes, and mortars. (141,113.) Apr. 21.
1113 (1919). Boillot and Daudignac. Apparatus for manufacturing cement. (141,142.) Apr. 21.
18,775 (1919). Roy. Rendering wood impermeable and recovering liquids contained therein. (140,361.) Apr. 28.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Bangerter. Metal alloy. 10,125. Apr. 12.
British Thomson-Houston Co. (General Electric Co.). Tungsten alloys. 10,756 and 10,835. Apr. 17 and 19.
Collins. Purification and recovery of tin. 10,959. Apr. 20.
Hurley. Process for galvanising or zinc-dipping articles. 10,238. Apr. 13.
Milner and Robinson. 10,302. See IX.
Minerals Separation, Ltd., and Wood. Concentration of oxidised ores. 10,929. Apr. 20.
Naef. Treatment of copper-nickel matte. 10,895. Apr. 20.
Newman, and Rustproofing Synd. Rustproofing ferrous metals etc. 11,106—8. Apr. 21.

COMPLETE SPECIFICATIONS ACCEPTED.

16,801 (1918). Cooper Research Co. Alloys. (120,565.) Apr. 28.
18,982 (1918). Riley. Coating steel or iron with white alloy. (141,092.) Apr. 21.
374 (1919). Worrall. Hard metal alloys. (141,122.) Apr. 21.
532 (1919). Waring. Open-hearth furnaces. (141,132.) Apr. 21.
10,604 (1919). Soc. Anon. de Commentry-Fourchambault. Alloys. (127,243.) Apr. 28.
10,637 and 25,975 (1919). Howgate. Annealing. (141,200.) Apr. 21.
16,133 (1919). Bellais. Gold alloys. (141,246.) Apr. 21.

22,296 (1919). Dutoit and Boever. Manufacture of brass. (132,520.) Apr. 28.
 24,410 (1919). Iversen. Solder for aluminium and other metals. (141,280.) Apr. 21.
 26,819 (1919). Cottineau. Obtaining the copper from lyes resulting from the treatment of cupriferous pyrites. (141,290.) Apr. 21.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Automatic and Electric Furnaces, Ltd., Barfield, and Wild. Electric muffle furnaces. 10,299. Apr. 13.
 Cornelius. Electric rotating furnaces. 10,293. Apr. 13.
 Fabriks Aktiebolaget Kronsten. Insulating and building material. 11,042. Apr. 21. (Sweden, 24.4.19.)
 Law (Smith). Storage batteries. 11,078. Apr. 21.
 Merritt. Electrolytic method, apparatus, and product. 10,561. Apr. 15. (U.S., 15.4.19.)
 Naylor, and Rushmores, Ltd. Secondary batteries. 11,198. Apr. 22.

COMPLETE SPECIFICATION ACCEPTED.

1475 (1920). Hepburn. Electrolysers. (141,305.) Apr. 21.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Catalpo, Ltd., and Fryer. Process of hydrolysis. 11,437. Apr. 24.
 Hargreaves. Manufacture of soap. 11,152. Apr. 22.
 Lancaster. Recovery of oils, fats, soap curds, etc., from sewage etc. 10,480. Apr. 15.
 Mason. Separation of grease and oil from liquids. 10,337. Apr. 13.
 Persapol Ges. Conversion of fatty acids or their glycerides into oleic acid-like fatty acids or their soaps. 10,335. Apr. 13. (Ger., 6.5.14.)
 Tattersall. 10,468. See VII.
 Townsend. Detergent. 10,934. Apr. 20.
 Tseng. Manufacture of soap. 10,236. Apr. 13.

COMPLETE SPECIFICATIONS ACCEPTED.

353 (1919). Schuck. Production of catalysts suitable for use in hydrogenation processes. (122,192.) Apr. 21.
 14,729 (1919). Erslev. See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

COMPLETE SPECIFICATIONS ACCEPTED.

758 (1919). Cleghorne, and Gayner Pneumatic Co. Materials for coating substances to protect them or render them non-porous. (141,414.) Apr. 23.
 27,926 (1919). Tsutsumi. Solid ink compound. (141,631.) Apr. 28.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Cordingley. Manufacture of compounded gutta-percha and balata for cases, bags, etc. 11,013. Apr. 21.
 Davidson. Treatment of raw rubber. 10,829. Apr. 19.
 Gray. Purification of india-rubber, gutta-percha, etc. 10,555. Apr. 15.
 Marshall. Vulcanising for manufacture of high-pressure expanded vulcanised rubber etc. 10,497. Apr. 15.
 Stevens. Preservation of vulcanised rubber. 10,599. Apr. 16.

COMPLETE SPECIFICATION ACCEPTED.

704 (1919). Gaisman and Rosenbaum. Vulcanising caoutchouc and caoutchouc-like substances. (141,412.) Apr. 28.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Jackson (Dorr Co.). Recovering proteids from waste liquors of hide-treating. 10,445. Apr. 14.
 Rohm and Haas Co. Tanning-materials. 10,309 and 10,998. Apr. 13 and 20. (U.S., 4.12 and 6.6.19.)
 Zignone. Manufacture of imitation leather. 10,229. Apr. 12. (Switz., 11.4.19.)

COMPLETE SPECIFICATION ACCEPTED.

9634 (1916). Miller. Artificial leather or leather substitutes. (141,385.) Apr. 28.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATION.

Bird. Recovering potash from waste products in cane-sugar and rum distilling. 10,282. Apr. 13.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATION.

Beanes. Preparation of grain for brewing etc. 10,219 and 11,223. Apr. 12 and 22.
 Bird. 10,282. See XVII.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Beanes. Treatment of grain for food for animals. 11,224 and 11,225. Apr. 22.
 Benton and Bishop. Flour-milling processes. 11,257. Apr. 23.
 Borrowman. Water-softening. 10,848. Apr. 19.
 Fryer. Insecticides, sheep-dips, etc. 10,937 and 10,938. Apr. 20.
 Hucks. Production of materials for flavouring liquids etc. 10,975. Apr. 20.
 Lancaster. 10,480. See XII.
 Rose. Preparation and storage of food. 10,224. Apr. 12.
 Spear and Spear. Manufacture of food product. 10,977. Apr. 20.
 Walter. 10,762. See I.

COMPLETE SPECIFICATION ACCEPTED.

14,729 (1919). Erslev. Adapting oil-cakes and the like for human food. (128,216.) Apr. 28.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATION.

Berk and Co., and Hood. Purification of organic bodies. 11,419. Apr. 24.

COMPLETE SPECIFICATIONS ACCEPTED.

16,093 (1918). Rockefeller Institute for Medical Research. Aromatic arsenical compounds. (120,382.) Apr. 21.
 353 (1919). Schuck. See XII.
 1338 (1919). South Metropolitan Gas Co., and Stanier. Manufacture of colourless organic compounds. (141,440.) Apr. 28.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Colour Photography, Ltd., Greene, and Thomson. Colour photography. 10,576. Apr. 15.
 Elmassian. Colour photography. 10,155 and 10,180. Apr. 12.
 Gorsky. Three-colour photography. 10,955. Apr. 20.
 Mills. Photographic transfer processes. 10,823. Apr. 19.

COMPLETE SPECIFICATION ACCEPTED.

28,287 (1919). Christensen. Hardening photographic films during development. (135,477.) Apr. 28.

I.—GENERAL; PLANT; MACHINERY.**PATENTS.**

Furnaces [; *Cylindrical gas-fired* —]. A. B. Chantaine, Marcinelle, Belgium. Eng. Pat. 18,473, 8.8.14. Int. Conv., 10.9.13.

THE gas and air are admitted under slight pressure through separate ports or burners tangentially to the inner wall of a cylindrical chamber, a gas inlet being arranged immediately behind each air inlet, so that the air is thrown outwards against the inner wall of the furnace; the pressures of the air and of the gas are independently adjustable. Separate outlets for products of combustion may be provided for each pair of gas and air inlets.—B. M. V.

Distillation; Continuous fractional —. A. M. O'Brien, Abadan, Persian Gulf. Eng. Pat. 140,140, 13.12.18. (Appl. 20,765/18.)

IN a series of stills for the continuous distillation of petroleum or other substance the stills decrease in size approximately in proportion to the amount of liquor to be distilled in them; the condenser tanks are also made in corresponding dimensions, and the same cooling water may be used in all of them, starting at the largest or coolest. Some (or all) of the products of combustion may be transferred from furnace to furnace, any additional heat necessary being made up by auxiliary burners or furnaces.—B. M. V.

Refrigerating processes; Solution for absorption —. C. Delaygue, Marseilles. Eng. Pat. 140,356, 23.12.13. (Appl. 367/20.)

IN the ammonia absorption refrigeration process the following is used as the absorbing liquor:—Ammonium nitrate 3 grms., trimethylamine 2 grms., liquefied ammonia gas 554 c.c., and water to 1 litre. The absorptive power of ammonium nitrate solution for ammonia is greatly increased by addition of trimethylamine.—B. M. V.

Gases; Electrical treatment of —. L. Bradley, East Orange, N.J., Assignor to Research Corporation, New York. U.S. Pat. 1,333,790, 16.3.20. Appl., 28.6.15.

THE apparatus comprises a number of rigid discharge electrodes, arranged vertically and axially within a number of opposing tubular electrodes, with radially adjustable flanges fixed longitudinally on the discharge electrodes. The discharge electrodes are connected at their upper and lower ends respectively to insulated frames above and below the tubular electrodes.—B. N.

Gases; Apparatus for electrical treatment of —. J. C. Davidson, Vancouver, B.C., Assignor to International Precipitation Co., Los Angeles, Cal. U.S. Pat. 1,334,231, 16.3.20. Appl., 3.2.17.

THE apparatus comprises a gas receiving chamber, a discharge electrode mounted on an insulator, and a gas inlet provided with a deflector surrounding the insulator, and situated in the path of the gas to protect the insulator from the deposition of material suspended in the gas. Means are provided for supplying liquid to the insulator to remove deposited material. The insulator comprises a bushing of fragile material extending through the wall of the gas chamber, and connected to it by a yielding medium; a conductor extends through the bushing and is connected to the discharge electrode, and there is a support for the conductor outside the chamber.—B. N.

Electrical purification of gases; Insulator for use in the —. Siemens-Schuckertwerke, G.m.b.H., Siemensstadt. Ger. Pat. 316,498, 13.10.18.

THE leads to the high-tension electrodes are em-

bedded in a number of insulating plates arranged within the precipitation chamber so that their planes are at right angles to the direction of flow of the gas. The plates occupy the upper part of the chamber, and their transverse length equals that of the chamber, so that no appreciable flow of gas occurs within the space between two adjacent plates. The high-tension electrodes are connected to the embedded leads and alternate with low-tension gauze electrodes.—J. S. G. T.

Electrical purification of gases; Appliance for the —. Siemens-Schuckertwerke, G.m.b.H., Siemensstadt. Ger. Pat. 318,132, 29.5.18.

THE material precipitated from the gas collects in a chamber, beneath the stream of gas, in which a number of easily removable slides or flaps are arranged at right angles to the direction of flow of the gas, in order to prevent motion of the gas in the collecting chamber.—J. S. G. T.

Electrical purification of dusty gases; Process for —. H. Püning, Münster. Ger. Pat. 318,772, 11.2.19.

AT intervals such a velocity is imparted to the gas stream as will enable it to detach and carry forward from the electrodes any deposited dust.—J. S. G. T.

Grinding machine. W. Travell, New York. U.S. Pat. 1,333,946, 16.3.20. Appl., 9.3.16.

A GRINDING mill comprises a shell provided with partitions having central openings and forming internal circumferential grooves containing grinding rollers. Adjacent partitions communicate through openings, out of alinement with respect to each other, and permitting passage of material.—B. N.

Filter; Continuous electrical mechanical —. A. F. Meston, Assignor to Research Corporation, New York. U.S. Pat. 1,334,160, 16.3.20. Appl., 23.2.18.

AN apparatus for filtering a liquid is provided with two electrodes, one of which is formed of a drum with a porous rim and mounted so as to be rotated, the speed of rotation varying in proportion to the current flowing through the circuit. The porous rim is surrounded by a filtering medium, and the liquid is passed between the electrodes.—B. N.

Catalysers; Composition for making —. C. Ellis, Montclair, N.J. U.S. Pat. 1,335,161, 30.3.20. Appl., 21.8.19.

A COMPOSITION which yields a catalyst when heated consists of a basic compound of nickel ground with a small amount of oil.—A. B. S.

Contact-substances; Ceramic support for —. V. Zieren, Berlin-Friedenau. Ger. Pat. 317,979, 25.10.17.

Porous ceramic material, preferably in the form of small tubes with projecting rims, is employed as a support for contact substances. The diameter of the tubes should not exceed 20 mm., and the thickness of the walls 2 mm. The material is impregnated with, for example, a platinum solution, and this reduced, so that the tube becomes coated internally and externally with metallic platinum.—J. S. G. T.

Recovering dissolved or finely divided material; Process for —. I. Ebers, Ahrensburg. Ger. Pat. 309,102, 9.1.18. Addition to 306,484 (this J., 1920, 258 A).

THE process of atomising the liquid flowing from the pressure vessel is assisted by introducing some of the vapour of the liquid at the jet through which the liquid is sprayed.—J. S. G. T.

Concentrating liquids; Process and apparatus for —. A. Herrlich, Halle. Ger. Pat. 315,035, 21.3.18.

THE liquid to be concentrated flows downwards over a number of hollow plates heated internally, so that the briskly boiling liquid on any plate impinges forcibly on the lower corrugated surface of the plate immediately above. The vapours flow over the corrugated surface to the circumference, where they strike against an annular baffle, any liquid which is separated passing, together with the residue of the liquor to be concentrated, down to the next lower heated plate.—J. S. G. T.

Concentrating or drying liquids; Process for —. A. Friedländer, Berlin-Halensee. Ger. Pat. 316,489, 3.9.18.

SUBSTANCES which swell on wetting, e.g., glue, are formed into plates, rods, etc., upon a skeleton frame, and these frames are supported parallel to one another in the liquid to be concentrated with so much space separating them that when the material swells adjacent frames do not touch each other or the walls of the chamber. The concentrated liquid may be drawn off, or the plates may be withdrawn and the thickened mass adhering to them removed mechanically.—J. S. G. T.

Fractionation of gaseous mixtures by partial condensation due to cooling under pressure. Ges. für Lindes Eismaschinen A.-G., Höllriegelskreuth. Ger. Pat. 316,343, 5.2.18.

THE choking of the fractionating apparatus is prevented by liquefying the solid portion of the solid and liquid fraction already separated from the gas prior to relieving the pressure. For this purpose heat exchange by counter-current is effected with the stream of gas to be fractionated.—J. S. G. T.

Filling material for absorption and reaction towers; Hollow bodies for use as —. V. Zieren, Berlin-Friedenau. Ger. Pat. 316,497, 19.12.18.

A NUMBER of rings of different angular patterns are arranged one upon another, and so joined that they form a coherent body, in which the component elements of similar pattern are arranged parallel to one another. Alternatively, rings of the same angular pattern may be used, but with the angles of the rings out of alinement in vertical series. The rings are composed of wire, either single or plaited, or may be cut from a tube and suitably shaped by notching and bending.—J. S. G. T.

Collecting and washing salts; Apparatus for —. H. A. Schmidt, Würzen. Ger. Pat. 316,702, 20.10.18.

By means of a movable mechanical device a number of stationary suction-filter vessels can be consecutively washed and the salt collected and conveyed to the next filter vessel of the series.—J. S. G. T.

Electrical heater for vapours or conducting gases. Allgem. Elektrizitäts-Ges., Berlin. Ger. Pat. 317,302, 8.5.18.

THE gas or vapour is itself used as heating resistance, the heater consisting of a number of parallel insulated pipes connecting the supply pipe for the gas with the delivery pipe. Insulated electrodes are introduced within the insulated pipes, and the electric current passes from these electrodes through the gas or vapour. The device is best operated with high tension current, and can be employed, for example, as a steam superheater in conjunction with an electrical steam boiler.—J. S. G. T.

Protecting apparatus and vessels used in chemical industry against the action of chemicals; Method of —. F. Schüller, Frankfurt. Ger. Pat. 318,033, 14.9.18.

A PROTECTIVE coating is formed of a number of thin

layers of stoneware alternating with layers of silicate or thin layers of glass. The first layer of stoneware is reinforced with metal insertions.

—J. S. G. T.

Prevention of frothing; Apparatus for —. A. Redlich, Vienna. Ger. Pat. 318,478, 26.9.18. Int. Conv., 10.10.17.

A NUMBER of chambers are in a circle around and carried by a shaft which rotates within the space where frothing may occur. The chambers are open only in the direction of rotation, and are each provided with a drainage tube for liquid resulting from bursting of the bubbles constituting the froth.

—J. S. G. T.

Grinding, mixing, and like machines; Scrapers for use in —. O. Shaw, Stockport, and F. Shaw and Co., Ltd., Bradford. Eng. Pat. 140,483, 30.9.18. (Appl. 15,845/18.)

Washing coal, coke, sand, and other materials; Apparatus for —. J. H. Harrison, Gateshead. Eng. Pat. 140,616, 15.4.19. (Appl. 9639/19.)

Purifying liquids [soiled lubricating oils] by sifting, depositing, and overflowing action; Apparatus for —. A. E. Lienart, Brussels. Eng. Pat. 140,677, 30.6.19. (Appl. 16,383/19.) Int. Conv., 10.3.19.

Furnaces; Means for consuming smoke in —. C. H. Halt, Indianapolis, Ind., U.S.A. Eng. Pat. 140,859, 2.1.19. (Appl. 162/19.)

Centrifugal apparatus with planetary movement. W. Mauss, Johannesburg. Eng. Pat. 140,962, 15.8.19. (Appl. 20,118/19.)

Emptying the contents of bins into furnaces or the like; Devices for —. B. E. D. Kilburn, London. From Sulzer Frères, Winterthur, Switzerland. Eng. Pat. 140,963, 20.8.19. (Appl. 20,458/19.)

Tunnel kilns. G. H. Benjamin, New York. Eng. Pat. 140,578, 6.3.19. (Appl. 5605/19.)

SEE U.S. Pat. 1,292,568 of 1919; J., 1919, 313 A.

Furnaces. A. Smallwood, London. U.S. Pats. 1,335,202-4, 30.3.20. Appl., 31.7.18.

SEE Eng. Pats. 125,744, 125,224, and 125,515 of 1918; J., 1919, 489 A, 372 A, 347 A.

Evacuating gases from vessels; Arrangement for —. H. Gerdien, Assignor to Siemens u. Halske A.-G., Berlin. U.S. Pat. 1,335,829, 6.4.20. Appl., 6.10.15.

SEE Eng. Pat. 14,918 of 1915; J., 1916, 101.

Drying organic materials. Ger. Pat. 315,497. See IIB.

Electro-osmotic process. Eng. Pat. 135,817. See XI.

Electro-osmotic dehydration. Ger. Pats. 316,444 and 316,494-6. See XI.

Electro-osmotic electrode. Ger. Pat. 316,593. See XI.

Displacement of liquids. Ger. Pats. 305,512 and 307,701-2. See XI.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Banded bituminous coal; Behaviour of the constituents of — on coking. Studies in the composition of coal. R. Lessing. Chem. Soc. Trans., 1920, 117, 247-256.

COKING tests were carried out on samples of fusain, durain, clarain, and vitrain (cf. J., 1919, 452 A,

614A) in an apparatus previously described (J., 1912, 465, 471), at 900° C. with a heating period of 7 min. and at 600° for 5 min. Fusain showed no outward change in appearance after coking, and had no tendency to cohere, the residue being a powder. Durain had a little greater tendency to cohere than fusain, but the particles retained their angular shape and form. Clarain showed considerable fusion and merging of particles. At 900° C. most of the fine and medium-sized particles lost their identity almost completely. The vitrain particles were well sintered and showed rounding of the angular edges, but the consolidation of particles did not go so far as with clarain. The coke specimens from vitrain were of the "mushroom" type. At 900° C. the free carbon formed with durain was considerably higher than with clarain and vitrain. On the other hand, at 600° C. no carbon was produced from durain, whilst the amounts from clarain and vitrain were only slightly reduced. The gas yields from clarain and vitrain were almost equal, and greater than that from durain. This seems to indicate a considerable difference in the chemical nature of the primary decomposition products of durain on the one side and of clarain and vitrain on the other.—W. P.

Banded bituminous coal; Mineral constituents of —. Studies in the composition of coal. R. Lessing. Chem. Soc. Trans., 1920, 117, 256—265 (cf. ante).

The peculiar behaviour on carbonisation of fusain, durain, clarain, and vitrain suggested that the results were influenced or accentuated by the catalytic effect of their mineral components. Considerable differences were found in the composition of the ashes from the four constituents. A large proportion of lime and soluble matter was found in fusain, and its presence was probably due to infiltration of calcium salts; the lime appears to be present in fusain as calcium carbonate. The thin veins of white mineral found along the cleavage planes contained 40—45% CaCO₃. The composition of the durain ash was comparatively simple, the ash consisting to the extent of 93% of alumina and silica. The composition of the ashes of clarain and vitrain suggest that they represent mainly the remainder of the original plant ashes.—W. P.

Coal; Action of — upon a photographic plate. E. Sinkinson. Chem. Soc. Trans., 1920, 117, 165—170.

The action of coal upon a photographic plate in the dark (cf. J., 1913, 969; 1918, 262A; 1919, 452A) depends upon the presence of oxygen. A sample of coal saturated with oxygen had less effect than an untreated sample, indicating that the absorption of oxygen is part of the mechanism of the action. Coals which had been heated above 500°—530° C. no longer had any action on a photographic plate. It is concluded that the ingredient causing the effect on a photographic plate in the dark is to be found in that portion of the coal which is extracted by pyridine, other than the resin (J., 1919, 752A).

—W. P.

Coal; Effect of excessive moisture in —. A. H. Thwaite. Coke Oven Managers' Assoc., 13.3.20. Gas World, 1920, 72, Coking Sect., 38—39.

The optimum moisture content for coal to be carbonised is given as 8—10%. The drainage of the washed coal would be considerably assisted if large lumps of good coal were crushed separately from inferior coal to a size from 2 in. cube downwards. From results obtained by Bone and Mellor it is estimated that 924 cu. ft. of gas is required to evaporate each 1.0% of water in a ton of coal above 80%. Excessive moisture leads to damage of the oven walls, and an increased carbonising period of

at least 2 hrs. for every 2% moisture above 10% is necessary. As regards the by-products, excessive moisture imposes heavy duties on the exhaustors, condensers, and (in the case of an indirect ammonia plant) the stills, which have to treat a weaker liquor than otherwise. On direct ammonia plants the effect is more serious, as all moisture from the coal is carried along with the gas, making it very difficult to maintain the necessary temperature for heating the ovens.—A. G.

Moisture in coals, etc.; Determining —. A. R. Myhill. Gas J., 1920, 150, 21.

The method comprises the distillation of 200 c.c. of water-free benzol, toluol, or xylol with 100 g. of coal, or other substance to be tested, up to 110° C. or over, the distillate being caught in a graduated receiver.—A. G.

Water-gas; Heat conservation and —. E. G. Stewart. London and S. Distr. Jun. Gas Assoc., 26.3.20. Gas J., 1920, 149, 750—755.

The factors governing the efficiency of a water-gas plant are discussed and it is pointed out that a counter-current of steam possesses a distinct advantage, leading to good reduction and the provision of a cool top zone. With the blow waste gases at 1800° F. (980° C.) the efficiency of the plant is given as 58%, whilst with the gases at 1500° F. (815° C.) and the carbon burning to 90% CO during the air blow, the thermal efficiency may be only 34%. Working results of a large carburetted water-gas plant at Fulham are given, together with a description of a waste-heat boiler of special design and of the economies effected by its use.—A. G.

Gas; Influence of impurities in — on the corrosion of meters and the blockage of service pipes. A. R. Myhill. Gas World, 1920, 72, 265.

DEPOSITS caused by impurities have been found to consist of iron rust, free sulphuric acid, iron sulphates, thiocyanates, ferrocyanides, pyridine and ammonium compounds, and free sulphur. Corrosion is facilitated by the presence of air and moisture and of cyanogen compounds and sulphide. An insoluble iron-ammonium-hydrogen ferrocyanide is produced when ammonia, ferric oxide, iron, and hydrocyanic acid react together, and pyridine forms a similar compound. Pyridine can be washed out of the gas by the use of cold water in the finishing scrubber, and the Williams process for the removal of cyanogen is recommended, ammonium thiocyanate being recovered.—A. G.

Benzol; Estimation of — in coke-oven gas and scrubbing oil. L. Shuttleworth. Gas World, 1920, 72, Coking Sect., 43.

DETAILS are given of the determination of benzol in coke-oven gas by absorption in petroleum oil and of the benzol in the oil by means of fractionation.—A. G.

Hydrocarbons; Analysis of — by the aid of bacteria. J. Tausz and M. Peter. Zentr. Bakt. u. Parasit., II. Abt., 1919, 49, 497—554. Chem. Zentr., 1920, 91, II., 261.

THE separation of paraffins from naphthenes can be effected by means of *B. aliphaticum*, *B. aliphaticum liquefaciens*, and the paraffin bacterium, which are described; they were isolated from garden mould by culture in organic and inorganic media containing *n*-hexane, cyclohexane, or paraffin oil. Paraffin bacteria are without action on naphthenes, henzenoid hydrocarbons, and some paraffins (*n*-hexane and *n*-octane), but attack higher paraffins such as hexadecane, triacontane, and tetratriacontane. The other two species are inert towards cyclic hydrocarbons and hexylene, but attack paraffins and *n*-caprylene and hexadecylene.

Destruction of the reactive hydrocarbons is complete even in the presence of hydrocarbons which are not attacked. The presence of very small proportions of aliphatic hydrocarbons in natural naphthenes or of impurity in artificial specimens is shown by the clouding due to bacterial growth of media containing the hydrocarbon under examination. The method can be used for the detection of paraffins in crude oils and their products and for the isolation of pure naphthenes. As regards enzymes urease was absent; diffusion lipase and proteolytic enzymes were shown in the case of *B. aliphaticum* and the paraffin bacterium, diastase by paraffin bacterium and *B. aliphaticum*, and catalase by both the aliphaticum species. New constants for hydrocarbons purified by means of the bacterial treatment are: 1,3-dimethylcyclohexane, b.p., 118°–120° C., sp. gr. at 20°/4° C., 0.771; $n_D^{20} = 1.42558$; 1,3,4-trimethylcyclohexane, b.p., 139°–140° C., sp. gr. at 20°/4° C., 0.789; $n_D^{20} = 1.4330$.—A. E. D.

Ceresin; Algebraic method for the detection of adulteration of — with paraffin. H. Smelkus. Chem.-Zeit., 1920, 44, 273–275, 286–288.

HOLDE's method (J., 1914, 242) consists in fractional precipitation from chloroform by means of alcohol; the paraffin can be detected by the diminished refractive index of the fraction in which it is concentrated. Equations and a graphical method are developed, for which the original should be consulted.—A. E. D.

Waste products in the petroleum industry; Working up of —. E. W. Albrecht. Chem.-Zeit., 1920, 44, 282.

THE thick acid tar produced in the treatment of lubricating oils with sulphuric acid is washed until free from acid. The residue is mixed with Trinidad asphalt and used for paving. The acid liquor is concentrated. The thin acid tar resulting from the treatment of solar oil with oleum is washed similarly and the acid recovered. The dilute alkali wash always contains emulsified oil. This is recovered by neutralising the alkali.—A. E. D.

Blast-furnace gas. Fowles. See X.

Carbon black. Perrott and Thiessen. See XIII.

Chlorhydrins and glycols from oil-gas. Brooks. See XX.

PATENTS.

Peat; Process for dehydrating —. C. Bouillon, Paris. Eng. Pat. 140,112, 17.1.18. (Appl. 987/18.)

PEAT, preferably finely divided, is treated with a cold dilute solution of calcium chloride or other substance capable of coagulating blood. (Reference is directed to Eng. Pats. 9911 of 1907, and 3367 and 25,385 of 1912; J., 1908, 553; 1913, 277, 1149.)

—A. G.

Peat and the like; Method and apparatus for treating [dehydrating] —. H. H. Hindshaw, Ann Arbor, Mich. U.S. Pat. 1,334,492, 23.3.20. Appl., 3.7.18.

PEAT is conveyed through a chamber, partly cylindrical and partly conical, by means of a screw, which breaks up the peat in the cylindrical part and compresses it in the conical part. The peat then passes to another chamber, where it is subjected to similar treatment, but under a vacuum to remove the air that was released from the air cells by the first treatment.—B. M. V.

Peat dryer. W. S. Jackson, Toronto, Canada. U.S. Pat. 1,334,495, 23.3.20. Appl., 6.5.18.

A HOLLOW shaft carrying radial agitators and a

hollow worm are rotated in a jacketed cylinder, through which the peat is passed. Steam is supplied to the jacket, to the worm, and to the shaft.

—B. M. V.

Peat; Process for treating —. R. Gruhl, Charlottenburg. Ger. Pat. 310,111, 23.8.17.

PEAT is rendered water-resistant by treatment with hydrochloric or nitric acid, chlorine, or acid salts of mineral acids, or by treatment first with chlorine and then with nitric or hydrochloric acid, or with a mixture of these acids. Moulded peat may be treated with dilute nitric acid and then heated, either with or without pressure. The gelatinous constituents of the peat are changed into substances which do not swell in water and are easily washed out; the washed peat may be moulded with or without pressure. After drying the treated peat has completely lost its swelling power and almost completely its water-absorbing power.—B. V. S.

Combustible mass of kieselguhr soaked with spirit. H. von Schütz, Berlin-Wilmersdorf. Ger. Pat. 316,346, 31.10.17.

KIESELGUHR which has been purified by successive washing, centrifuging, extraction with acids, washing, and calcining will absorb up to four times its weight of alcohol.—D. F. T.

Light-coke product and method of making same. Method of utilising fuel. H. Rodman, Edgewood, Pa., Assignor to Rodman Chemical Co., East Pittsburgh, Pa. U.S. Pats. 1,334,404 and 1,334,405, 23.3.20. Appl., 22.6.15.

CRUSHED coking coal is fed on to a heated tumbling mass of separate non-viscous particles, the mass being of sufficient size relatively to the amount of coal fed in to insure a separation of the coal during the coking process, so that the coal is subjected to a tumbling action whilst in the viscous state.

—A. G.

Gases; Production of rich — by means of petroleum residues and apparatus therefor. Soc. de Chimie et Catalyse Industrielles, Paris. Eng. Pat. 140,128, 15.10.18. (Appl. 16,820/18.) Int. Conv., 20.2.18.

PETROLEUM residues are vaporised and the vapours passed through retorts at 500°–600° C., containing a metallic catalyst, e.g., iron or copper. The gases thus obtained are used to enrich blue water-gas or other poor gas, whilst the liquid hydrocarbons which distil up to 150° C. may be used as a motor fuel.—A. G.

Gas-cleaning device. J. Keith and D. B. Bain, London. Eng. Pat. 140,170, 12.2.19. (Appl. 3405/19.)

THE gas (producer-gas, blast-furnace gas, or the like) passes through a casing provided with two revolving paddle wheels in series, on one of which water is caused to play. The tar is thus condensed, and the tar-water mixture is separated from the gas partly by the action of the second paddle-wheel and partly by a centrifugal fan in a separate chamber.—A. G.

Ammonia; Method for recovering — in gasification processes. Bergmann-Elektrizitäts-Werke A.-G., Berlin. Ger. Pat. 298,891, 21.3.16.

A high yield of ammonia is obtained by mixing steam with the air required for combustion, the supply of steam and air being adjusted to suit the nature of the material being gasified and the size and type of gas producer.—W. J. W.

Producer-gas plants; Obtaining by-products from —. J. Pintsch A.-G., Berlin. Ger. Pat. 316,500, 15.9.17.

THE hot producer-gas, after passage through an

apparatus in which it preheats the air supply to the producers, is cooled and scrubbed free from ammonia, the ammoniacal liquor obtained is used as the cooling medium in the coolers for the gas from the producers, and then, while warm, is employed to enrich the air supply with water vapour and ammonia. The air supply, before it is led into the producers, is freed from ammonia, and is completely saturated with water vapour.—L. A. C.

Gasoline; Apparatus for manufacture of —. R. F. Bacon and B. T. Brooks, Pittsburgh, Pa., and C. W. Clark, Chicago, Ill., Assignors to Gulf Refining Co., Pittsburgh, Pa. U.S. Pat. 1,334,731, 23.3.20. Appl., 1.2.15.

AN apparatus for cracking and distilling petroleum oils under pressure consists of a receptacle for the oil and means for heating the oil to the temperature of distillation, for feeding the oil under pressure, and for maintaining the pressure at 60—300 lb. per sq. in. The lower settling zone of the receptacle is in free communication with the upper cracking and distilling zone, so as to remove continuously by gravity the particles of coke and tar produced.—J. W. D.

Gas generators or the like; Apparatus for agitating the fuel in — and means for operating the same. G. H. Bentley and E. G. Appleby, London. Eng. Pat. 140,891, 13.3.19. (Appl. 6336/19.)

Gas-fired furnaces. Eng. Pat. 18,473. See I.

Distillation. Eng. Pat. 140,140. See I.

Ammonium sulphate. Ger. Pats. 299,742 and 299,752. See VII.

Hydrocarbons. Ger. Pats. 309,281—2. See XX.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

PATENTS.

Dry distillation or drying of organic materials [wood, waste cellulose lyes, etc.]; Process and furnace for the —. Aktiebolaget Cellulosa, Stockholm, and G. F. Magnuson, Gelle, Sweden. Ger. Pat. 315,497, 11.1.17. Int. Conv., 1.12.16.

THE gases evolved during drying or dry distillation are utilised to transmit heat from a heating chamber to the material to be treated (organic material either alone or mixed with inorganic material). The heating chamber is arranged between two treatment chambers, and injectors or similar devices are provided to effect the desired circulation of the gases, which pass from above downwards over the material to be treated. A pressure chamber may be arranged above the heating chamber and the treatment chambers, and is provided with suitable openings whereby the hot gases may be forced into the upper part of the treatment chambers.—J. S. G. T.

Vacuum tubes. N. V. Philip's Gloeilampenfabrieken, G. Holst, and E. Costerhuis, Eindhoven, Holland. Eng. Pat. 139,860, 5.2.19. (Appl. 2879/19.)

THE gas in a vacuum tube is renewed automatically or added to by means of a device, comprising a gas reservoir containing two electrodes, or a heating filament or like device for heating the gas; the reservoir is connected to one end of a U-tube containing mercury, the other end being closed. The U-tube is connected to the vacuum tube

through a porous plug in a branch tube, the latter being connected to the U-tube at a point between the ends.—B. N.

Electrical discharge tube. C. E. Green and J. H. Clough, Schenectady, N.Y., Assignors to General Electric Co. U.S. Pat. 1,334,150, 16.3.20. Appl., 11.7.16.

AN electrical discharge device comprises an envelope containing a rare gas, a magnesium cathode, and an anode.—B. N.

Cathode for vapour-filled or gas-filled discharge tubes. W. Gormershausen, Berlin-Schöneberg, and A. Partzsch, Berlin-Karlshorst. Ger. Pat. 315,990, 8.7.17.

THE cathode is constituted of two parts. The one is connected with the source of current, but does not furnish any electronic emission, this being derived from a second metal of lower vaporising temperature placed in the immediate neighbourhood but insulated from the first. The vaporisable metal may be, for example, iron, nickel, copper, or aluminium, and the cathode itself platinum.

—J. S. G. T.

Vacuum tubes, especially Röntgen tubes. C. H. F. Müller, Hamburg. Ger. Pat. 317,358, 5.1.16.

URANIUM is used for the active portion of the incandescent cathode.—A. E. D.

Negative arc-carbons for use in high-power search-lights. Gebr. Siemens and Co., Berlin-Lichtenberg. Ger. Pat. 317,639, 5.8.11.

BREAKAGE of the carbons by shock may be entirely prevented or very much reduced by coating the carbons lightly for some little distance past the contact screws with copper or other metal.

—J. S. G. T.

Distillation; Process of destructive —. C. Turner, Irlam. U.S. Pat. 1,336,261, 6.4.20. Appl., 12.6.18.

SEE Eng. Pat. 117,645 of 1917; J., 1918, 540 A.

Drawing refractory metal. Eng. Pat. 139,065. See X.

III.—TAR AND TAR PRODUCTS.

Benzol in gas etc. Shuttleworth. See IIa.

Hydrocarbons. Tausz and Peter. See IIa.

PATENTS.

Wood tar or the like [lignite or peat tar]; Extraction of higher fatty acids and other organic acids from —. E. Börnstein, Berlin. Ger. Pat. 315,417, 6.9.18. Addition to 314,358.

TAR from lignite, peat, and the like is treated as described in the chief patent (J., 1920, 57 A). The oil obtained by distilling lignite tar at 220°—250° C. with superheated steam is freed from pitch, treated with an alkali carbonate, and washed with water. A portion of the arachidic acid separates as an acid salt and is removed by filtration, neutral oils are extracted from the filtrate by treatment with a light oil, and higher fatty acids are then precipitated by the addition of a mineral acid and distilled at 15 mm. Palmitic, oleic, and arachidic acids distil above 190° C.; the tars mentioned do not yield abietic acid.—L. A. C.

Oil-like products; Process for obtaining neutral —. M. Wendriner, Berlin. Ger. Pat. 316,998, 13.6.14.

THE saturated wash liquors obtained by extracting crude benzene and tar oils with alkalis and sul-

phuric acid respectively are mixed in equivalent proportions. The neutral oily mixture (pyridine and phenols) so obtained may be purified by fractionation, filtration, etc., or the wash liquors may be separately purified before mixing.—B. V. S.

αβ-Dibromotetrahydronaphthalene and Δ¹-dihydronaphthalene; Production of —. J. von Braun. Ger. Pat. 316,218, 27.8.18.

TETRAHYDRONAPHTHALENE is treated with the calculated quantity of bromine at 100°–140° C., and the resulting αβ-dibromotetrahydronaphthalene (m. p. 69° C.) is submitted to the action of magnesium and ether or of tin and alcohol with formation of Δ¹-dihydronaphthalene, b.p. 85° C. at 15 mm., solidif. pt. –8° C.—D. F. T.

Bituminous material [pitch or the like, in filamentary form]; Manufacture of —. H. Wade, London. From The Barrett Co., New York. Eng. Pat. 133,466, 15.10.18. (Appl. 16,847/18.)

Tar soap. Ger. Pat. 316,258. See XII.

IV.—COLOURING MATTERS AND DYES.

Phthaleins and fluorans. M. Copisarow. Chem. Soc. Trans., 1920, 117, 209–218.

IN most cases the formation of phthaleins is accompanied by the production of substances belonging to the o-ketonic acid, hydroxyanthraquinone, and fluoran series. Zinc chloride as a condensing agent is more efficient than concentrated sulphuric acid, giving a 46% yield of phenolphthalein, against 26% with the acid, and 59.3% yield of α-naphtholphthalein, compared with 22.4% with sulphuric acid. The zinc chloride is most efficient when prepared by evaporating to dryness a faintly acid solution of the salt. Hydrogen chloride as a condensing agent favours the formation of hydroxyanthraquinones and phthaleins, but it is not very effective unless used in conjunction with zinc chloride. Boric acid used with sulphuric acid improves the yield of phenolphthalein to 31%, 8% of fluoran and 5% of hydroxyanthraquinones being produced at the same time. o-Cresolphthalein was obtained in 46% yield by the zinc chloride condensation, but p-cresol gave only a 26% yield of 2,7-dimethylfluoran and 2.5% of 4-hydroxy-1-methylantraquinone. Similarly β-naphthol in contradistinction to α-naphthol gave no phthalein, but only a 12% yield of β-naphthylfluoran. These facts provide evidence that the hydroxyl group in the phthaleins takes up the para position to the central C atom of the phthaloyl group.—G. F. M.

PATENT.

Tetrakisazo dyes; Blue —. J. Dedichen, Berlin-Halensee, Germany, Assignor to The Chemical Foundation, Inc. U.S. Pat. 1,319,852, 28.10.19. Appl., 6.11.16.

See Eng. Pat. 101,967 of 1916; J., 1917, 1174.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Nitrocellulose from wood pulp. Woodbridge. See XXII.

Finger-prints. Mitchell. See XXIII.

PATENTS.

Wool and allied materials in the raw state or at any of the various stages of manufacture; Treatment for cleansing [scouring] of —. T. Hollis and A. Woodmansey, Leeds. Eng. Pat. 137,747, 14.6.19. (Appl. 15,007/19.)

THE material is cleansed, either cold or at a tem-

perature not exceeding 90° F. (32° C.), in open machines by means of oil or oils (mineral, fatty, or coal tar oils), e.g., petroleum fractions not lighter than kerosene, olive oil or other similar non-drying oils, re-distilled creosote or residual oils such as anthracene oil or "green" oil, or oils or fatty acids modified by sulphonation, by amidation, by the addition of alkali, of soap, glycerin, or other emulsifying agents. The media may be used separately, or in any desired combination, or in any sequence; water may be present in small quantities according to the media. The media are removed, according to their properties, by means of soluble oils, mild alkalis, or weak soap solutions. Where fibrous material has been treated with soluble oil or soluble oil solutions or with modified oils, it may be passed forward for carding or preparing with a percentage of oil or solution still remaining, which, however, may be easily removed by mild alkaline treatment in the backwater (backwashing) or in later operations of manufacture.—L. L. L.

Lubricant for use in weaving, braiding, rope-making, and the like. L. Minton, Manchester. Eng. Pat. 140,174, 19.2.19. (Appl. 4114/19.)

A MIXTURE of ground mica and powdered aluminium, together with tallow, soap, etc., or with water or oil, is used as a lubricant for the yarn in weaving and similar machines, particularly in the manufacture of lace curtains.—J. F. B.

Fibre from typha seeds; Treatment of — for use in manufacturing textiles. Jata-Werk für pflanzliche Füllstoffe G.m.b.H., Dresden. Ger. Pat. 305,578, 28.9.17.

THE fibres or hairs from typha seeds are hardened and made suitable for spinning by treatment for one or more hours in a 15% solution of sodium chloride boiling under about 1 atm.—L. A. C.

Textile fibres; Method for the preparation of —. Verein der Spiritus-Fabrikanten in Deutschland, Berlin. Ger. Pat. 316,414, 30.11.18.

CERTAIN vegetable juices are applied to the treatment of textile fibres for the removal of the substances which are susceptible to decomposition by enzymes. Of the rhizomes, roots, and tubers of value for the above purpose the potato is typical; typha fibres, for example, may be submitted to the amylolytic and proteolytic action of the juice of the potato at 40°–50° C.—D. F. T.

Textile fibres from Malva crispa and Lavatera trimestris. A. Hammer, Berlin. Ger. Pats. (A) 316,951 and (B) 316,952, 3.9.18.

(A) THE roots of *Malva crispa* are subjected to a retting process, and the fibres are stripped by breaking the woody portions. The root fibres are finer and softer than the stem fibres. (B) The bast of *Lavatera trimestris* or *Malope grandiflora* is treated by the cold or hot water retting system or by boiling and subsequent breaking or spraying with jets of water, whereby a long flax-like fibre is obtained. By further resolution by means of chemicals, a fibre having more of the character of cotton is produced.—J. F. B.

Mulberry bark; Manufacture of fine spinning fibres from —. G. Mark, Bretnig. Ger. Pat. 317,043, 15.9.18.

FIBRES isolated in the usual manner are caused to shrink and become curly by the action of strong alkalis. The product is pliable and very fine, and is easily spun by the methods employed for cotton or wool spinning.—J. F. B.

Soda-cellulose, particularly wood-cellulose; Process of producing —. E. L. Rinman, Djursholm, Sweden. U.S. Pat. 1,319,360, 21.10.19. Appl., 23.5.18.

THE raw material is boiled with caustic soda lye

(60 g. per litre) in presence of a contact substance, which is insoluble in caustic soda lye and furthers reduction, e.g., mercury.

Cymene obtained in sulphite-cellulose process; Method of recovering —. L. Akerblom, Stockholm. U.S. Pat. 1,333,694, 16.3.20. Appl., 11.2.18.

THE gases from the cellulose digester are led into a receptacle containing acid for a subsequent digestion; the greater portion of this acid is then tapped off from the lower end of the receptacle and the remainder, together with the cymene floating on the liquor, is conducted into a second receptacle. The level of liquid in the latter is then raised by introducing a liquid heavier than cymene, so that the cymene is forced up into a narrow portion of the receptacle from which it is drawn off.—J. F. B.

Adhesive for articles made of cellulose derivatives. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 317,412, 26.10.17.

THE dimethyl, diethyl, or β -methyl ester of adipic acid may be employed as an adhesive for articles made of cellulose derivatives.—L. A. C.

Counter-board and process of making same. A. L. Clapp, Marblehead, Mass., Assignor to The Metalite Co., Amesbury, Mass. U.S. Pat. 1,332,541, 2.3.20. Appl., 28.6.17.

FIBROUS cellulose is treated with alkali without the application of heat; the stock thus obtained is formed into sheets, and the sheets are dried.

—J. F. B.

Paper; Process of manufacturing —. A. Imaoka, Tokyo, Japan. U.S. Pat. 1,333,255, 9.3.20. Appl., 9.7.19.

THE outer bark of abaca is boiled in a solution of caustic alkali, whereby the non-fibrous part of the bark is caused to gelatinise, and the gelatin-like substance thus obtained is caused to cover the fibres for the manufacture of paper.—J. F. B.

Paper, boards, and paper textiles; Process for sizing and waterproofing —. P. E. Altmann, Dresden-Tolkewitz. Ger. Pat. 304,205, 2.8.17.

THE material is impregnated with a mixture of saponified beeswax, water-soluble oils, and talc, and the sizing agent is fixed by means of alum solution.

—J. F. B.

Emulsions for sizing paper; Manufacture of —. G. Muth, Nuremberg. Ger. Pat. 316,345, 27.6.16.

COUMARONE resin, or a similar coal-tar resin, is emulsified with an aqueous solution of a vegetable or animal glue or a similar colloid, and a solution of an aluminium salt is added; casein may be employed as the colloid if it is first converted to the water-soluble form.—L. A. C.

Impregnating paper and the like; Machines for —. C. W. Mayer, Rochester, N.Y., U.S.A. Eng. Pat. 140,861, 6.1.19. (Appl. 399/19.)

Paper felt; Manufacture of — for roofing or the like. H. Wade, London. From The Barrett Co., New York. Eng. Pat. 127,994, 12.6.18. (Appl. 9693/18.)

SEE U.S. Pat. 1,305,404 of 1919; J. 1919, 572 A.

Distillation or drying. Ger. Pat. 315,497. See IIa.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Bleaching apparatus and the like. J. R. Fish, Bolton. Eng. Pat. 137,947, 5.3.19. (Appl. 5429/19.)

MECHANISM outside and inside the kier is provided

to give to a hollow plaiting machine arm, at the extremities of which are rollers, a rotation about an axis for spreading the cloth, and a reciprocating motion so as to cause it to plait the fabric radially. The rollers at the ends of the plaiting machine arms draw the fabric into the kier. By means of a differential gear the combined motions are made to take place simultaneously.—L. L. L.

Dyeing machine; Automatic yarn hank —. Textilmaschinen Fabrik A.-G., Zurich, Switzerland. Eng. Pat. 128,586, 20.6.19. (Appl. 15,611/19.) Int. Conv., 20.6.18.

TWO connected frames are movable intermittently, simultaneously, and in the same direction along the side walls of the dyeing trough, which are toothed on their upper edges. Forks are provided for lifting the hank-carrying rods from the toothed side walls and for carrying them through the trough. The apparatus is practically free from machine parts above the dyeing trough, and consequently the injurious effect of the vapours from the bath is reduced to a minimum.—L. L. L.

Dyeing or similarly treating yarns in the form of hanks, skeins or bundles; Machines for —. A. Ashworth, Manchester. Eng. Pat. 137,980, 1.5.19. (Appl. 10,822/19.)

AN improved form of the machine described in Eng. Pat. 113,575 (this J., 1918, 204 A) comprises a single rectangular tank without any central compartment or division. The dye liquor is pumped directly into the tank, and passes through a wire gauze screen to ensure even distribution. The skeins or bundles of material are laid full length across the tank in the direction of the flow of liquor. The dry yarn is compressed by a lid fitting the tank with a sliding fit and having its upper edges bevelled, to receive a rope of cotton or other packing upon which an angle iron frame is clamped.—L. L. L.

Fibre-treating machine. H. M. Dudley, Philadelphia, Pa. U.S. Pat. 1,328,987, 27.1.20. Appl., 4.12.18.

A FIBRE chamber is provided with removable foraminous bottom and top plates, and with a spider follower, which abuts against the top plate and moves it with respect to the bottom plate. Means are provided for forcing liquid in either direction through the device.—L. L. L.

Dyeing machine. Hosiery-dyeing machine. Fabric-treating device. Textile-treating device. H. M. Dudley, Philadelphia, Pa. U.S. Pats. (A) 1,334,526, (B) 1,334,527, (C) 1,334,528, and (D) 1,334,529, 23.3.20. Appl., (A) 20.11.17, (B) 5.6.18, (C) 22.1.19, and (D) 25.2.19. (Cf. J., 1920, 228 A.)

(A) THE machine consists of a receptacle in which are hollow perforated spindles on which fibres may be wound. The fibres can be compressed on the spindles by means of removable bars, and means are provided for forcing a liquid through the spindles and so through the fibre, through the fibre into the hollow spindles, and through the whole machine. (B) A cylindrical machine contains a series of reels placed end to end and formed by a perforated spindle on which are spaced perforated discs. The fibres are wound on these reels and covered with perforated plates. A liquid can be forced through the spindle and fibre in either direction. (C) The fabrics to be treated are contained in a chamber which can be revolved at varying rates. A liquid can be forced through the fabric chamber. (D) The fibre is wound on beams in a dyeing machine. Means are provided for passing a liquid through the series of beams in outward and longitudinal directions simultaneously.—A. J. H.

Fibrous material; Method of colouring —. C. H. Dennison, Wollaston, Mass., Assignor to American Rubber Co. U.S. Pat. 1,332,974, 9.3.20. Appl., 10.8.16.

THE fibrous material is subjected to the action of a solution of cadmium chloride and of a polysulphide of an alkaline-earth metal, and is finally treated with a vulcanisable plastic compound.

—J. F. B.

Fibrous material; Method of colouring —. W. A. Gibbons, Flushing, N.Y., Assignor to American Rubber Co. U.S. Pat. 1,332,982, 9.3.20. Appl., 10.8.16.

FIBROUS material is coloured and rubberised by dipping it in a solution of potassium antimony tartrate and a solution of ammonium sulphide, whereby antimony sulphide is formed directly in or upon the fibres, and the material is then coated with a vulcanisable plastic compound.—J. F. B.

Dyes; Means of fastening — in material and waterproofing same. H. Sharp, Leeds. Eng. Pat. 140,222, 3.4.19. (Appl. 8360/19.)

DYED fabrics are treated successively with solutions of alum, of paraffin wax, rosin, and curd soap, and of Epsom salts (magnesium sulphate).

—A. J. H.

Textile fabrics; Finishing or lustring —. J. B. Lomax, Furness Vale. Eng. Pat. 137,710, 15.4.19. (Appl. 9532/19.)

THE fabrics are passed through a Schreiner calendaring machine under much heavier pressure and at a higher temperature than is usually employed, the Schreiner calender roll being heated to a blue heat or to as high a temperature as the cloth will withstand without injury. Whilst still hot and under tension the fabric is treated on one or both sides with a finishing liquor, composed preferably of a boiling solution of alizarin oil. The fabric is wound on rollers and left in the wet state for 10–12 hours, and then stretched and stentered.

—L. L. L.

Finishing fabrics. J. H. Wrigley, Manchester, and A. B. Henshilwood, Bradford. Eng. Pat. 137,948, 6.3.19. (Appl. 5538/19.)

SHEET rubber or rubber-covered fabric or other elastic material is wound together with the fabric to be treated, so that it forms a layer between adjacent coils of the fabric. When such a roll is treated in a beetling machine, although the more prominent fibres, or surfaces, receive the bulk of the pressure, this is transmitted by the elastic material to the less prominent surfaces, and a more uniform finish is obtained without undesirable "watered" or "moirée" effects.—L. L. L.

Beetling and treatment of fabrics. J. D. Lumsden, W. R. Mackenzie, E. H. Robinson, and M. Fort, Almondbank, Perthshire. Eng. Pat. 137,968, 5.4.19. (Appl. 8616/19.)

THE beetling properties of cotton, linen, union, or other fabrics composed mainly or wholly of vegetable fibres are improved by removing oils, fats, waxes, or resinous products from the fabrics. For this purpose the material is subjected to a dry-cleaning treatment, or a treatment with a volatile solvent. In the case of goods to be bleached the solvent treatment may precede bleaching, thereby ensuring good beetling properties irrespective of the bleaching treatment accorded. This process is particularly useful when applied to the beetle-finishing of coloured bordered goods, and linen and cotton goods generally containing threads which are also often required to withstand bleaching.

—L. L. L.

Bleaching-boiler plant. C. Tütsch, Winterthur, Switzerland. U.S. Pat. 1,335,925, 6.4.20. Appl., 21.9.18.

SEE Ger. Pat. 309,974 of 1917; J., 1919, 497 A.

Dyeing, bleaching, or like treatment of cops; Apparatus for —. W. Resch, Binningen, Assignor to A. Clavel, Basle, Switzerland. U.S. Pat. 1,335,459, 30.3.20. Appl., 28.6.19.

SEE Eng. Pat. 117,630 of 1918; J., 1919, 71 A.

Emulsifying liquids. Ger. Pat. 314,017. See XII.

Washing and cleaning. Ger. Pat. 317,402. See XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Ammonia; Catalytic synthesis of —. M. Guichard, Vavon, Cornec, Cantagrel, Stevenson, Apard, and Bourdiol. Bull. Soc. d'Encour., 1920, 132, 71–102.

THE authors have specially studied the characteristics of various catalysts, chiefly the non-alkali metals, which do not demand absolute dryness of the gaseous mixture. The life of catalysts such as iron, cobalt, nickel, tungsten, and molybdenum is greatly extended by admixture with refractory substances, such as magnesia and alumina, and greater activity is secured by using a combination of two metals. Of 400 catalysts examined iron-molybdenum appears to be the most resistant, and to give the highest concentration of ammonia, viz., more than 4% as compared with:—iron, less than 1%; molybdenum, less than 1.5%; nickel, 3%; and cobalt, 3%. The method of preparation is important, and the most satisfactory results are obtained by precipitation of ferric nitrate with ammonium molybdate, ignition, and subsequent reduction. In absence of moisture, uranium gives good results, but its combinations, uranium-cobalt and uranium-molybdenum, are inferior, whilst uranium-nickel is unsatisfactory, and uranium-tungsten almost entirely inactive. The effect of catalytic poisons is briefly discussed. A partial study has been made of the cycle of operations, involving circulation of the compressed gases, heat recovery, absorption of ammonia under pressure, and best arrangement of the furnace.—W. J. W.

Nitrogen and hydrogen mixture; Preparation of — by decomposition of ammonia. R. O. E. Davis and L. B. Olmstead. J. Ind. Eng. Chem., 1920, 12, 316–317.

ANHYDROUS ammonia was decomposed into a mixture of hydrogen and nitrogen ($3H_2:N_2$) by passing it through an electrically-heated tube furnace containing iron turnings or steel wool as catalyst, and subsequently absorbing the undecomposed ammonia. The ammonia remaining in the scrubbed gases rarely exceeded 0.2%. For ordinary operations a yield of 100 cb. ft. per hour of the gaseous mixture was obtained, the temperature being maintained at 675° C. The average period of contact with the catalyst was about 5 secs. When copper is used as catalyst a temperature about 200° C. higher and longer contact are required. (Cf. J. C. S., June.) —C. A. M.

Ammonia and formates; Production of — from cyanides, ferrocyanides, and cyanised briquettes. G. W. Heise and H. E. Foote. J. Ind. Eng. Chem., 1920, 12, 331–336.

IN the hydrolysis of cyanides in an autoclave under varying pressures good yields of ammonia were obtained by steaming under a pressure of 50 lb., and a quantitative yield at 200 lb. pressure. Ferro-

cyanides were hydrolysed more slowly, a maximum yield of 46% being obtained after steaming for 44 hours at a pressure of 300–330 lb. In the case of cyanised briquettes yields of over 90% were obtained by steaming for 30 to 45 mins. at a pressure of 300–330 lb. A temperature of 600° C. was required to obtain good results with steam at atmospheric pressure, whilst with steam at 100 lb. pressure a temperature of 400° C. was satisfactory.

—C. A. M.

Electrolysis of mixed solutions of alkali salts. Phenomena of electrolytic migration. M. H. van Laer. *Rec. Trav. Chim.*, 1920, 39, 301–329.

In the electrolysis of a solution of sodium hydroxide in a diaphragm cell the current yield is small and variable. The electrolysis of a solution of sodium carbonate presents the same fundamental characteristics, and in this case as the electrolysis proceeds the resistance of the cell increases owing to the formation of sodium bicarbonate. The introduction into the solution of an oxy salt of sodium such as the chlorate, sulphate, or nitrate prevents the formation of the bicarbonate, and consequently a cathode liquid very rich in sodium hydroxide can be obtained. The nitrate gives the best result, and the current yield increases with the concentration of the oxy salt. Using this same method with a saturated solution of barium nitrate holding barium carbonate in suspension as anolyte, and a rotating anode, crystals of barium hydroxide were ultimately obtained from the cathode liquid, which at the start was a weak solution of the hydroxide. (*Cf.* J. C. S., ii., 282.)—W. G.

Aluminates; Hydrolysis of alkali — and methods of determining the concentration of hydroxyl ions in concentrated solutions of alkali hydride. R. Fricke. *Z. Elektrochem.*, 1920, 26, 129–151.

THE hydrolysis of alkali aluminates is a time reaction. So-called crystalline alumina is not really crystalline; its properties vary with the age of the product. The ageing of alumina is proportional to the hydroxyl ion concentration of the alkaline solution in equilibrium with it. Aged solutions of alkali aluminates contain considerable quantities of colloidal alumina. The hydroxyl ion concentration of concentrated alkali solutions can be estimated by an electrometric method. (*Cf.* J. C. S., June.)

—J. F. S.

Sulphuric ions; Detection of masked — in complex compounds. P. Job and G. Urbain. *Comptes rend.*, 1920, 170, 843–845.

In complex cobalt compounds the total SO_4 ion present is estimated by precipitation in the usual way as barium sulphate and the free SO_4 ion by precipitation in the cold as benzidine sulphate. The difference gives the masked SO_4 ion present in the complex compound.—W. G.

Hypoiodous acid; Estimation and kinetics of — in acid solution. V. Cofman. *Bull. Soc. Chim.*, 1920, 27, 234–239.

THE fact that hypoiodous acid, but neither free iodine nor any other iodine compound, gives stable iodo compounds almost instantaneously with phenols forms the basis of the following method for the estimation of this acid. To a measured volume of the solution under examination potassium iodide is added, the solution is acidified, if necessary, and titrated with standard thiosulphate. The operation is then repeated, but this time an excess of phenol is added before the potassium iodide. The difference in the amount of iodine found by the two titrations is a measure of the amount of hypoiodous acid present. (*Cf.* J. C. S., ii., 329.)—W. G.

Boric acid; Acidimetric estimation of —. J. A. M. van Liempt. *Rec. Trav. Chim.*, 1920, 39, 358–370.

If a suitable excess of mannitol, glycerol, or hævulose is added to the solution, boric acid may be estimated electrometrically by measuring the hydrogen-ion concentration of the solution after successive additions of standard alkali.—W. G.

Ferrates; Influence of superimposed alternating current on the anodic formation of —. G. Grube and H. Gmelin. *Z. Elektrochem.*, 1920, 26, 153–161.

CONCENTRATED solutions of sodium ferrate may be obtained by electrolysis of a 40% solution of sodium hydroxide at 35° C. in a divided cell using a platinum cathode and an iron anode. The current to be employed is 3.33 amp./sq. dm. direct current with 5.0 amp./sq. dm. alternating current superimposed. (*Cf.* J. C. S., June.)—J. F. S.

Gases; Action of finely divided —. C. Zengheis. *Comptes rend.*, 1920, 170, 883–885.

By passing hydrogen, generated in an external vessel, through filter paper or parchment (as used for dialysers) into solutions of mercuric chloride, potassium chlorate, etc., the author has obtained noticeable reduction of these salts. Similarly carbon dioxide in aqueous solution was reduced to formaldehyde and trioxymethylene. Under somewhat similar conditions certain oxidations can be effected, and nitrogen can be caused to combine with hydrogen in the cold. The author considers that the reactions brought about by gases in their nascent or occluded state are not necessarily due to any excess of free energy possessed by free atoms, but simply to the extremely finely divided state of the gases.—W. G.

Hydrogen; Sorption of — by palladium at low temperatures. J. B. Firth. *Chem. Soc. Trans.*, 1920, 117, 171–183.

AT the temperature of liquid air “active” palladium is “inactive” so far as absorption of hydrogen is concerned, and immersion in liquid air tends to retard the activity when the temperature is allowed to rise once again to the normal. Similar effects were produced after cooling at 0° C. At these low temperatures, however, there is rapid occlusion or “adsorption” of hydrogen. The limiting temperature for absorption or diffusion into the interior of the metal is probably about 0° C.; below this temperature adsorption only occurs, from 0°–150° C. adsorption followed by absorption, and above 150° C. absorption only.—G. F. M.

Absorptive power of sugar charcoal; Effect of heating on the — for sulphur dioxide. R. M. Winter and H. B. Baker. *Chem. Soc. Trans.*, 1920, 117, 319–320.

THE specific gravity of purified sugar charcoal was increased from 1.76 to 1.81 by heating to 900° C. in a muffle furnace for 40 hours, and at the same time the amount of sulphur dioxide capable of being absorbed by 1 grm. increased from 97 c.c. to 288 c.c. —G. F. M.

Decomposition of sodium amalgam. Müller and Riedel. *See X.*

Ammonia recovery. Donath. *See XVII.*

Nitrogen in nitrates. Beckett. *See XXII*

PATENTS.

Acid plant. E. L. Larison, Anaconda, Mont. U.S. Pat. 1,334,381, 23.3.20. Appl., 10.2.19.

A CHAMBER for use in the manufacture of sulphuric acid consists of interior masonry walls and a lead

sheathing supported by the walls and by a framework, the members of which are also supported by the walls.—A. R. P.

Acetic acid of high concentration; Production of — from gases containing acetic acid vapour. Konsortium für elektrochem. Ind., G.m.b.H., Munich. Ger. Pat. 305,125, 29.6.16.

Gas containing acetic acid vapour is scrubbed in a tower, or the like, with a counter-current of water, which is fed in at such a rate that the whole or the greater part leaves the apparatus as vapour together with the gas.—L. A. C.

Nitric acid; Preparation of — from hot nitrous gases. Chem. Fabr. Griesheim-Elektron, Frankfurt. Ger. Pat. 304,322, 11.8.16.

Hot nitrous gases are passed through one or more towers supplied with dilute nitric acid from succeeding absorption towers so as to evaporate part of the water and increase the concentration of the acid to 67%.—A. B. S.

Hydrochloric acid and magnesia; Preparation of —. P. Beck, Nordhausen. Ger. Pat. 304,342, 7.8.17.

MAGNESIUM chloride or oxychloride is mixed with 2–5% of magnesium sulphate previously dissolved in water, the mixture formed into balls or briquettes and heated in the presence of water or steam. The production of magnesia dust is thereby prevented, and the yield of hydrochloric acid is greater than when more magnesium sulphate is present.

—A. B. S.

Sodium sulphate; Process of making —. Salzwirk Heilbrunn, A.-G., Heilbronn, Germany. Eng. Pat. 134,214, 20.10.19. (Appl. 25,676/19.) Int. Conv., 24.1.18.

A MIXTURE of sodium bisulphate and sodium chloride is heated to a temperature sufficiently high to yield molten sodium sulphate. Impurities are removed by passage of air or steam through the molten mass and/or the addition of an alkali, such as lime, baryta, or sodium carbonate. A finely divided product may be obtained by cooling with agitation, and crystalline sodium sulphate may be prepared by addition of the requisite amount of water. The hydrochloric acid evolved is freed from traces of hydrogen sulphide and sulphurous and sulphuric acids by passage over sodium chloride at a temperature slightly higher than that of the charge; the preheated salt is then employed for recharging the furnace.—L. A. C.

Phosphorus and phosphoric acid; Manufacture of —. W. H. Waggaman, Washington, D.C. U.S. Pat. 1,334,474, 23.3.20. Appl., 1.8.19.

BRIQUETTES prepared by compressing a finely ground mixture of natural phosphates, silica, a solid fuel, and a binder, are heated in a furnace to drive off phosphorus and phosphoric acid; flames and hot gases are then directed on to the surface of the molten mass, whereby further quantities of phosphorus and phosphoric acid are obtained.

—L. A. C.

Thorium; Process of separating — from other metals of the rare earths. O. Dietsche, Berlin-Lichtenberg, Germany, Assignor to The Chemical Foundation, Inc. U.S. Pat. 1,335,157, 30.3.20. Appl., 15.4.15.

THORIUM is separated from the other metals of the rare earths by treating a solution of these metals with a soluble fluorine compound corresponding to the amount of thorium present.—A. B. S.

Ammonium nitrate; Manufacture of —. Berlin-Anhaltische Maschinenbau-A.-G., Berlin. Ger. Pat. 299,602, 18.9.15.

NITRIC acid is diluted with a cold saturated solu-

tion of ammonium nitrate, and during the neutralisation of the acid with ammonia gas the solution is cooled by constantly circulating a portion through a cooler.—L. A. C.

Ammonium sulphate; Partial replacement of sulphuric acid by a bisulphate solution in the manufacture of —. Berlin-Anhaltische Maschinenbau-A.-G., Berlin. Ger. Pat. 299,742, 16.7.15.

A SOLUTION for the partial replacement of sulphuric acid in the manufacture of ammonium sulphate is prepared by dissolving a bisulphate in the liquor condensed from the gas both before and during the hot separation of tar.—L. A. C.

Ammonium sulphate; Manufacture of — from gas liquor and gypsum. Chem. Ind.-A.-G., and F. Wolf, Bochum. Ger. Pat. 299,752, 20.5.16.

A MIXTURE of gas liquor and gypsum is treated with carbon dioxide under pressure, and the solution of ammonium sulphate obtained is filtered and freed from hydrogen sulphide by heating and passage over hog iron ore.—L. A. C.

Ammonium sulphate; Manufacture of — from calcium sulphate and ammonium carbonate. Badische Anilin- und Soda-Fabrik, Ludwigshafen. Ger. Pat. 300,724, 30.7.16.

THE crude gypsum is calcined before treatment with ammonium carbonate to facilitate filtration of the ammonium sulphate solution from the lime sludge.—L. A. C.

Magnesia; Production of — for artificial stones and ceramic purposes. K. Schmid, Altmügeln. Ger. Pat. 303,870, 7.7.15.

MAGNESIA for use in the manufacture of artificial stone and for ceramic purposes is prepared by heating ammonium magnesium carbonate, obtained by adding ammonium carbonate to the waste liquor of the potash industry. The recovery of the ammonium salt from the mother liquor makes the process remunerative.—A. B. S.

Hydrogen sulphide; Preparation of — from sulphates. Apparate-Vertriebs Ges.m.b.H., Berlin-Wilmersdorf. Ger. Pat. 304,231, 14.7.16.

SULPHATES are reduced by means of hydrocarbons, e.g., natural gas or the like, at 840°–1050° C., and hydrogen sulphide is liberated from the sulphides formed by means of gases containing carbon dioxide.—A. B. S.

Hydrogen sulphide; Preparation of — from calcium sulphate. P. Beck, Nordhausen. Ger. Pat. 305,103, 9.8.17.

CALCIUM sulphate is converted into calcium sulphhydrate by treating it with barium hydrosulphide, the resulting barium sulphate being reduced to sulphide and the latter converted into the hydrosulphide.—A. B. S.

Potassium chloride; Extraction of — from carnallite by the cold process. Kaliwerke Grossherzog von Sachsen A.-G., Dietlas, and K. Hepke, Dorndorf. Ger. Pat. 316,215, 17.11.18.

As an improvement in the cold process of extracting potassium chloride from carnallite whereby the quantity of mother liquor employed is much reduced, a portion of the potassium chloride emulsion is withdrawn from the extraction apparatus, at a point situated between the inlet for the carnallite and the inlet for the extraction liquor, where the maximum concentration of magnesium chloride is not attained and where the liquor is sufficiently mobile to allow of the separation of the kieserite by gravity.—L. A. C.

Iron; Recovery of — from liquors containing ferric salts. H. Frischer, Zehlendorf. Ger. Pat. 317,633, 26.3.18.

IRON is removed from liquors containing ferric salts in solution by reducing the iron to the ferrous state and afterwards re-oxidising it to a basic ferric salt. Thus, an alum liquor may be treated first with barium sulphide (which forms ferrous sulphate) and then with barium peroxide, which forms basic ferric sulphate, $\text{Fe}_2(\text{OH})_2\text{SO}_4$. The reduction may also be effected by scrap iron or aluminium and the oxidation with sodium peroxide.—A. B. S.

Copper sulphate and hydrogen; Preparation of — by electrolysis. S. Simonek, Budapest, and P. Mike, Gödöllő, Hungary. Ger. Pat. 317,691, 2.12.16.

COPPER sulphate and hydrogen are produced by passing an electric current through dilute sulphuric acid in a cell divided into two parts by a diaphragm, using a copper anode.—A. B. S.

Sulphur retort. W. E. Clifford, Salt Lake City, Utah, and T. H. Green, Springfield, Ill., Assignors to United Filters Corp., New York. U.S. Pat. 1,332,512, 2.3.20. Appl., 25.1.19.

THE retort is formed in two sections, one being movable relatively to the other. A driving shaft is journaled in the fixed section and a rotary cage for the sulphur-bearing material is carried by the movable section. The cage telescopes within the fixed section of the retort as the movable section is moved toward and away from it. Co-operating clutches on the driving shaft and cage respectively are automatically brought into and out of operation as the cage is telescoped in and out of the fixed retort section.—J. F. B.

Sulphur; Production of — from calcium sulphate. Badische Anilin- u. Soda-Fabrik, Ludwigshafen. Ger. Pat. 302,433, 21.9.16.

A MIXTURE of calcium sulphate and fuel, with or without silicious or argillaceous matter to form a thin slag, is heated in a shaft furnace with a limited amount of air, so as to liberate the sulphur, which is removed from the furnace gases by washing with water. The slag may be used as a hydraulic cement.—A. B. S.

Sulphur; Purification of —. J. J. Hood, London. Eng. Pat. 140,844, 27.11.18. (Appl. 19,585/18.)

SEE U.S. Pats. 1,331,645-6 of 1920; J., 1920, 295 A. Spent oxide may be leached with a solvent of sulphur and the solution purified by the process described.

Sulphuric acid containing nitrogen oxides [; Treatment of —]. Norsk Hydro-Elektrisk Kvaestofaktieselskab, Christiania, Norway. Eng. Pat. 130,968, 13.6.19. (Appl. 14,958/19.) Int. Conv., 9.8.18.

SEE U.S. Pat. 1,319,586 of 1919; J., 1919, 945 A.

Bichromates; Process for transforming alkaline monochromates into —. Soc. Ind. de Prod. Chim., Paris. Eng. Pat. 122,172, 23.12.18. (Appl. 21,633/18.) Int. Conv., 5.1.18.

SEE U.S. Pat. 1,326,123 of 1919; J., 1920, 190 A.

Ammonia; Catalytic oxidation of —. M. Taliani, Isola Liri, Italy. Eng. Pat. 140,833, 8.4.18. (Appl. 5979/18.)

SEE U.S. Pat. 1,270,989 of 1918; J., 1918, 580 A.

Pyrites furnace. E. C. Bracq, Paris. U.S. Pat. 1,335,271, 30.3.20. Appl., 9.11.18.

SEE Eng. Pat. 118,094 of 1918; J., 1919, 252 A.

Ammonia recovery. Ger. Pat. 298,891. See IIA.

Potassium compounds. Eng. Pat. 131,920. See IX.

Alkali-bearing rocks. Ger. Pat. 315,461. See IX.

Purifying zinc solutions. Eng. Pats. 138,946-8, 138,950, and 138,954. See X.

Oxygen and hydrogen. U.S. Pat. 1,331,668. See XI.

Electric furnace. Ger. Pat. 316,319. See XI.

Electrochemical reactions. Ger. Pat. 317,502. See XI.

VIII.—GLASS; CERAMICS.

Sillimanite refractories; The Malinile process for the production of —. A. Malinovsky. J. Amer. Ceram. Soc., 1920, 3, 40-68.

THE complete conversion of clay into crystalline sillimanite cannot be effected in a firebrick kiln, but is readily accomplished in a blast or cupola furnace in the presence of coke. The excess of silica in the clay is volatilised, probably after being reduced first to silicon carbide and then to silicon, which ignites spontaneously with great evolution of heat. Iron, if present, acts as a catalyst, effecting the more complete reduction of the silica and forming some ferro-silicon, which sinks to the bottom of the furnace. Titanium is desirable, as it restrains the combination of iron with alumina and allows it to act wholly on the silica. A little iron is carried out by the blast as magnetite, but most of the iron remains in the metallic state. If cryolite is present the volatilisation of the silica is very rapid, and the alumina combines with some of the silica. Lime retards the formation of sillimanite. Felspar and mica facilitate the conversion into sillimanite above 1300° C. by dissolving the clay, sillimanite crystallising out of the fused solution. After heating for about three hours the contents of the cupola begin to boil, and the silica volatilises in dense bluish and finally brown fumes for about 2 hours, after which the process is completed, but the air blast is continued so as to produce a porous mass of sp. gr. 2.62-2.86, which is allowed to cool, then crushed, and the iron removed magnetically. The product is crude sillimanite melting at cone 36-40 (1790°-1920° C.); pure sillimanite melts at 1815° C. The product is highly refractory, not sensitive to sudden changes in temperature, and is not affected by acids, even hydrofluoric acid. It consists chiefly of intertwined crystals of sillimanite, but may contain a little amorphous sillimanite, glass (apparently also sillimanite), and slag. The crystals vary in length from 0.01 to 0.50 mm.—A. B. S.

Refractories; Physical characteristics of specialised —. (A) M. L. Hartmann and O. A. Hougen. (B) M. L. Hartmann and J. F. Kohler. Trans. Amer. Electrochem. Soc., 1920, 349-354, 355-358. [Advance copies.]

(A) *Spalling losses.*—Five to ten bricks of the same kind were dried, weighed, and used as "headers" in the front wall of an oil-fired furnace, where they were heated to 1350° C. for one hour. They were then removed and the heated end exposed for 15 mins. to a blast of cold air from a nozzle 2 in. diameter, delivering 27 cu. ft. per min. The operation was repeated ten times, and the percentage loss of weight was regarded as the spalling loss. The least spalling (0-12%) occurred with carborundum bricks and grade A fireclay; 43-65% with bauxite, zirconia, and grades B and C fireclays, whilst

chrome, silica, and magnesia bricks were completely destroyed with less than ten treatments. All the bricks cracked during the test. (b) Abrasion tests were made by cutting a groove in the ends of each brick so as to expose the maximum area to cutting and then applying the bricks at a constant pressure of 25 lb. for 5 mins. to a carborundum grinding wheel. The bricks were tested when cold and also immediately after heating to 1350° C. The variations in bricks of the same kind and even from the same manufacturer were large. The relative resistance to abrasion of the cold bricks is shown by the following figures, which represent the difference in the depth of the groove before and after the test: carborundum, zirconia, and bauxite, 0.01–0.2 in.; grade C fireclay, magnesia, and chrome bricks, 0.05–0.07 in.; silica bricks, 0.17 in.; grades A and B fireclay, 0.25–0.26 in. The corresponding figures for bricks at 1350° C. are: carborundum and bauxite, 0.1–0.4 in.; zirconia, recrystallised carborundum, and grade C fireclay, 0.06–0.07 in.; grades A and B fireclay, 0.09–0.11 in.; chrome, 0.27 in.; magnesia, 1.25 in.; the silica brick spalled too badly to permit any abrasion test. —A. B. S.

Pottery; The use of American raw materials in the manufacture of white ware —. A. V. Bleininger and W. W. McDanel. J. Amer. Ceram. Soc., 1920, 3, 134–148.

SATISFACTORY white pottery can be made from raw materials of American origin.

Silicious sagger mixtures. M. R. Hornung. J. Amer. Ceram. Soc., 1920, 3, 69–76.

IN making saggars, chert, especially if it has been calcined, is more satisfactory than quartzite. To produce a sagger which neither shrinks nor expands about 60–65% of raw chert or quartzite or their equivalent must be present. Finely ground silicious materials are preferable to coarser ones. The addition of calcined flint-clay reduces the tendency to spall. A mixture of raw clay 50, calcined flint clay 20, and chert 30% is recommended. —A. B. S.

Terra-cotta glazes; Development of —. E. C. Hill. J. Amer. Ceram. Soc., 1920, 3, 13–25.

THE effect of adding tin oxide or barium or magnesium carbonate to a Bristol or terra-cotta glaze containing felspar, flint, clay, whiting, and zinc oxide was investigated. Tin oxide increases the whiteness of a glaze, but if more than 7% is added it tends to produce crawling and cracks. The fusibility of the glaze is reduced in proportion to the tin oxide or barium carbonate added. Magnesium carbonate if present in the proportion of more than 0.20 equivalent of MgO tends to cause crawling and cracks. Addition of magnesia makes the glaze more refractory. A decrease in the proportion of clay present usually increases the fusibility of the glaze, but decreases the gloss in glazes high in clay. Addition of clay increases the fusibility only when there is at least 0.40 equivalent of CaO present. —A. B. S.

Tin oxide in glazes; Substitutes for —. R. H. Minton. J. Amer. Ceram. Soc., 1920, 3, 6–12.

IN leadless terra-cotta and porcelain glazes the presence of zinc oxide is useful. Tin oxide and its substitutes and barium oxide increase opacity more than clay. In a terra-cotta glaze the presence of both barium and zinc oxides improves opacity. Leukonin (sodium metantimonate) and antimony oxide, where they can be used, are almost as efficient as tin oxide in producing opacity, but zirconium oxide (terrar) appears to be the best substitute for tin oxide when it can be obtained at a sufficiently low price. Alumina produces matt glazes. —A. B. S.

PATENTS.

Double-walled glass vessel on the Dewar system and process for manufacturing the same. Isola Ges. für Wärme und Kälte-Isolierung m.b.H., Berlin. Eng. Pat. 137,541, 9.1.20. (Appl. 744/20.) Int. Conv., 13.10.15.

DEWAR vessels with walls 2–5 mm. thick and up to 5–10 litres capacity may be made if the vessels, as soon as they are formed, are placed in a casing of metal, asbestos, or other substance, previously heated to a high temperature (400°–500° C.), and the casing and its contents placed in a hot oven, heated to 700°–800° C., and then cooled very slowly. —A. B. S.

Glass; Method of and apparatus for drawing —. A. E. Spinasse, Mount Vernon, Ohio. U.S. Pat. 1,336,056, 6.4.20. Appl., 16.10.15.

THE glass article is drawn from a portion of the glass segregated by a device having sloping polished walls and means for circulating water through it so as to cool it sufficiently to avoid adhesion of the glass. —A. B. S.

Ceramic article [; Refractory —] and method of making same. W. M. W. Horn, Teplitz, Austria, Assignor to The Chemical Foundation Inc. U.S. Pat. 1,335,167, 30.3.20. Appl., 28.10.14.

A REFRACTORY ceramic material, suitable for the heating elements of radiators and capable of withstanding changes in temperature between 20° and 120° C., consists of plastic fireclay, ground grog, and lean kaolin, and is substantially free from quartz, felspar, alkalis, and alkaline earths. —A. B. S.

Tunnel oven; Water-smoking in a —. F. K. Meiser, Nuremberg. Ger. Pat. 315,774, 18.2.19.

THE hot flue gases from the preheating zone are withdrawn from the interior of the tunnel and caused to heat a series of pipes through which is passed a supply of air, the latter being applied under pressure to water-smoke the goods. —A. B. S.

Kilns; Operation of continuous down-draught chamber —. F. K. Meiser, Nuremberg. Ger. Pat. 316,535, 27.6.17.

HER air for combustion is led independently and simultaneously by means of supplementary flues into two chambers being fired. If required, in one chamber the supply of air may be restricted to ensure reducing conditions, whilst in the other oxidising conditions may prevail. By the proposed arrangement double the customary quantity of air is passed through the cooling chambers and a correspondingly larger amount of waste heat is recovered. —A. B. S.

Artificial grinding stones; Production of — for wet grinding. M. Mudrack, Wolfenbüttel. Ger. Pat. 317,583, 3.5.18.

WATERPROOF artificial grindstones are made of a mixture of magnesium chloride lye, "hydraulic magnesia," and quartz, carborundum, emery, or emery-substitute, with oil, wax, or grease. —A. B. S.

Enamelling and annealing furnace. P. Dupont, Paris. U.S. Pat. 1,335,279, 30.3.20. Appl., 14.3.18. SEE Eng. Pat. 114,618 of 1918; J., 1918, 700 A.

Pottery and other like articles; Casting of —. B. J. Allen, Blythe Bridge, and R. Y. Ames, Gidea Park. U.S. Pat. 1,336,180, 6.4.20. Appl., 14.8.17.

SEE Eng. Pat. 110,649 of 1916; J., 1917, 1274.

Ceramic support for contact substances. Ger. Pat. 317,979. See I.

Protecting apparatus. Ger. Pat. 318,033. See I.

Magnesia. Ger. Pat. 303,870. See VII.

IX.—BUILDING MATERIALS.

Portland cement; Thermal phenomena in the setting of —. F. Killig. Zement, 1919, 8, 499—501, 511—515, 523—527, 536—538, 548—551. Chem. Zentr., 1920, 91, 11., 280—281.

The amount of heat evolved during the setting of cement was determined by means of a calorimeter, and tests with a number of samples indicate that measurement of the heat evolved may be more satisfactory than the use of Vicat's needle for investigating the setting of cement.—A. B. S.

PATENTS.

Potassium compounds; Process for recovering — in connection with cement manufacture. A. Mond, London. From The International Precipitation Co., Los Angeles, Cal., U.S.A. Eng. Pat. 134,920, 13.11.18. (Appl. 18,554/18.)

Sodium or magnesium chloride or other alkaline-earth chloride is added to a cement raw mix, or to the fuel, or to both, and the cement mix is heated to a temperature sufficient to drive off any potassium chloride present without melting the mixture. The condensed vapour or dust is collected (e.g., by electrical precipitation) and leached with water and the potassium chloride recovered by evaporation and crystallisation. Any insoluble potassium compounds in the residue after leaching are recovered by adding this residue to another portion of cement raw mix and proceeding as before. The mother liquor is used instead of part of the water in a wet grinding cement plant or it is employed to wet the fuel in a dry grinding plant so as to use any surplus sodium chloride present, or part of the mother liquor may be used for this purpose and the remainder as a solvent of potassium salts in fresh quantities of dust.—A. B. S.

Alkali-bearing rocks; Treatment of — [for production of cement with volatilisation of the alkali]. F. Krupp A.-G. Grusonwerk, Magdeburg-Buckau. Ger. Pat. 315,464, 7.7.18. Addition to 304,080 and 308,690 (J., 1918, 374 A; 1919, 179 A).

By the addition of sulphur, iron sulphide, etc. to the calcium salt (calcium sulphate) mentioned in the chief patent, and by the action of the oxygen in the air and the furnace gases, the acid is liberated and caused to combine, in presence of steam, with the alkali, the potassium sulphate formed being condensed in suitable chambers.—W. J. W.

Concrete; Reinforced — for marine purposes. L. R. Ferguson, Philadelphia, Pa., U.S.A. Eng. Pat. 140,126, 3.10.18. (Appl. 16,100/18.)

In reinforced concrete partly submerged in seawater, the metallic reinforcement in the portion above the water-level or exposed to the atmosphere is made of electro-plated, galvanised, or other rust-resisting or non-corrodible metal to prevent cracking and spalling of the concrete.—A. B. S.

Asbestos-bearing rock; Process of treating —. Asbestos cement. H. S. Ashenhurst, Assignor to J. D. Hubbard, Chicago, Ill. U.S. Pat. 1,317,852 and 1,317,853, 7.10.19. Appl., 18.11.18.

An asbestos cement having a volume-weight of about 25 lb. per cub. ft. is prepared by grinding waste "asbestos sand" in a ball mill,

separating the asbestos by treatment on a screen with 900 meshes per sq. in., and then allowing it to fall freely through a long pipe. When the product is used as a cement, the alkali silicates present pass into solution and are decomposed by atmospheric carbon dioxide with liberation of silicic acid, which acts as a binder.

[Heat-] insulating material made of basic magnesium carbonate and fibres; Production of —. Lipsia Chem. Fabr., Mügeln. Ger. Pat. 303,310, 16.3.15.

Basic magnesium carbonate is precipitated on mineral, vegetable, or animal fibres, and is either drained in a press or allowed to set therein. The process may be carried out in two stages, normal magnesium carbonate being precipitated from a saline liquor and then converted into the basic carbonate by boiling with water.—A. B. S.

Xylolite; Production of non-expanding —. E. Kühn, Bahnhof Gleschendorf. Ger. Pat. 317,493, 22.5.19.

The addition of 1—2% of wood ashes to the materials customarily used for xylolite prevents it cracking as a result of expansion.—A. B. S.

Lime-sand bricks; Hardening —. G. Beil, Königsberg. Ger. Pat. 317,845, 15.6.18.

By placing the freshly-made bricks in a suitable tunnel and heating them by means of exhaust steam before passing them into the ordinary hardening chamber, the time required for hardening is reduced by three hours.—A. B. S.

Slag; Utilisation of — as a building and other constructional material. T. F. Hoare, Bradford. Eng. Pat. 140,874, 12.2.19. (Appl. 3470/19.)

Sulphur from calcium sulphate. Ger. Pat. 302,433. See VII.

Magnesia. Ger. Pat. 303,870. See VII.

Moulded articles. Eng. Pat. 124,669. See XI.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Carbon in iron [: Determination of —]. T. D. Yonsen. Trans. Amer. Electrochem. Soc., 1920, 43—58. [Advance copy.]

In estimating carbon in irons containing less than 0.10% errors occur chiefly in absorption and weighing. To eliminate these errors a method is described in which the carbon dioxide is separated by freezing with liquid air, and is then allowed to expand into a known volume and the increase in pressure noted. The analysis is carried out in three stages, namely, with the sample (a) evacuated cold, (b) heated to 600° C. in vacuo, and (c) heated to 1000° C. in oxygen. This differentiates between the carbon due to gas admitted into the tube when introducing the sample, that existing as adsorbed carbon monoxide or dioxide, and that existing in combined form. In the case of electrolytic iron the amounts of carbon found for the three stages were 0.005%, 0.005%, and 0.005% respectively. By taking suitable precautions in sampling, weighing, etc. the accuracy of the determination is within $\pm 0.0001\%$.—J. W. D.

Carbon determination in ferro-alloys. C. J. Rottmann. Trans. Amer. Electrochem. Soc., 1920, 59—68. [Advance copy.]

By the method described an accuracy of 0.002% can be consistently attained in estimating carbon

in ferro-alloys such as vanadium-, chrome-tungsten-, and chrome-nickel steels. The sample is cleaned with ether in a Soxhlet apparatus, then heated *in vacuo* for several hours at 450° C., and finally heated in oxygen at 1000° C., the carbon dioxide being absorbed in a new type of potash bulb which is compact, light, easily manipulated, and gives complete absorption. Combustion is carried out in a thick-walled silica tube heated either by a nichrome resistance wire or by a carbon resistance furnace. If the latter furnace is used the tube has to be well protected with alundum to prevent penetration of external carbon. No advantage is gained by heating to 1450° C.—J. W. D.

Corrosion of wrought iron; Influence of enclosed slag on the —. L. T. Richardson. Trans. Amer. Electrochem. Soc., 1920, 99—104. [Advance copy.]

PIECES of Swedish iron and wrought iron taken on transverse and longitudinal sections were exposed to the weather for varying periods. The loss in weight and depth of the corrosion pits were found to be approximately the same in both transverse and longitudinal sections. Microscopical examination of the sections in no case gave visible evidences of slag inclusions hindering the corrosion, as several of the slag layers were penetrated, and it is concluded that any superior resistance to corrosion that wrought iron may have is not due to slag inclusions, but to some other cause such as purity of the iron.—J. W. D.

Mild steel; Slag inclusions and decoxidation processes in —. P. Oberhoffer and K. d'Huirt. Stahl u. Eisen, 1919, 39, 165—169, 196—202.

By the oxidation of iron at high temperatures two non-metallic products make their appearance in the microstructure, *viz.*, FeO and Fe₂O₃, the proportion of FeO to Fe₂O₃ increasing as the temperature is raised. The oxides occur sometimes as isolated particles, sometimes as cell walls around the crystal of iron. These oxides decarburise steel. In the molten condition iron oxide compounds exert a solvent action on iron, which may be retained in solid solution on cooling. Ferrous and ferric oxides form solid solutions with each other. Up to 400° C. treatment with hydrogen for 20 min. produces no change, but at 500° C. in 40 min. the bright constituent of the eutectic formed by melting mill-scale is reduced to metallic iron. It is not till 950° C. is reached that the darkest constituent is reduced, 30 min. being required. The exact nature of the two constituents cannot be determined. By forging at 900° C. oxide inclusions in electrolytic iron were appreciably deformed. The mean size of these inclusions was about 25μ, the lower part of the melt containing a larger number than the upper parts. A eutectic structure of Fe-FeO, very similar in character to that of the Cu-Cu₂O eutectic, was observed, which eutectic is very rich in iron. Manganese oxide occurs in greyish particles which, unlike ferrous oxide, are well crystallised, the melting point being higher than that of iron. These inclusions are found towards the top of the ingot. There appears to be some tendency to form a MnO-Fe eutectic. Deoxidation occurs more readily with manganese alloys containing carbon than with pure manganese, and, in addition, the higher manganese alloys reacted with oxygen more readily than those containing less manganese. The ratio of the manganese oxide to ferrous oxide is given as 2.7:1 in oxidised melts rich in manganese. The reaction $\text{FeO} + \text{Mn} = \text{MnO} + \text{Fe}$ does not represent the conditions, no pure MnO being formed. The oxide inclusions appear to consist of more than one constituent, and are in part reducible by hydrogen at 950° C.—F. C. Th.

Carbon steel; Mechanism of quenching in —. P. Chevenard. Rev. Mét., 1919, 16, 17—79. (Cf. J., 1918, 91 A, 420 A.)

IN the case of the more reversible steels where the transformation on cooling occurs entirely at a high temperature, 600°—700° C. (Ar₁), the material is brought into the normal condition and consists of pearlite or troostite, while where the $\gamma \rightarrow \alpha$ change occurs between 150° C. and 300° C. the structure is martensitic. Between these two extremes a complete gradation of intermediate stages may be realised, both Ar₁ and Ar₂ being present and the structure comprising both troostite and martensite. By studying the volume changes of wires very delicate indications of the internal changes occurring can be obtained. To obtain in a steel the same intensity of hardening the cooling must be more and more rapid as the maximum temperature is brought nearer to the Ac point. Further, as the carbon content is lowered the rapidity of quenching must be increased to obtain the same effect. Martensite appears to be an unstable solid solution of carbon in α -iron. A full description of the apparatus designed for registering the dilation of steel wires with temperature is given.—F. C. Th.

Galvanised steel; Metallographic study of —. Y. Tajiri. Soc. Naval Architects, Japan. Engineering, 1920, 109, 327—329.

IN the case of mild steel galvanised by immersion in molten zinc at 650° C., the layer in contact with the steel surface is very hard and brittle. The crystals are perpendicular to the steel surface, and appear to consist of an iron-zinc alloy with 5% Fe. The cleavages are weakest in a direction perpendicular to the steel surface, and under shock this layer tends to split in this direction. Between this layer and the outer layer of zinc is generally found a granular structure of lower iron content (about 2%), which is less hard and brittle and less easily broken. A hexagonal structure may be developed. A decarburising action of the zinc is noted on the surface of the metal after long immersion. It is considered better practice to galvanise the steel before wire drawing rather than afterwards on account of the loss of strength in the latter case due to the heating. As regards the influence of galvanising, the ultimate strength of a mild steel bar was reduced from 25.2 to 24.0 tons, and the yield point from 22.0 to 19.0 tons per sq. m., whilst the elongation (% on 10 diams.) increased from 24.7 to 25.

—F. C. Th.

Copper; Effect of cold drawing on physical properties of commercially pure —. W. E. Alkins. Inst. of Metals, 12.3.20. [Advance copy.] 62 pages.

THE abnormality previously observed in the change of tensile strength with progressive reduction of area (this J., 1918, 587 A) is shown to occur at a reduction which varies with the previous annealing treatment and with the severity of the successive reductions of area. The curves expressing the relationship of the percentage reduction of area with tensile strength, scleroscope hardness, specific volume, and elongation in the tensile test all show two distinct portions connected by an approximately horizontal step over a small range. The breaks all occur at approximately the same reduction. A periodicity in the drawing operation is suggested by the differential tarnishing of the wires. If tarnished wires are lightly re-polished a banded appearance is found which bears some relationship to the diameter of the wire. In the preparation of wires of high electrical conductivity better results are obtained from a large number of light draughts than from fewer heavier ones.—F. C. Th.

Copper; Influence of cold rolling on physical properties of —. F. Johnson. Inst. of Metals, 12.3.20. [Advance copy.] 31 pages.

When copper containing 99.91% Cu and 0.05% O was rolled abnormalities in the effect of rolling upon the physical properties were detected at the following reductions in thickness: 17, 31, 50, 67, 88, suggesting a periodic effect. The ordinary mechanical properties in tension and the lateral spread of the metal during rolling agreed well. The minimum elongation was recorded with a reduction of thickness of about 90%, after which it increased with further rolling from 11.2% to 13.65% after a reduction of 97.8%. The true elastic limit after a reduction of 50% was 8.1 tons per sq. in., and after 58%, 15.8 tons.—F. C. Th.

Copper; Effects of hydrogen on —. W. C. Hotherhall and E. L. Rhead. Inst. of Metals, 12.3.20. [Advance copy.] 10 pages.

SOUND castings can be obtained from copper in which about 0.02% of hydrogen is present if the metal is cast at a suitable temperature. Unless, however, a considerable amount of arsenic is present a badly piped ingot will be produced. Sulphide distributed more or less evenly throughout the metal in the untreated state is concentrated at the crystal boundaries in hydrogenised copper. This lowers the electrical conductivity and renders the copper liable to fail prematurely under load. The hydrogen present is believed to exist in solid solution.—F. C. Th.

Copper; Idiomorphic crystals of electro-deposited —. W. E. Hughes. Inst. of Metals, 12.3.20. [Advance copy.] 14 pages.

WELL-FORMED crystals were obtained under the following conditions:—The bath consisted of 1½ kilos. $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 200 c.c. perchloric acid (sp. gr. 1.12), and 4½ litres of water. The cathode was an iron cylinder lightly coated with copper. The current density decreased during the work from 14 to 12.5 amps. per sq. ft., the voltage being 0.4. At room temperature 3 or 4 days were required for well-formed crystals. These are believed to be tetrakis-hexahedra, and are certainly not octahedra.—F. C. Th.

Brass; Removal of internal stress in 70:30 — by low-temperature annealing. H. Moore and S. Beckinsale. Inst. of Metals, 12.3.20. [Advance copy.] 11 pages.

THE minimum time in which the internal stresses in cold-worked 70:30 brass were completely removed by low-temperature annealing were:—At 200° C., 96 hrs.; 225°, 48; 250°, 5; 275°, 1 hr.; 300°, 20 min.; 325°, 5 min. The minimum times in which notable softening was produced were found to depend on the original hardness. The harder the brass, the lower the temperature and the shorter the time of annealing required to cause a reduction of hardness. For a Brinell hardness not exceeding 165 (2 mm. ball, load 40 kilos.) the internal stress can be completely removed without appreciable loss of hardness by annealing for 1 hr. at 275° C. or for shorter times at slightly higher temperatures or at 250° C. for 5 hrs. At 100° C. the internal stress is not appreciably reduced even after many weeks.—F. C. Th.

[*Corrosion of 70:30 brass condenser tubes.*] Fifth Report of the Corrosion Committee of the Inst. of Metals. G. D. Bengough, R. M. Jones, and R. Pirret. Inst. of Metals, 12.3.20. [Advance copy.] 71 pages.

IN condenser tubes of 70:30 brass the surface layer consists of structureless and highly distorted metal, the resistance of which to corrosion by saline and fresh waters is undoubtedly much greater than

that of the underlying crystalline metal. Whenever this surface layer is penetrated corrosion of the tube will proceed at an increased rate. The thickness of the layer is usually of the order of 0.01 mm. The types of corrosion of brass condenser tubes may be divided into five classes. *General thinning* is usually too slow to be of any serious consequence in practice, but in fresh water, especially in the presence of free acid, rapid general thinning may occur. In 6 weeks water containing 3 pts. HCl per 100,000 caused a loss of thickness of 2–4%. *Deposit attack* is the principal cause of pitting. In sodium chloride solution the cuprous oxide formed on the surface gradually changes to cuprous chloride. When this adheres it is gradually changed to cupric chloride and cuprous oxide; the corrosive action of the former is very rapid, the action being both recurrent and local. Foreign bodies, especially colloids, lying in the tube, act as loci for such pitting. It is necessary, therefore, to keep the tubes clean. *Layer dezincification* is characterised by a disintegration of the tube with the re-deposition of copper over large areas, and occurs under both marine and fresh water conditions. In the latter case it is often associated with acid water. *Local dezincification* is a type of deposit attack stimulated by the presence of foreign bodies. It occurs only in sea-water, and is always associated with adhering white salt (colloidal zinc oxychloride with some carbonate). It occurs spontaneously at 40°–50° C. in a few days. Great and unexplained differences are found in the behaviour of different batches of tubes to this action. *Water-line attack*.—The increased corrosion of a brass tube partly immersed in sea-water occurs not at the water line, but up to as much as 2 cm. above it. The attack is not uniform, but is concentrated at areas covered by salt. Electrolytic protection of brass tubes slows down but does not necessarily inhibit corrosion, currents as large as 20–25 amps. per 1000 sq. ft. being insufficient.—F. C. Th.

Copper-aluminium-zinc alloys rich in zinc. E. H. Schulz and M. Wachler. Metall u. Erz, 1919, 16, 170–176, 195–201.

THE solubility of aluminium in zinc is 0.75% at 380° C. and at room temperature. The addition of 2% of copper produces no change in the diagram for the zinc-rich zinc-aluminium alloys, but beyond this content a ternary eutectic appears containing 6.5 Al and 2% Cu, and melting at 370° C. The addition of 7% of copper prevents the formation of blow holes. By the addition of aluminium to zinc containing copper a rapid increase of hardness occurs up to 3% Al, after which no change occurs up to 10%. Addition of copper, especially up to 3%, to zinc-aluminium alloys, produces a marked hardening. An alloy with 3.5% Al and 4% Cu has the same hardness as one containing 9% Cu and free from aluminium. A given hardness is attained with the minimum additions if both copper and aluminium are used. The tensile strength curves of the ternary alloys bear little or no relationship to those of hardness. Both copper and aluminium raise the tenacity of zinc fairly proportionally to the content, the effect of copper being 3 or 4 times as great as that of aluminium. A maximum stress of 15 kilos. per sq. mm. may be obtained by the addition of 4.7% Cu, or 4% Cu and 3% Al, or 2% Cu and 9% Al. Pure zinc possesses no elastic limit, plastic deformation occurring under all loads. By addition of copper elastic limits up to 22 kilos. per sq. mm. at 7% Cu and 30 kilos. per sq. mm. at 10% Cu are obtained. The effect of aluminium is very similar. In the ternary alloys the elastic limit is related to the hardness. An elastic limit (in compression) of 30 kilos. per sq. mm. is obtained with the ternary alloys containing 11% Cu and 11% Al, 4% Cu and 1% Al, 2% Cu and 2% Al, or 1% Cu and 5% Al.—F. C. Th.

Zinc alloys with aluminium and copper. W. Rosenhain, J. L. Haughton, and K. E. Bingham. *Inst. Metals*, 12.3.20. [Advance copy.] 57 pages.

THE constitution of zinc alloys containing up to 10% Cu and 15% Al is illustrated by diagrams showing the primary, secondary, tertiary, and quaternary separation. Addition of 1% of copper to zinc makes possible a reduction of 90% in thickness by cold rolling without signs of cracks; a further 2% of aluminium causes cracks to appear at a much earlier stage. For hot rolling the alloys a temperature just below the transformation temperature $\eta + \gamma = \beta$ (285° C.) gives better results than one above it. Tensile tests on chilled castings cast at as low a temperature as possible showed that with a constant copper content, the addition of aluminium raised the tensile strength to a marked extent. With more than 4% Al the addition of copper gave a maximum tensile strength at 3%. In hot-rolled alloys the addition of both copper and aluminium raised the tensile strength. Heat treatment had practically no effect on the mechanical properties of chilled bars, but a considerable one on the hot-rolled alloys. Annealing an alloy with 3% Cu and 7% Al at 300° C. raised the tensile strength and considerably reduced the elongation. This anomalous behaviour is attributed to the formation of a large amount of amorphous material during the later stages of rolling, which tends to recrystallise readily. The ageing properties of the alloys render them useless for most purposes. When tested one year after rolling most of the alloys had fallen off considerably in ductility and slightly in tensile strength. Alloys containing Zn 88, Cu 4, and Al 8%, recommended by Guillet and Bernard (this J., 1918, 769 A), undergo a certain amount of shrinkage with ageing, and in the rolled condition deteriorate so as to become useless after a few months. Where a small amount of shrinkage is not an objection alloys containing 3% Cu and 6–15% Al are useful where great strength is required. Special attention is drawn to the great improvement in the mechanical properties of zinc, both in the cold-rolled and hot-rolled conditions, by the addition of a small quantity of copper, e.g., Zn 99 Cu 1.—T. H. Bu.

Bronze plating. F. C. Mathers and S. Sowder. *Trans. Amer. Electrochem. Soc.*, 1920, 83–86. [Advance copy.]

BRONZE can be deposited from a bath containing 5% potassium hydroxide, 0.5% potassium cyanide, 0.38% ammonium stannic chloride, and 1.5% potassium copper cyanide at 40°–50° C., and a current density of 0.4 amp. per sq. dm. (3.75 amp. per sq. ft.), using bronze anodes, with good results. The anodes, however, do not corrode satisfactorily in an oxalate bath, and the authors therefore recommend the use of copper anodes and a bath consisting of copper and tin oxalates dissolved in ammonium oxalate, together with a little potassium sulphate, citric acid, and ammonium citrate or similar salts. The tin content of the bath is maintained by occasionally precipitating copper from a portion of it with metallic tin.—A. R. P.

Tin-phosphorus alloys. A. C. Vivian. *Inst. Metals*, 12.4.20. [Advance copy.] 35 pages.

A SERIES of alloys was prepared in sealed glass tubes embedded in sand in an enclosing steel case, a thermocouple being cemented into contact with the glass tube. Up to 8% P the alloys were obtained by carefully fusing a high-percentage product at atmospheric pressure. From 6.5% to 20% P conjugate solutions occur, but between 16% and 30% P there is little sign of conjugate solutions remaining in the cooled ingot. Above 30% elementary phosphorus appears, but conjugate solutions are roughly indicated at high temperatures. Up to about 12% P the ground mass contains 14.7% P, which

represents the maximum solid solution (α_1) of tin in Sn_3P_2 (α_2). The $\alpha\beta$ eutectic contains 20.5% P and freezes at 540° C. The β phosphide contains 25.8% P, corresponding to Sn_3P_4 . Above 26% P the γ phosphide (about 44% P) occurs as black strings. The $\beta\gamma$ eutectic contains about 31.3% P and freezes at 530° C. Above 44% P the alloys contain red phosphorus. The vapour phase is necessary for the formation of alloys above 2.5% P. The tin arrest decreases with the phosphorus content to nil at 14.5%. From the data obtained a temperature-concentration diagram up to 50% P is suggested.

—T. H. Bu.

Manganiferous silicate slags; Reduction of —. E. F. Kern. *Trans. Amer. Electrochem. Soc.*, 1920, 221–232. [Advance copy.]

SPECIALLY prepared manganiferous silicate slags were reduced experimentally in an electric furnace to produce manganese-silicon-iron alloys of varying composition. Anthracite was used as reducing agent, and the most suitable temperature for reduction was found to be 1470° C. The amount of available metal recovered from the slags varied from 19 to 90%, the loss being due to incomplete reduction and volatilisation. By treatment of a lead blast-furnace zinc slag in a similar manner 75% of the lead and 70% of the zinc were distilled off, and 91% of the iron, 95% of the manganese, and 62% of the copper recovered.—J. W. D.

Electrolytic chromium. G. J. Sargent. *Trans. Amer. Electrochem. Soc.*, 1920, 275–292. [Advance copy.]

A COON deposit of chromium may be obtained by electrolysis of a solution containing 24.5% CrO_3 and 0.3% $\text{Cr}_2(\text{SO}_4)_3$ at 20° C. with a current density at the cathode of 10 amps. per sq. dm. A yield of 0.1 grm. of chromium per ampère-hour is obtained with a voltage of 3 volts and an electrode distance of 2.5 cm. The light grey deposit has scleroscope hardness 75, is unaffected by air, oxygen, or chlorine below 300° C., and may be obtained up to 1.25 cm. thick. Lead anodes give the best results, as the chromic acid reduced at the cathode is re-oxidised at the anode and the composition of the bath thus kept constant. No deposit of chromium is obtained if there is no chromium sulphate present.—A. R. P.

Calcium; Electrolytic production of —. P. H. Brace. *Trans. Amer. Electrochem. Soc.*, 1920, 69–82. [Advance copy.]

THE electrolyte (pure calcium chloride) was first melted by alternating current in an Acheson graphite pot fixed on a water-cooled base and surrounded with insulating material. When the pot was full of molten electrolyte the alternating current was switched off and direct current used for the electrolysis. The anodes were made of graphite and a round-ended iron nut screwed on to a copper rod acted as cathode, which was rotated during the experiment and gradually withdrawn as the deposit grew. A current of 200 amps. was used and was kept uniform to within one amp. during the whole run. The electrolyte was prepared by heating calcium chloride with a small quantity of calcium and stirring with an iron rod until no more gas was evolved and then pouring into iron moulds. Satisfactory deposits in the form of rods about 14 in. long and $\frac{1}{8}$ in. diam. were obtained, which could be worked into wire and sheet by the usual methods. The metal contained 1.08% Cl; sp. gr., 1.46; elect. resist., 6.77 microhms per cm. cube at 22° C.—A. R. P.

Gold and silicon; Alloys of —. C. di Capua. *Atti R. Accad. Lincei*, 1920, 29, i., 111–114.

GOLD and silicon are miscible in all proportions in the liquid condition, but are completely immiscible

in the solid state. The melting point of gold is lowered by about 800° C. by 6–7% Si. (Cf. J.C.S., ii., 323.)—T. H. P.

Sodium amalgam; Influence of various metals on the decomposition of — by water. E. Muller and A. Riedel. Z. Elektrochem., 1920, 26, 101–109.

The rate of decomposition of sodium amalgam by water is very much accelerated by the presence of a small piece of chromium, ferromolybdenum, ferrovanadium, or ferrotungsten. The catalysis is due to an increase of surface caused by the solution and reprecipitation of the catalyst. It will be possible to apply this action to the mercury electrode process for the manufacture of sodium hydroxide if it is found possible to remove entirely the finely divided catalyst from the mercury at the end of the process; otherwise the whole current will not be used in forming sodium amalgam but a portion will be used in liberating hydrogen.—J. F. S.

Metals; Alteration of — by cold working and alloying. P. Ludwik. Z. Ver. deuts. Ing., 1919, 142–144. Chem.-Zeit., 1919, 43, Rep., 308.

A GENERAL account of the effect of cold work on a crystalline aggregate is given. The irreversible changes due to alloying are probably different in nature from the changes due to cold work.

—F. C. Th.

Thermal electromotive force; Study of — as an aid to the investigation of the constitution of alloy systems. J. L. Haughton. Inst. Metals, 12.4.20. [Advance copy.] 25 pages.

THE forms of the E.M.F.-temperature or E.M.F.-composition curves for various alloy systems are described (cf. Broniewski, Ann. Chim. Phys., 1912, 25, 5). The author has investigated theoretically the effect of the arrangement of the constituents in eutectiferous systems, and finds that if they are arranged in series alternately the number of sections does not affect the result. If the constituents are arranged in parallel the E.M.F. generated will be the same as with the series arrangement if the resistance of the constituents is the same; otherwise it will be less. These deductions were verified by experiments on copper and nickel. Practical details are given for the investigation of alloy systems by measurement of the thermal E.M.F., and possible errors due to the Seebeck, Peltier, Thomson, and Benedicks effects are considered.—T. H. Bu.

Blast-furnace gas; Production of power from —. S. H. Fowles. Inst. Elect. Eng., 11.3.20. Engineering, 1920, 109, 443–445.

It is estimated that 1,000,000 h.p. is being constantly wasted in Great Britain in the form of potential energy from blast-furnace gas. The use of blast-furnace gas for boiler-firing is uneconomical, as even with modern plant and clean gas only 30% efficiency is attained. In present practice 40% of the gas is used on the stoves, 15% on the blowers, and none on the boilers, so that 45% is available for other purposes, equivalent to 3000 k.w.-hours per furnace per hour. The furnace blowers require 800 h.p., taking 0.2 ton of coal and 15% of the gas, the total B.Th.U.s required being 20,824,000. The economy on 50 furnaces amounts to 400,000 tons of coal per annum, which is almost equal to the coal required to produce the total units sold in 1911–12 by the 303 electricity works in the United Kingdom. The Halberg-Beth plant for gas-cleaning is recommended, the potash being recovered and the moisture being removed from the gas, making it better for gas-engine use. The residual dust is given as 0.002 gm. per cub. m.

—A. G.

Ternary alloys; Model for representing constitution of —. W. Rosenham. Inst. of Metals, 12.3.20. [Advance copy.] 8 pages.

PATENTS.

Chromium-steel alloy. J. L. Cox, Assignor to Midvale Steel and Ordnance Co., Philadelphia, Pa. U.S. Pat. 1,333,651, 16.3.20. Appl., 1.5.19.

THE alloy contains over 1% but less than 1.5% of carbon and over 20% but less than 60% of chromium.—T. H. Bu.

Titaniferous iron ore; Process for treating —. J. J. W. H. van der Toorn, Assignor to Industrie & Mijnbouw Maatschappij "Titan," The Hague, Netherlands. U.S. Pat. 1,331,001, 16.3.20. Appl., 25.3.18.

A MIXTURE of finely-ground titaniferous iron ores, coal, and limestone flux is briquetted, and the briquettes are reduced to obtain metallic iron. (Cf. Eng. Pat. 114,309 of 1918; J., 1919, 108A.)

—T. H. Bu.

Steel; Treating —. E. F. Kenney, Philadelphia, Pa. U.S. Pat. 1,331,379, 23.3.20. Appl., 20.11.19.

STEEL shapes are finished in a rolling mill at a red heat, allowed to cool to between 500° and 1200° F. (260°–650° C.), and placed in a furnace maintained between these temperatures until the heat of the different parts is substantially equalised. They are then withdrawn and straightened.—A. R. P.

Sheet-iron or other metal; Process for coating — [with metals]. H. Lippmann, Berlin. Ger. Pat. 310,137, 23.3.18.

THE metal to be coated is treated with aluminium, or given a preliminary coat of aluminium paint (Ger. Pat. 312,480; J., 1919, 729A), which is burnt on; the metal is then treated with zinc chloride solution or other solution which acts in a similar manner, and finally subjected to a rolling operation in the bath of the molten coating metal.—A. R. P.

Hardening of articles made of wrought iron; Process for the superficial —. J. Jönsson, Hamburg. Ger. Pat. 317,087, 9.12.17.

WROUGHT iron or other iron low in carbon may be hardened superficially by heating it in contact with small pieces, flakes, or powder of iron rich in carbon (such as cast iron) and a flux until the cast iron melts.—A. B. S.

Ores; Treatment of —. F. Krupp A.-G. Grusonwerk, Assignees of W. Kauffmann, Magdeburg-Buckau, Germany. Eng. Pat. 109,271, 1.9.17. (Appl. 12,590/17.) Int. Conv., 15.9.15.

ORES are calcined or roasted in a revolving cylindrical furnace, to which air is admitted through a series of tuyeres distributed along its length in such a way that the amount of air admitted through each tuyere corresponds to the amount of fuel to be burnt in the vicinity. By this means complete roasting is effected without any sintering taking place and without the application of external heat even if the ore is poor in combustible constituents.

—A. R. P.

Zinc solutions; Purification of —. S. Field, and The Metals Extraction Corporation, Ltd., London. Eng. Pats. (a) 138,946, 22.5.18, (b) 138,950, 25.6.18, and (c) 138,951, 16.11.18. (Appls. 8535, 10,708, and 18,832/18.)

(A) Impure zinc sulphate solution is heated to 80°–100° C. and treated with ozonised air in the presence of a metallic oxide, hydroxide, or carbonate to aid in the precipitation of the metals to be removed. The solution is agitated during treatment. The use of a mixture of manganese dioxide and lead dioxide produced in the electrolysis of a zinc

solution with lead anodes is recommended. (b) Two metals in contact, neither of which is identical with any foreign metal to be removed, are introduced into the solution to be purified. One metal is electro-negative to the metals to be removed and the other either the metal of the solution or electro-positive thereto. Generally the amalgam of the metal forming the base of the solution to be purified is used. The solution is preferably acidified and heated to 70°–100° C. (c) Zinc sulphate solutions containing copper are purified from nickel, copper, and cadmium by agitating at 80°–100° C. with zinc fume or "blue powder" in the presence of a small quantity of acid. The nickel is reduced to 0.5 part per million or less. If the solution is free from copper a small quantity of a soluble copper salt is added. Further acid is added to the zinc solution as required during purification. The zinc may be revived for re-use by washing with a dilute acid.—T. H. Bu.

Zinc solutions; Purification of — H. L. Sulman, S. Field, and The Metals Extraction Corporation, Ltd., London. Eng. Pats. (A) 138,947, and (b) 138,948, 28.5.18. (Appls. 8331-2/18.)

The neutral impure zinc sulphate solution is freed from iron, arsenic, antimony, copper, and cadmium, and then treated (a) with zinc fume or "blue powder" (preferably first washed with acid) at 80°–100° C., whereby the nickel content is reduced to 0.5 part per million or less; and (b) with lead peroxide or manganese dioxide, or a mixture of both (e.g., the mud obtained from electrolytic cells during the deposition of zinc from manganiferous zinc solutions with lead anodes) at 80°–100° C., which reduces the cobalt to the order of 0.5 part per million, or less.—T. H. Bu.

Zinc; Production of good, workable — from inferior material. Siemens-Schuckertwerke G.m.b.H., Siemensstadt. Ger. Pat. 301,335, 9.10.15.

Impure zinc may be freed from sulphur, oxygen, lead, iron, cadmium, and arsenic by heating it to a temperature at which it melts but does not volatilise or oxidise appreciably, and then passing steam or carbon dioxide through it or treating it with charcoal or wood.—A. B. S.

Zinc and other metals; Refining — W. Nestmann, Köln-Kalk. Ger. Pat. 317,511, 1.1.19.

The metal is purified by fusing it and allowing it to stand for a long time in the molten condition. Specifically heavier impurities separate during the solidification of the metal, and after removing these the metal is again fused to allow of the separation of other impurities.

Zinc alloy; Production of a — F. Kammerer, Pforzheim. Ger. Pat. 308,527, 9.12.17.

An alloy of high melting point, moderate hardness, and considerable tenacity and high resistance to compression is obtained by mixing 91–98 parts of zinc with 2–6 parts of copper and manganese, the manganese itself amounting to 0.5–1.7 parts.—D. F. T.

Alloy. F. M. Becket, Assignor to Electro-Metallurgical Co., Niagara Falls, N.Y. U.S. Pat. 1,333,151, 9.3.20. Appl., 26.9.19.

An iron alloy containing 10–45% of chromium, 3–25% of manganese, 0–3% of carbon, and 0–3% of silicon is machinable, resists oxidation at high temperatures, has a low magnetic permeability, and responds readily to heat treatment.—T. H. Bu.

Alloy; Metal — J. B. Grenagle, Catonsville, Md., Assignor to The Rare Metals Reduction Co. U.S. Pat. 1,334,089, 16.3.20. Appl., 16.7.17.

An alloy containing Zr 6.8%, Cr 53.5%, and Ta 39.7%.—T. H. Bu.

Alloy for backing electrotypes and for the manufacture of type metal. M. Schlötter, Berlin-Wilmersdorf. Ger. Pat. 316,369, 27.8.16.

THE tin of the usual lead-antimony-tin alloy is replaced wholly or in part by mercury and the antimony by a suitable smaller quantity of arsenic. An alloy containing Pb 99.4%, As 0.4%, and Hg 0.2% is capable of giving the same number of impressions as one containing Pb 75%, Sb 18%, and Sn 7%.—D. F. T.

Metal; Process of drawing refractory — J. Hisamoto, Nagoya, and Toyo-Kagaku-Yakin-Kabushiki-Kaisha, Tokyo, Japan. Eng. Pat. 139,065, 23.4.19. (Appl. 10,169/19.)

A BAR of refractory metal, e.g., tungsten, is surrounded by a refractory oxide in a metal tube, from which air and moisture are exhausted. The tube, raised to a suitable temperature, is hammered and worked and then drawn into a filament, the tube metal and oxide being finally removed from the core of refractory metal.—T. H. Bu.

Lead; Refining — T. H. Leathart, and Locke, Blackett and Co., Ltd., Newcastle-on-Tyne. Eng. Pat. 140,278. (Appls. 13,597, 25.5.19, and 17,785, 16.7.19.)

ZINC, arsenic, antimony, and other volatile impurities are removed from lead by treating the molten metal with sodium or with phosphorus, mercury, and potassium, or each separately, and then heating the mixture to redness or blowing air or steam, or both, through it. An alloy of 99 parts of lead with one part of the purifying agent is preferably prepared and then one part of one or more of these alloys is added to fifty parts of the lead to be treated.—A. R. P.

Solder for metals, particularly aluminium. R. Iversen, Christiania. U.S. Pat. 1,332,899, 9.3.20. Appl., 27.10.19.

THE solder is composed of tin, zinc, copper, aluminium, and manganese.—T. H. Bu.

Aluminium; Soldering substance for — with process for obtaining it. A. M. Ayala, Madrid. U.S. Pat. 1,333,237, 9.3.20. Appl., 8.3.19.

A solder for aluminium, having a fusion point varying from 180° to 250° C., is composed of 37–43% of zinc, 37–42% of tin, and 17–24% of lead.—T. H. Bu.

Ores; Process of concentrating — J. T. Terry, jun., Assignor to Terry Mining and Metallurgical Corp., Salt Lake City, Utah. U.S. Pat. 1,334,420, 23.3.20. Appl., 16.1.19.

THE powdered ore is treated in water containing a metal-ammonia salt, and the concentrate is recovered by flotation.—B. N.

Copper; Method and apparatus for precipitating — from hot solutions. F. Laist, Anaconda, Mont. U.S. Pat. 1,333,985, 16.3.20. Appl., 8.9.19.

A COPPER-BEARING material is leached with an acid solution, and the copper-rich solution is impregnated with sulphur dioxide and heated to precipitate the copper. The steam is used to preheat another quantity of solution.—T. H. Bu.

Sulphidation and flotation of minerals. J. W. Thompson, Superior, Ariz., and J. T. Terry, jun., Salt Lake City, Utah, Assignors to Metals Recovery Co., New York. U.S. Pat. 1,334,720, 23.3.20. Appl., 16.11.16.

SULPHIDATION of ores preparatory to flotation is carried out in an apparatus consisting of a number of centrifugal pumps, means for feeding the ore pulp to the first of the pumps, and means

for supplying hydrogen sulphide between the pumps so that it is intimately mixed with the pulp.
—J. W. D.

Sulphidation and flotation of ores. J. W. Thompson, Superior, Ariz., Assignor to Metals Recovery Co., New York. U.S. Pat. 1,334,721, 23.3.20. Appl., 16.11.16.

OXIDISED ores which have been sulphidised by the action of a soluble sulphide are subjected to a preliminary aeration treatment before flotation.
—J. W. D.

Concentrating oxidised ores by sulphidation and flotation. Concentrating oxidised ores by flotation. J. M. Callow and J. T. Terry, jun., Salt Lake City, Utah, and J. W. Thompson, Superior, Ariz., Assignors to Metals Recovery Co., New York. U.S. Pats. (a) 1,334,733 and (n) 1,334,734, 23.3.20. Appl., 16.11.16 and 25.11.16.

THE ore pulp is subjected to (a) a combined sulphidising and frothing agent treatment, or (n) a preliminary treatment with a frothing agent, followed by sulphidation, and the resulting sulphidised pulp is subjected to a froth-flotation process.—J. W. D.

Furnace; Smelting —. A. Jones, Los Angeles, Cal., Assignor to U.S. Smelting Furnace Co., Belleville, Ill. U.S. Pat. 1,334,459, 23.3.20. Appl., 28.5.17.

THE furnace consists of a cylindrical body with removable conical ends, both the furnace and the ends being lined with refractory material. The inner part of each end carries a supporting track for rotating the furnace, and the outer part is provided with a central opening.—J. W. D.

Roasted ores containing copper, zinc, silver, etc. [e.g. chloridised pyrites cinders]; *Process for leaching* —. W. Braumüller, Duisburg. Ger. Pat. 309,161, 2.5.15. Addition to 307,648 (this J., 1919, 910 A).

THE hot roasted mass is made into a pulp with a little of the leaching liquid, and run into movable vessels (trucks or vats), from which it is transferred as required to the leaching tanks. By this means loss of material is avoided and the distributing device can be either dispensed with or replaced by a simple cone.—A. R. P.

Pyrites cinder; Process and kiln for chloridising-roasting of —. J. Simon, Frankfurt. Ger. Pat. 317,601, 3.8.17.

PYRITES cinder is mixed with salt and 0–2% of coke breeze and fed into an air-tight shaft-kiln, and air is blown through it in an upward direction. The kiln may have two grates, one of which rotates so as to ensure continuous removal of the roasted product and thus increase the output of soluble copper with a minimum quantity of salt.—A. B. S.

Manganiferous slags and ores; Process for working up — by wet chlorination. Farbenfabr. vorm. F. Bayer and Co., Leverkusen. Ger. Pat. 309,278, 30.9.17.

MANGANESE ores or slags are heated at a high temperature under pressure with a solution of ferrous chloride (e.g. spent pickling liquor). The manganese dissolves as chloride, while iron is precipitated (compare Ger. Pat. 307,174; this J., 1919, 951 A).—A. R. P.

Electric arc welding, cutting and repairing [using alternating current]. C. J. Holslag, South Orange, N.J., U.S.A. Eng. Pat. 126,271, 14.11.18. (Appl. 18,689/18.) Int. Conv., 30.4.18.

Roasting of ores or concentrates. U. C. Tainton, Martinez, Cal., U.S.A. Eng. Pat. 140,943, 13.6.19. (Appl. 14,989/19.)

SEE U.S. Pat. 1,310,455 of 1919; J., 1919, 643 A.

Roasting furnace. F. Fouarge, Swansea. U.S. Pat. 1,335,490, 30.3.20. Appl., 24.4.17. Renewed 4.2.20.

SEE Eng. Pat. 102,595 of 1916; J., 1917, 142.

Electric blast-furnace. J. Bibby and J. O. Boving, Assignors to Electro-Metals, Ltd., London. U.S. Pat. 1,336,017, 6.4.20. Appl., 16.1.19.

SEE Eng. Pat. 132,616 of 1918; J., 1919, 867 A.

Zinc solutions; Purification of —. H. L. Sulman and S. Field, Assignors to The Metals Extraction Corporation, Ltd., London. U.S. Pat. 1,336,386, 6.4.20. Appl., 12.5.19.

SEE Eng. Pat. 138,948 of 1918; preceding.

XI.—ELECTRO-CHEMISTRY.

Electric furnaces; Step induction regulator for —. P. B. Short. Trans. Amer. Electrochem. Soc., 1920, 35—42. [Advance copy.]

A DETAILED description is given of an electrical control equipment recently designed for twenty electric furnaces in Norway, consisting of a combination induction-type regulator, a selector switch, a transfer switch, a series transformer, and a suitable panel for meters and switches.—B. N.

Electrolysis of alkali salts. Van Laer. See VII.

Ferrates. Grube and Gmelin. See VII.

PATENTS.

Primary galvanic batteries with unalterable electrolyte. E. W. Jungner, Kneippbaden, Sweden. Eng. Pat. 118,843, 30.8.18. (Appl. 14,106/18.) Int. Conv., 4.9.17.

AN electropositive porous carbon electrode having one surface exposed to the air, and a substance, capable of facilitating depolarisation by the oxygen of the air, deposited on this surface, is floated on the electrolyte, so that the air-depolarising substance is only partly soaked by the liquid. The electrode may be made in the form of a frame containing pieces of electrically conducting carbon, with plates or ribs pressed against the carbon pieces by springs serving as terminals for the electrode. An oxide of a metal (Cu, Ag, Mn, Hg) may be used as the depolarising substance. The cell may be covered by a porous cover, admitting air to the air-depolarising substance, but preventing or delaying evaporation from the electrolyte. The electro-negative electrode may be composed of zinc, iron, tin, lead, or aluminium in an electrolyte of a sodium or potassium salt, or iron, tin, or lead in an ammonium salt electrolyte, the latter also containing not more than 10% of an alkali metal or ammonium hydroxide or carbonate. The metal combines with the acid radical of the electrolyte, whilst a soluble alkali hydroxide is also formed, which transforms the electronegative metal into an insoluble hydroxide, regenerating the original electrolyte. The metallic electrode may consist of a horizontal grate with vertical parallel bars or ribs connected together, the limbs being of different length, and with an extension outside the cell for manually imparting a shaking motion to the electrode so as to remove the deposited metallic hydroxide.—B. N.

Moulded articles [insulators, resistance blocks, etc.]; Manufacture of —. The British Westinghouse Electric and Manufacturing Co., Ltd., London. From Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa., U.S.A. Eng. Pat. 124,669, 10.9.18. (Appl. 14,722/18.)

DRY hydraulic cement is mixed with a dry fusible impregnating material, such as resin or pitch, preferably coumarone or indene-resin, with or without addition of asbestos and barytes or other inert filler; the mixture is wetted, moulded, allowed to set, and then heated to fuse the impregnating material.

Electro-osmotic process for removing water from materials. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. Eng. Pat. 135,817, 25.6.18. (Appl. 10,455/18.) Int. Conv., 20.10.17.

WATER is removed electro-osmotically from vegetable, animal, or mineral substances by first treating the material, which is to be dried, with the water-containing electrolyte flowing away from material under electro-osmotic treatment; or the material may be brought to the osmotic apparatus in the form of a thin magma, by washing it from its natural bed by a jet of water obtained from the liquid flowing away from material under electro-osmotic treatment.—B. N.

Displacement of liquids in materials [e.g., nitrocellulose] by other liquids, by the aid of electro-osmosis; Process for —. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. Ger. Pats. (A) 305,512, 18.3.17, (B) 307,701, 6.6.17, and (C) 307,702, 21.6.17.

(A) THE material to be treated is subjected to the action of the electric current between electrodes, the displacing liquid being introduced at one electrode and the displaced liquid removed at the other. The material is separated from the electrodes by diaphragms. The process is accelerated by the use of electrolytes, viz., by those containing OH-ions if the liquid moves towards the cathode and by those containing H-ions if it moves towards the anode. In displacing water from nitrocellulose by alcohol, for example, alcohol is fed in at the anode, and the displaced water collects at the cathode, both electrodes being perforated. The cellulose bodies for which the process is particularly suitable, are, for electro-osmotic purposes, almost without exception electro-negative, so that the process is favourably influenced by the addition of a small quantity of ammonia, caustic soda, etc. (B) The displacing liquid is introduced and the displaced liquid removed at places corresponding to the specific gravities of the respective liquids. For example, the displaced fluid is drawn off at the lowest point if its specific gravity is greater, and at the highest point if its specific gravity is lower, than that of the displacing fluid. (C) The displacing fluid is cooled before use, enabling stronger currents to be employed without the risk of attaining dangerous temperatures.—J. S. G. T.

Electro-osmotic dehydration of animal, vegetable, and mineral substances; Process for —. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. Ger. Pat. 316,444, 14.2.18.

A MOVING electrode is employed which serves also as a conveyor for the material to be treated. The electrode may be in the form of an endless band, which is periodically displaced relatively to the other (stationary) electrode, for the purpose of filling the dehydration space, and relatively to a scraper or to the material itself for purposes of emptying. The band electrode is also provided with a suitable cut-out for purposes of emptying.—J. S. G. T.

Electro-osmotic dehydration apparatus; Process for feeding material to be treated in —. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. Ger. Pat. 316,494, 21.10.17.

THE material to be treated is fed along those electrodes at which liquid is removed, the feed opening being placed adjacent to this electrode, or the channels for feeding the material passing through the electrodes. The fresh material thus meets a very fluid mass of material, which opposes very little resistance to its passage, and the layer of dehydrated material is not disturbed.

—J. S. G. T.

Electro-osmotic dehydration apparatus. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. Ger. Pat. 316,495, 21.10.17.

DISTANCE pieces in the form of pegs, frames, etc., and insulated from one of the electrodes, are disposed between the electrodes. The channels for conveying the material to be treated may serve also as distance pieces, being for this purpose mechanically attached to the electrode at which the dried material is separated.—J. S. G. T.

Electro-osmotic dehydration of vegetable, animal and mineral materials; Apparatus for —. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. Ger. Pat. 316,496, 21.10.17.

THE material to be treated is subjected to treatment in a so-called chamber-press, the plates of which are employed as electrodes, one or both of these being of a permeable nature. The frames are provided with channels covered with filter-cloth or wire gauze and connected to an outlet channel.

—J. S. G. T.

Electrodes for electro-osmotic purposes; Double —. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. Ger. Pat. 316,593, 21.10.17.

A GRID of corrugated sheet iron is covered on both sides with a thin removable layer of wire gauze. Alternatively, two perforated metal sheets may be connected by bolts or the like and covered on both sides with wire gauze.—J. S. G. T.

Electrodes for electric furnaces. Det Norske Aktieselskab for Elektrokem. Ind. Norsk Industri-Hypotekbank, Christiania. Eng. Pat. 137,811, 8.9.19. (Appl. 22,068/19.) Int. Conv., 17.1.19.

AN electrode consisting of a metallic casing filled with a carbonaceous mass (see Eng. Pats. 116,853 and 119,018; this J., 1918, 429 A, 661 A) is heated to a comparatively low temperature, preferably in the furnace in which it is to be used, part of the heat being generated by current passed into the mass through the metal casing. Internal ribs, with or without projections on the casing, ensure good electrical contact between the internal mass and the casing, and the latter is provided also with holes through which the gases, evolved during the baking, escape. The casing is made in sections, so that as the lower part is consumed a fresh section may be added to the top containing the stamped electrode mass.—B. N.

Electrode. K. Tornberg, Lynn, Mass., Assignor to General Electric Co. U.S. Pat. 1,334,820, 23.3.20. Appl., 28.8.18.

THE electrode comprises a metal tube, a film of water-glass on the inner surface thereof, a filling of magnetite mixture in the tube, and a seal on the top of the filling consisting of a solidified button of plastic material having about 0.5% of anhydrous potassium fluoride incorporated in it.—B. N.

Carbon electrodes; Production of a strong protective coating for —. Ges. für Teerverwertung-m. b. H., Duisberg-Meiderich. Ger. Pat. 316,450, 6.11.17.

A coating which does not flake off at the temperature of the electric furnace is produced by treating the electrodes after baking, and while hot, with a mixture of metallic oxides and water-glass or like substance.—J. S. G. T.

Electric furnaces. E. Fenton, Loughborough. Eng. Pat. 139,843, 21.6.19. (Appl. 21,831/18.)

A VERTICAL electric furnace comprises a stationary base and a body portion adapted to be rotated relatively to the base, the latter being provided with a discharge opening through which the material is uniformly fed downwards by rotation of the upper portion. The upper and lower portions are separated by a water-seal into which flows water projected on to the furnace for cooling purposes; the overflow passes over the outer surface of the base of the furnace. To ensure uniform clearance at the adjacent extremities of the upper and base portions of the furnace, a circular lining consisting of detachable segmental plates is provided.—B. N.

Electric furnace. P. de Miles, New York. U.S. Pats. (A) 1,317,863 and (B) 1,317,864, 7.10.19. Appl., 25.1.18.

(A) A CRUCIBLE of electrically resistant material is supported by a number of spaced electrodes, which in turn are supported by arms, and means are provided for exerting pressure on the electrodes to cause them to make good contact with the crucible. (B) An electrical resistance rod extends through a hollow casing and is engaged by spaced electrode terminals, and means, insulated from the electrodes, are provided for exerting pressure on the electrodes to ensure good contact with the resistance rod.

Electric furnace. W. K. Booth, Assignor to The Booth Electric Furnace Co., Chicago, Ill. U.S. Pat. 1,332,795, 2.3.20. Appl., 3.2.19.

THE furnace body, with contact members mounted on it, is supported so that it can turn on an axis, adjustable electrode clamps being supported on the ends of the furnace body and engaging the electrodes extending through the ends. The contact members are connected with the clamps by flexible conductors supplying current through a second set of contact members engaging the first-named contacts.—B. N.

Electric furnace and method of operation. L. C. H. Groeger, Chicago, Ill. U.S. Pat. 1,333,914, 16.3.20. Appl., 5.4.19.

THE heating chamber of the furnace is provided with a hearth terminal and a number of co-acting side electrodes which extend towards each other from the sides, and are so disposed above the bath as to enable an arc to be established directly between their inner ends. The electrodes may be tilted, and an arc may be established directly between the electrodes, or between the electrodes and the bath, with the circuit completed through the hearth terminal.—B. N.

Electric furnace. M. R. Trembour, Watervliet, N.Y., Assignor to Ludlum Electric Furnace Corp., Albany, N.Y. U.S. Pat. 1,333,950, 16.3.20. Appl., 24.3.16.

THE furnace is provided with a melting hearth, ovoid in plan, and with a dish bottom constricted towards the opposite ends of its major axis, where doors are placed for charging and discharging. A removable low arched roof, similar to the hearth in shape, is provided with arches at the opposite

ends of its major axis, thus completing the door openings. Vertically-adjustable electrodes, passing through the roof, are arranged along the major axis of the furnace.—B. N.

[Electric] furnace for endothermic gas reactions [e.g., oxidation of nitrogen]. Elektrochem. Werke, G.m.b.H., Berlin. Ger. Pat. 316,349, 24.3.17. Addition to 268,410.

THE gases, after passing through the hot zone between the electrodes, are conveyed through a long, water-cooled tube constructed of a thin material which is a good conductor of heat and of such a diameter that the velocity of the gases through the tube is several hundred metres per second. The temperature is reduced almost instantaneously by about 1000° C., and a nitric oxide concentration of 2.5% is attained.—L. A. C.

Electrochemical reactions in gases and vapour by means of the alternating discharge; Process and apparatus for effecting —. H. Spiel, Vienna. Ger. Pat. 317,502, 19.7.18. Int. Conv., 19.7.17.

AN absorbing liquid functions as one or other of the electrodes or as an auxiliary electrode. The arc is struck between a liquid surface and a tubular electrode through which a brisk stream of drops flows. Electrolytic decomposition of the absorbing liquid is prevented by employing current of frequency above 500 per sec. In this manner air may be converted into an aqueous solution of nitric acid. In similar manner, by discharge of oxygen in the presence of organic or inorganic liquids, oxidation processes may be effected, and hydrogenation and chlorination similarly in presence of hydrogen and chlorine respectively. Organic substances may be synthesised by the discharge of carbonaceous gases or vapours over water and other liquids.—J. S. G. T.

Electrolytic cell. U. C. Tainton, Johannesburg, Transvaal. U.S. Pat. 1,334,419, 23.3.20. Appl., 5.11.18.

THE cell comprises a casing consisting of a number of ring sections secured together, with a fixed electrode, provided with a central opening, secured in the interior of each alternate casing. A shaft, extending through the casing, is journaled in and insulated from the head members, and electrodes are secured on the shaft at points intermediate of the fixed electrodes. A rotary motion is given to the shaft and electrodes carried by it. Means are provided for passing an electrolyte from one end of the casing to the other. Each fixed electrode is enclosed in, but spaced from, a fabric covering, forming a chamber about each fixed electrode for the reception of an electrolyte, and preventing the circulating electrolyte from mixing with the one about the fixed electrodes and from coming in contact with the latter.—B. N.

[Electrolytic] oxygen and hydrogen generator. G. Miller, Los Angeles, Cal. U.S. Pat. 1,334,668, 23.3.20. Appl., 17.6.19.

A CELL member, open at one end and closed at the other, made of non-metallic, non-conducting, non-absorbent material and having a transverse partition, is mounted in a receptacle containing an electrolyte with the open end downwards, the transverse partition being spaced slightly above the floor of the container, and electrodes are mounted within the chambers with a gas conduit leading from each of them.—B. N.

Electro-osmotically removing liquids from materials; Apparatus for —. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. Eng. Pat. 135,818, 25.6.18. (Appl. 10,457/18.) Int. Conv., 20.10.17.

SEE Ger. Pat. 316,495 of 1917; preceding.

Electrolytic cell. J. Harris, Lakewood, Ohio, U.S.A.
Eng. Pat. 140,563, 21.2.19. (Appl. 4382/19.)

SEE U.S. Pat. 1,297,157 of 1919; J., 1919, 425 A.

Electrolytic cell. H. C. Jenkins, London. U.S.
Pat. 1,335,338, 30.3.20. Appl., 3.9.19.

SEE Eng. Pat. 132,561 of 1918; J., 1919, 829 A.

Electric furnace. C. W. Speirs, Assignor to
Morgan Crucible Co., London. U.S. Pat.
1,335,079, 30.3.20. Appl., 10.6.19.

SEE Eng. Pat. 129,760 of 1918; J., 1919, 686 A.

Electrical treatment of gases. U.S. Pats. 1,333,790
and 1,334,231. See I.

Electrical purification of gases. Ger. Pats. 316,498.
318,432, and 318,772. See I.

Electrical filter. U.S. Pat. 1,334,160. See I.

Electrical heater. Ger. Pat. 317,302. See I.

Copper sulphate and hydrogen. Ger. Pat. 317,691.
See VII.

Pencil drawings. Ger. Pat. 317,444. See XIII.

Alcohol and ethyl acetate. Eng. Pat. 140,115.
See XX.

Successinylacetic acid esters. Ger. Pat. 300,672.
See XX.

XII.—FATS; OILS; WAXES.

Oils and fats; Purification of —. B. Lach.
Oel u. Fettind., 1919, 1, 363—364, 389—391,
414—416. Chem. Zentr., 1920, 91, 11., 251.

VARIOUS fats and oils can be purified by treatment with nascent hydrogen (zinc and acid). A white product may thus be obtained from dirty tallow, and the method gives good results with distilled olein. Rancid fats may be rendered fit for use by boiling them with live steam, and treating them while boiling with 1 to 2% of alum, which is added, little by little, as a concentrated solution. The fat is neutralised etc. after an hour. The method is applicable to bone fat, margarine, etc.—C. A. M.

Soap; Analysis of —. Weber. Seifens.-Zeit., 1919, 46, 652. Chem.-Zeit., 1920, 44, Rep., 98.

For factory control water in soap is best determined by Fahrion's method (J., 1910, 1065) when carbonates are absent, but for the analysis of filled soaps, lubricating soaps, and soap powder distillation with xylene gives the best results. Free alkali is determined by Davidsohn's method, a solution of the soap in 60—70% alcohol being treated with barium chloride, and then titrated with standard acid without filtering. Alkali carbonate is determined by direct titration of a 50% alcoholic solution of the soap, and deducting the amount of acid neutralised by the free alkali. In determining the fatty acids by extraction with ether, the ethereal solution is left for 12 hours over dehydrated sodium sulphate, and then filtered, and the residual sodium sulphate washed with ether. In the case of soaps containing coconut or palm-kernel oils, Goldschmidt's method is used. After evaporation of the ether, 2 to 3 grms. of the fatty acids is titrated with N/2 alcoholic alkali solution, and the result calculated on 1 gm. of fatty acids. Four grms. of the soap is then dissolved in sodium silicate solution, and treated with hydrochloric acid, the fatty acids extracted with ether, the ethereal solution washed with sodium chloride solution and evaporated, and the residue dissolved in about 55 c.c. of neutral alcohol and treated as described.—C. A. M.

Myricyl alcohol; Brodie's —. A. Gascard.
Comptes rend., 1920, 170, 886—888.

By careful analysis of the corresponding iodide and comparison of the alcohol, iodide, and corresponding saturated hydrocarbon with known adjacent members in the same series, it is shown that the myricyl alcohol obtained from beeswax has the formula $C_{31}H_{63}OH$. (Cf. J. C. S., June.)—W. G.

PATENTS.

Fats, oils, and waxes and their acids; Process for decolorising —. Vereinigte Chem. Werke A.-G., and K. Lüdecke, Charlottenburg. Ger. Pat. 317,448, 22.1.18.

FATS or the like, after hydrolysis of the neutral constituents, are treated with chlorine, and the products are hydrolysed by heating in the absence of water with the addition of substances capable of absorbing hydrochloric acid. The product is distilled with superheated steam under reduced pressure, yielding a fat free from odour and suitable for use in soap manufacture.—L. A. C.

Lipase; Manufacture of preparations containing —. R. Willstätter, Munich. Ger. Pat. 317,503, 25.9.18. Addition to 316,504 (J., 1920, 306 A).

LIPASE is absorbed from emulsions or suspensions by large seed-shaped granules of pumice stone, wood or animal charcoal, diatomite, or the like.—L. A. C.

Liquids or solutions insoluble in water; Process for dissolving or emulsifying —. Chem. Fab. Flörsheim, Dr. H. Noerdlinger, Flörsheim. Ger. Pat. 314,017, 19.12.15.

SULPHONATED compounds prepared according to Ger. Pat. (J., 1920, 164A) are employed for emulsifying or rendering miscible with water liquids which in themselves are immiscible therewith. Mineral or fatty oils mixed with the sulphonated compounds can be used as drilling oils or lubricants. Disinfectant liquids are made with mixtures of these compounds with phenols or tar oils; laundry or fat-removing liquids by combining them with hydrocarbons of the aromatic series or with chloro derivatives of aliphatic or aromatic hydrocarbons; and with various other substances, mixtures useful in the textile, dyeing, and leather industries are prepared.—J. F. B.

Tar soap; Manufacture of —. W. Schmidt, Elberfeld, and E. Heuser, Darmstadt. Ger. Pat. 316,258, 15.3.18.

WOOL-, lignite-, or coal-tar is extracted with water to dissolve volatile organic acids, and is then saponified to prepare a tar soap. The aqueous extract is evaporated to remove the volatile acids, and the dissolved tar, which separates as a syrupy liquid, may also be saponified.—L. A. C.

Washing and cleaning; Process for —. K. Horkenbach, Duisberg. Ger. Pat. 317,402, 13.9.17.

A DETERGENT is used containing sodium carbonate or a mixture of sodium carbonate and water-glass, together with 1 to 5% of a dried extract from peat or humic coal. The extract is prepared by treating the peat or humic coal with water or an alkaline or acid solution, either at the usual or high temperatures, and under either normal or reduced pressure, separating insoluble matter, and evaporating the colloidal solution obtained.—L. A. C.

Catalysts. U.S. Pat. 1,335,161. See I.

Fatty acids from wood tar, etc. Ger. Pat. 315,417. See III.

Linsced oil substitute. Ger. Pat. 317,731. See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Carbon black; its properties and uses. G. St. J. Perrott and R. Thiessen. J. Ind. Eng. Chem., 1920, 12, 324—331.

Most of the carbon black now manufactured in the United States is made by the "channel" system, in which the black from natural gas is deposited upon the smooth under-surface of steel channels by means of burners 3 to 4 in. below them. The channels are arranged in tables of eight, and are given a slow reciprocating motion whereby the deposited black is scraped from them into hoppers. The quality of the product varies with the amount of air, the speed of scraping, and the pressure of the gas. In accordance with the conclusions of Bone and his co-workers (J., 1915, 786), gases rich in ethane and higher homologues give greater yields of carbon by this method. The temperature of the channels is maintained at about 300° C. At the present time only relatively small quantities of lampblack are used for printers' ink, and then only in admixture with carbon black. Carbon black is used in rubber mixings and in the paint trade; its sp. gr. is 1.8 and it has a greater tinting and covering capacity than any other black. *Testing methods:* The tinting strength and colour are estimated by comparison with a standard black, the former with a mixture of the black and zinc oxide and the latter with the black itself, in both cases mixed with oil. Chemical tests include those for moisture, ash, and acetone extract. An acetone extract exceeding 0.1% indicates adulteration with a poorly calcined lampblack. A sample containing more than 0.2% ash is probably adulterated with mineral black or charcoal. Most blacks for ink contain 2—4% moisture, though certain kinds may contain up to 7%. Some blacks will absorb as much as 15% of their weight of moisture. Specifications are suggested for printing ink, rubber, and paint. The use of the torsion viscosimeter will indicate the behaviour of a black in practice. When equal weight of blacks with the same amount of raw linseed oil are tested by means of MacMichael's apparatus (J., 1915, 1226), the lowest readings are given by those kinds valued by ink makers for their "length." *Chemical analysis:* Carbon blacks contain 85—95% amorphous carbon, 1—7% water, 0.5—0.8% hydrogen, and 2—8% oxygen (partly as CO and CO₂, partly as fixed oxygen). A black yielding a "long" ink is usually low in carbon and high in volatile matter and oxygen, whilst "shorter" blacks show the reverse. Typical analyses are given. As a rule, "long" blacks are more hygroscopic than "short" blacks. They may also be distinguished microscopically in freshly-prepared mixtures with their lithographic varnish, "short" blacks gradually agglomerating into groups of 20 to 100 particles, whereas the particles of "long" blacks remain completely dispersed after several hours.—C. A. M.

Finger-prints. Mitchell. See XXIII.

PATENTS.

White [antimony oxide] pigment; Manufacture of —. A. L. Barbe, Asnières, France. Eng. Pat. 140,301, 24.7.19. (Appl. 18,392/19.)

ANTIMONY oxide obtained by roasting the sulphide is separated and introduced as an aqueous suspension into a vat fitted with agitators. An alkaline solution, preferably lime, is then added, the mixture well agitated, and a current of oxygen or compressed air forced in by means of a pipe fitted to the lower part of the vat until any sulphite present is eliminated.—A. de W.

Painting and priming compositions; Manufacture of —. Flakes A./S., and E. Schou, Copenhagen. Eng. Pat. 140,147, 4.1.19. (Appl. 290/19.)

PAINTS or priming compositions are prepared by gradually adding oleaginous materials, e.g., drying oils, varnishes, solutions of waxes or tars, etc., to a thin,ropy, aqueous solution of emulsifying ingredients, i.e., a thin solution or fine mixture of starch, casein, glue, gums, or the like which is kept in constant motion, the emulsion being thereafter treated by homogenising machinery so that the particles of the oleaginous material are reduced to a minutely divided state (diam. 10 μ or less). Further proportions of oil or water, or pigment prepared in oil or water, may be added without destroying the emulsion.—A. de W.

Resins and turpentine; Recovery of — from resinous woods. W. H. Sawyer, Auburn, Me. U.S. Pat. 1,331,679, 23.3.20. Appl., 26.2.19.

CHIPS of resinous woods are treated so as to break down the cellular structure without reducing the fibre length, and extracted with a cold volatile alkaline solvent, the latter being then separated from the wood and the alkali volatilised.—A. de W.

Resin-emulsions; Preparation of —. Badische Anilin- und Soda-Fabrik, Ludwigshafen. Ger. Pat. 307,123, 23.1.17.

ARTIFICIAL resins and resin-like substances such as condensation products of formaldehyde with aromatic compounds are dissolved in high-boiling organic solvents, and these solutions intimately mixed with soap solutions. The formaldehyde-naphthalene condensation product (Ger. Pat. 207,713; J., 1909, 431) gives with monochlorobenzene and Turkey-red oil, a fine emulsion of the consistency of ointment which is miscible with water to a milk-like translucent liquid, but requires the addition of gelatin or similar substance to form dilute preparations.—B. V. S.

Resin soaps; Manufacture of —. O. Braemer, Hamburg. Ger. Pat. 317,344, 2.3.18.

NEEDLES or other portions of conifers containing turpentine are treated with oxidising agents and saponifying agents, such as alkalis, either consecutively or simultaneously. The oxidising agent, e.g., sodium peroxide or percarbonate, swells and opens up the plant tissue and promotes the extraction of resin. In the treatment of ground fir needles the cellulose constituents may be allowed to remain in the soap as a filler. The brown colour of the soap may be removed by bleaching agents.—J. F. B.

Artificial resin; Manufacture of an —. C. C. Bohler, Dresden. Ger. Pat. 317,267, 19.9.18.

ANHYDROFORMALDEHYDEANILINE, either alone or with the addition of a small percentage of organic or inorganic acids and alkalis, is heated to 100°—200° C., and a vulcaniser, such as sulphur chloride, is added either during the period of heating or to the final product.—L. A. C.

Linseed oil; Substitute for —. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 317,731, 8.5.17.

A SUBSTITUTE for linseed oil is made from the products of condensation or polymerisation of aldol, acetaldehyde, crotonaldehyde, or other aldehydes, in presence of alkalis, sodium acetate, etc. The products yield elastic, glossy lacquers, especially in the presence of driers.—A. B. S.

Pencil drawings on paper; Fixation and blackening of —. A. Grimert, Weimar. Ger. Pat. 317,441, 11.5.19.

THE paper is placed between metal plates or rollers

and subjected to electrolysis, water being a satisfactory electrolyte for the purpose.—W. J. W.

Plastic luting composition; Manufacture of a —. Gebr. Heyl und Co. A.-G., Charlottenburg. Ger. Pat. 317,138, 13.11.17.

A PLASTIC luting composition is composed of naphthenic acids and an oxide of a heavy metal, e.g., iron oxide.—L. A. C.

Recovering solvents [from coatings on aeroplane parts etc.]; Apparatus for use in —. E. I. du Pont de Nemours and Co., Assignees of T. Baker, Wilmington, Del., U.S.A. Eng. Pat. 123,752, 20.2.19. (Appl. 4276/19.) Int. Conv., 19.1.18.

Hydrocarbons. Ger. Pats. 309,281-2. See XX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; Action of certain organic accelerators in the vulcanisation of —. G. D. Kratz, A. H. Flower, and C. Coolidge. J. Ind. Eng. Chem., 1920, 12, 317—324.

THE accelerating effect of certain substances such as diphenylthiourea is due to their tendency to decompose under the vulcanisation conditions into simpler substances containing an active nitrogen group to which the acceleration is attributable. The same accelerating influence is shown by substances containing the same active nitrogen group in their primary nucleus, if these are used in molecularly equivalent quantities. For example, the activity of diphenylthiourea is practically equal to that shown by an amount of aniline corresponding to the quantity formed in the decomposition of the former. The activity of the parent substance is reduced by replacing the hydrogen in the active nitrogen group by other and larger groups. The activity of the nitrogen in certain groups is most readily explained on the assumption that the valency of the nitrogen is increased from the trivalent to the pentavalent condition, with the temporary addition of sulphur.—C. A. M.

PATENTS.

[Rubber] composition for use as floor covering and method of manufacturing same. W. Stocks, Fitzroy, Victoria. Eng. Pat. 121,136, 29.11.18. (Appl. 19,763/18.) Int. Conv., 27.10.17.

Rubber; Method of reclaiming —. D. F. Twiss, Assignor to The Dunlop Rubber Co., Birmingham. U.S. Pat. 1,335,926, 6.4.20. Appl., 6.12.18.

SEE Eng. Pat. 122,249 of 1918; J., 1919, 152 A.

Colouring fibrous material. U.S. Pats. 1,332,974 and 1,332,982. See VI.

Piperidine piperidylthiocarbamate. Ger. Pat. 316,009. See XX.

XV.—LEATHER; BONE; HORN; GLUE.

Glue jellies; Testing the strength of —. W. H. Low. J. Ind. Eng. Chem., 1920, 12, 355—356.

A MODIFICATION of Smith's glue tester consists essentially of a U-tube containing mercury covered with a layer of coloured water and provided with an adjustable scale on one limb. The other limb is extended at right angles and then bent downwards and is connected to a rubber pressure bulb, whilst a vertical capillary tube extending downwards and terminating in an inverted thistle funnel covered with a rubber diaphragm, is fused to the middle of the extension. Steady pressure is applied

to the bulb until the water in the capillary tube is forced downwards from an initial to a final mark, and the height of water in the U-tube is then read on the scale. The glue jelly in a glass vessel is then fixed against the rubber diaphragm by means of a movable support, and the force required to drive the water between the two points on the capillary tube again measured, the figure previously found for the rubber diaphragm alone being deducted. The method is sensitive and gives concordant results, which enable glues to be graded with comparative accuracy.—C. A. M.

Glue; Determination of the tensile strength of —. G. Hopp. J. Ind. Eng. Chem., 1920, 12, 356—358.

THE glue is melted, poured into moulds of polished iron, 12 in. square and $\frac{1}{4}$ in. deep, and allowed to set. The glue-sheet is dried on fine-meshed wire round which the air can circulate, and the drying completed under pressure while protected from air currents. Strips about 7 in. long, and with an average thickness of 0.1 in. and width of 0.33 in., are cut from the sheet, and the tensile strength determined by means of a Schopper machine. The average tensile strengths of commercial glue used in three different factories were respectively 13.24, 8.52, and 11.57 lb. per sq. in.—C. A. M.

PATENTS.

Condensation products [tanning substances] from aminohydroxynaphthalenesulphonic acids and formaldehyde; Production of —. Deutsch-Koloniale Gerb- und Farbstoff-Ges. m.h.b., Karlsruhe. Ger. Pat. 315,871, 11.6.14. Addition to Ger. Pat. 313,523 (Eng. Pat. 18,174 of 1913; J., 1917, 93).

CONDENSATION between 2 mols. of a salt of an aminohydroxynaphthalenemono- or di-sulphonic acid and about 1 mol. of formaldehyde in aqueous solution, if necessary with the addition of an acid, is effected more rapidly and completely if the mixture is heated to boiling than at ordinary temperatures.

—L. A. C.

Emulsifying liquids. Ger. Pat. 314,017. See XII.

Karaya gum compositions. U.S. Pats. 1,334,356-8. See XVII.

XVI.—SOILS; FERTILISERS.

Lime; Action of — in the soil. J. Hughes. J. Bath and W. and S. Counties Soc., Vol. 13. Reprint. 11 pages.

QUICKLIME was mixed with four times its weight of soil and exposed in the air-dried state indoors and in the moist state outdoors. Two types of soil were used containing respectively 3.85% and 14.97% of organic matter and combined water. In both cases where the material was exposed in the moist state the whole of the lime was carbonated within six weeks. With eleven Herefordshire soils it was found that, in every case where the magnesia was in excess of the lime, full crop results could not be obtained.—W. G.

Phosphates; Decomposition of — with bisulphate [for the production of superphosphate]. B. Neumann and K. Kleylein. Z. angew. Chem., 1920, 33, 74—77.

WHEN sulphuric acid above 47.5° B. (sp. gr. 1.49) is saturated with bisulphate, the acidity of the solution is diminished, but with weaker acids the bisulphate increases the acidity. Bisulphate can, therefore, only be used advantageously with the weaker acids. In the preparation of super-

phosphate from natural phosphate, the best yield of soluble phosphoric acid, about 92% theory, was obtained with acid of 48° B. (sp. gr. 1.498) when the theoretical quantity of sulphuric acid was used, whilst when $\frac{1}{2}$ of the theoretical quantity of acid was used, the yields were fairly constant at 68 to 72% with acids varying from 31.3° to 53° B. (sp. gr. 1.277–1.58). When, however, a mixture of $\frac{1}{2}$ of the theoretical quantity of acid and a quantity of bisulphate equivalent to the remaining $\frac{1}{2}$ was used, the yield increased with decreasing strength of acid, reaching a maximum of 88.6% with acid of 31.3° B. (sp. gr. 1.277). The use of bisulphate is likely to affect unfavourably the physical properties of the superphosphate, particularly when the natural phosphate used contains a high proportion of calcium carbonate.—E. H. R.

Calcium cyanamide; Inorganic impurities in crude —. J. Baumann. Chem.-Zeit., 1920, 44, 275–276.

MAGNESIA in particular, and in lesser degree silica, disturbs the working of the electric furnace. Sulphur decreases the yield of ammonia obtainable from calcium cyanamide by removing nitrogen as thiocyanate; it might be profitable to increase the sulphur content materially if thiocyanate were recovered as a by-product. The residue of lost nitrogen is found in the autoclave sludge. During the oxidation of ammonia to nitric acid silica is deposited upon and poisons the platinum catalyst if silicon hydride is present (cf. Ger. Pat. 276,720; J., 1915, 28).—A. E. D.

Nitrate nitrogen and commercial organic nitrogen; Relative availability of —. Field and cylinder experiments. A. W. Blair. J. Ind. Eng. Chem., 1920, 20, 262–264. (See also Lipman and Blair, J., 1918, 521 a.)

CYLINDER experiments, extending over 7 years, on the relative efficiency of sodium nitrate and dried blood in loam soil and in mixtures of loam with 10–100% of coarse sand, showed that the superiority of the nitrate was well maintained until the proportion of sand amounted to about 70%. Only when the proportion of sand was 100% did the crop yield (dry substance) from blood exceed that from nitrate. In field plot trials extending over two 5-year rotations the average yield of dry substance for the five-year periods was 500 lb. per acre more from sodium and calcium nitrates than from organic nitrogen (dried blood, fish, and tankage). The average nitrogen recovery of all the nitrate plots for the 10 years was 37.4% as compared with 26.5% for the organic fertiliser plots. —J. H. L.

Chloropierin and higher plants. Bertrand. See XIXb.

PATENTS.

[Calcium] cyanamide; Process for granulating —. L. L. J. Barbé, Saint Maurice, France. Eng. Pat. 140,331, 1.10.19. (Appl 24,039/19.) Int. Conv., 21.3.19.

COARSELY-DIVIDED crude calcium cyanamide is reduced in a crusher to pieces of the size of an egg, then freed from carbide by treatment with water, and the pieces reduced to granules of the required size by means of crushing stamps, the formation of powder being avoided as far as possible. The granules are coated with powdered calcium cyanamide in a suitable machine and finally passed through sieves.—W. J. W.

Boiling and drying organic matter, more particularly slaughterhouse waste; Process for —. K. Niessen, Munich. Ger. Pat. 317,818, 7.2.15.

THE material is heated in a closed vessel surrounded

by a steam jacket. After vapours have ceased to be evolved from the material, steam is shut off from the jacket and turned on to a second jacket surrounding the first. Simultaneously fresh air is passed through a jacket surrounding the outer steam jacket into the inner vessel, the necessary cocks etc. being all appropriately turned by one operation, to effect the desired flow of steam and air.—J. S. G. T.

XVII.—SUGARS; STARCHES; GUMS.

Calcium salts; Removal of — from carbonated [beet sugar] juices. V. Stanek. Z. Zuckerind. Czechoslov., 1919, 44, 45–47, 53–58.

CARBONATATION alone is incapable of removing all the calcium salts of organic acids present in beet juices, and the addition of sodium carbonate or sulphite is advised. Better results were obtained when the sodium carbonate or sulphite was added before the completion of carbonatation, and also when both the first and second saturations were carried out at boiling point. A further improvement was effected by increasing the amount of calcium carbonate precipitated, especially at the second saturation, the amount of calcium sulphate and colloidal calcium salts thrown down thus being increased. Taking advantage of these observations, and without adding more sodium carbonate or sulphite than that equivalent to the calcium salts present in the juice, it was possible to reduce the calcium content present after clarification to at least 2 mg. per 100 c.c.—J. P. O.

Beet-sugar after-product massecuites; Ammoniacal frothing of —. K. Urban. Z. Zuckerind. Czechoslov., 1919, 44, 21–24.

DURING the 1918–19 campaign the author noticed that an after-product beet massecuite sometimes frothed considerably after being 24 hrs. in the coolers, carbon dioxide and also ammonia being evolved. The phenomenon is attributed to “frothy fermentation,” the carbon dioxide being produced by reaction between the invert sugar and the amino-acids (Maillard, J., 1912, 144), and the ammonia being formed by the decomposition of the ammonium salts of amino-acids during concentration and crystallisation. After the massecuite had remained in the coolers for about 48 hrs., and the temperature had fallen to 70°–75° C., the frothing subsided.—J. P. O.

Ammonia evolved during clarification and evaporation [in the beet sugar factory]; Recovery of the —. E. Donath. Z. Zuckerind. Czechoslov., 1919, 44, 61–64.

AMMONIA is constantly present in the steam arising from the liming and carbonatation tanks, the heaters, and the evaporators of the beet sugar factory. According to the author's estimate, the amount of ammonium sulphate that might be recovered in the clarification department alone in the case of a plant slicing 6000 quintals of roots in 24 hrs. is 3827 kg. per day. Recovery might be effected by absorption in towers, after first partially cooling the vapour and then passing it over caustic lime; or the process of separation as ammonium sulphite in a centrifugal fan might be applied (cf. Ger. Pat. 281,095; J., 1915, 612).—J. P. O.

Sucrose; Examination of Herzfeld's double polarisation method of determining —. R. F. Jackson and C. L. Gillis. Facts about Sugar, 1920, 10, 90.

MEASUREMENTS of the rate of decomposition of invert sugar in the presence of hydrochloric acid at different temperatures lead the authors to conclude

that the time of heating prescribed in the Herzfeld method of determining sucrose (viz., 3–5 min. while raising to 68°–70° C., and 5 min. at 69° C.) is the cause of considerable error. They believe a total period of 4½ min., and a temperature of 60° C., to be sufficient, which conditions necessitate the re-establishment of the factor for calculating the result. When applying the double polarisation method to products containing much invert sugar, an error arises owing to the conditions of the direct and inversion readings not being comparable, the direct reading being made in the presence of basic lead acetate, and the second in that of hydrochloric acid. Since, however, certain neutral salts (e.g., sodium chloride) affect the rotation of invert sugar in the same direction as hydrochloric acid, it is proposed to add a suitable amount of sodium chloride to the solution used for the direct reading, the inversion reading being taken in the presence of hydrochloric acid as usual.—J. P. O.

Sucrose; Double polarisation methods for the determination of — and a suggested new method. G. W. Rolfe and L. F. Hoyt. J. Ind. Eng. Chem., 1920, 12, 250–253.

THE difference between the inversion constant for the Herzfeld conditions (142.66° V.) and for the invertase method (141.7° V.) is due to the influence of the acid on the rotation of the invert sugar, and if the acid is neutralised before the inversion reading the difference becomes still greater. Inversion with very dilute acids at high temperatures gives constants closely approaching that for the invertase method, but the cooled inverted solution takes hours to attain a constant rotation. As the result of a study of mono- and trichloroacetic acids as hydrolytic agents, the authors suggest that the following method, which enables the direct and inversion readings to be made on solutions of the same acidity and sugar-content, and which, tested on low-grade Cuban sugars and a refinery syrup, gave results much nearer to those obtained by the invertase method than the Herzfeld procedure gave. A solution of the normal weight of the sample is clarified with basic lead acetate, made up to 100 c.c. and filtered, as usual. For the direct reading 50 c.c. is transferred to a 100 c.c. flask, treated with 15 c.c. of a 20% solution of monochloroacetic acid, made up to volume, and polarised within 15 mins. after the addition of the acid. For inversion, about 50 c.c. of this acidified solution, transferred to a 50 c.c. flask which is then tightly stoppered by tying down the cork, is heated in vigorously boiling water for 30 mins., or for 60 mins. if a large quantity of lead acetate has been used for clarification. The solution is afterwards cooled and allowed to stand for at least 2 hours before being polarised. The constant 141.0 is used in calculating the sucrose. Excess of basic lead acetate equivalent to 1 c.c. in a half-normal sugar solution does not affect the inversion nor produce troublesome precipitates. The solution does not darken during inversion to the same extent as under the conditions prescribed by Herzfeld.

—J. H. L.

Sucrose; Determination of — in cane molasses by Walker's double polarisation method. A. Brodie. Int. Sugar J., 1920, 22, 174.

WALKER'S method (J., 1917, 153) has been found to give results that are 0.3–0.5% lower than the truth, due mainly to the hydrolysis of sucrose by the excess of phosphoric acid in the liquid used for the direct reading at laboratory temperature. Half the quantity of phosphoric acid (i.e., 10 instead of 20 c.c. of a 10% solution) gives results that are correct within the limit of experimental error. Horne's solid basic lead acetate with which clarification is effected may contain sufficient moisture to cause an appreciable error.—J. P. O.

Sugar; Effect of concentration on the deteriorative activity of mould spores in —. N. Kopeloff, S. Byall, and L. Kopeloff. J. Ind. Eng. Chem., 1920, 12, 256–257. (See also this J., 1919, 592 A.)

IN mixtures of blackstrap molasses with different proportions of sugar syrup of 60° Brix, inoculated with spores of various moulds, the amount of sucrose inverted during storage for 4 months at 23°–30° C. was found to vary with the "moisture ratio," i.e., percentage of mixture/(100–polarisation). In pure sterilised sugar coated with similar mixtures in a centrifuge, so as to give moisture ratios ranging from 0.08 to 0.2, and heavily inoculated, the invert sugar-content increased in all cases (in some cases four-fold) during one month's storage, and in general to a greater extent the higher the moisture ratio, but irregularities were observed attributable to differences in the degree of inoculation and the amount of syrup mixture on the crystals. *Aspergillus Sydowi*, *Bainier*, *A. niger*, and *Penicillium expansum*, in the order named, produced the greatest deterioration.—J. H. L.

Bagasse; Deterioration of — on keeping. C. Janssen. Archief Suikerind. Nederl.-Indië, 1919, 37, 1974–1975. Int. Sugar J., 1920, 22, 169.

Loos and Schweizer (Archief, 1919, 27, 1772–1775) found the sucrose content of bagasse to diminish at a rapid rate. After 3 hours, for example, the sucrose content had fallen from 3.17 to 1.77%, and after 6 the polarisation of the extract was 0°. This observation is not confirmed by the author, who was unable to observe any diminution in the sucrose content of bagasse after keeping it at the prevailing laboratory temperature for 8 hours.—J. P. O.

PATENTS.

Sugar cane juice or the like; Apparatus for filtering —. A. Azpiazu, Habana, Cuba. U.S. Pat. 1,332,877, 9.3.20. Appl., 14.10.18.

THE apparatus comprises a series of conical filtering surfaces, arranged one above another, an inclined receiving bottom with a discharging channel for the filtrate below each filtering surface, and conduits for discharging the filtration residue from one filtering surface to the next below it through the intermediate receiving bottom.—J. H. L.

Decolorising liquids; Process for — especially sugar juices. Verein der Zuckerindustrie in Böhmen, Prague. Ger. Pat. 317,449, 1.8.17.

THE decolorising medium, e.g., carboraffin (Staneek, J., 1919, 192 A), or similar substance, is deposited from a mixture with water, dilute sugar solution, etc., in the frames of a filter-press, through which the liquid to be treated is then passed in the usual way.—B. V. S.

Karaya thickener and process of making the same. Dried soluble karaya-gum composition and process of making. Casein composition. H. V. Dunham, Meunt Vernon, N.Y. U.S. Pats. (A) 1,334,356, (B) 1,334,357, and (C) 1,334,358, 23.3.20. Appl., (A) 19.7, (B) 26.11, and (C) 25.3.19.

(A) KARAYA gum is dissolved in water containing a quantity of sodium bicarbonate equal to about 3% of the weight of the gum, and the solution is dried. (B) Karaya gum is soaked in water containing a small quantity of a weak alkali, a lacteal fluid is then added, and the mixture dried. (C) A composition containing soluble casein and soluble karaya gum.—L. A. C.

Dextrin and other modified starch products; Manufacture of —. H. C. Gore, Takoma Park, Md. U.S. Pat. 1,335,162, 30.3.20. Appl., 21.6.19.

A SUITABLE amount of acidulated water is added to a quantity of starch, together with a small amount

of dextrin to act as a binder; the mixture is formed into an open mass, dried, and dextrinised in a current of warm air.—A. B. S.

VIII.—FERMENTATION INDUSTRIES.

Fructose [levulose]; Fermentation of — by *Lactobacillus pentoaceticus*, N. Sp. W. H. Peterson and E. B. Fred. J. Biol. Chem., 1920, 41, 431—450.

THIS organism will decompose levulose with the formation of acetic acid, lactic acid, carbon dioxide, and considerable amounts of mannitol. The latter substance is, however, itself attacked if the fermentation is prolonged, and the final products are then acetic and lactic acids and carbon dioxide. It is feasible that an intermediate stage in the decomposition is represented by malic acid.—J. C. D.

Acetone and butyl alcohol fermentation of starch by Bacillus granulobacter pectinovorum. H. B. Speckman. J. Biol. Chem., 1920, 41, 319—343. (Cf. J., 1919, 155 τ .)

THE starch is converted into dextrose by an exoenzymic action. The dextrose then passes into the cells and is oxidised to acetic and butyric acids, which are reduced in part to the corresponding alcohols. The origin of the acetone is regarded as not proven.—J. C. D.

Red wines and fruit juices; Acidimetry of —. A. M. Macmillan and A. Tingle. J. Ind. Eng. Chem., 1920, 12, 274—276.

THE acidity of red wines may be determined in presence of phenolphthalein as indicator by using the spectroscope to ascertain the end-point (cf. J., 1918, 117 τ , 443 A). Other coloured liquids may be titrated in a similar way, but the details of procedure must be adapted to individual cases. The method may be used also with methyl red; 100 c.c. of liquid containing 3 c.c. of a 0.05% solution of this indicator, when viewed through a depth of 45—50 mm., shows in presence of acid a strong absorption band in the green, which disappears sharply when the neutral point is reached.

—J. H. L.

Vinegar; Determination of oxalic acid in —. A. Bau. Daut. Essigind., 1919, 23, 358—361, 366—368. Chem. Zentr., 1920, 91, II., 298.

THE author's calcium acetate reagent (J., 1918, 524 A, 671 A) gives far more reliable results than a saturated solution of calcium sulphate, and can be applied without previously neutralising the vinegar. Normal fermentation vinegar contains no oxalic acid, but this may be formed if over-oxidation has taken place. Commercial acetic acid may contain glyoxalic acid, which forms oxalic acid on oxidation. The quantity of oxalic acid in such a case is too small to be detected by the calcium sulphate reagent, but can be found by the calcium acetate reagent.—J. F. B.

Bacteria and hydrocarbons. Tausz and Peter. See IIIA.

Preservatives. Perry and Beal. See XIXB.

PATENTS.

Sauce and process of making the same [from soya beans]. S. Satow, Sendai, Japan. U.S. Pat. 1,332,448, 2.3.20. Appl., 5.5.17.

A MIXTURE of soya bean, roasted grain, water, and koji is "fermented," alcoholic or other harmful fermentations being prevented, e.g., by addition of common salt, and the filtered liquid is evaporated to dryness in vacuo.—J. H. L.

Fermented beverages; De-alcoholising —. G. B. Sippel and G. J. Patitz, Assignors to P. Schoenhofen Brewing Co., Chicago, Ill. U.S. Pat. 1,333,457, 9.3.20. Appl., 8.2.18.

THE hot fermented beverage, mixed with a heated current of gas, is introduced in a finely divided form into a de-alcoholising chamber, where it encounters cross currents of a heated inert gas. The finely divided liquid is maintained throughout at a temperature approaching 100° F. (38° C.).—J. H. L.

Yeast; Manufacture of —. Verein der Spiritus-Fabrikanten in Deutschland, Berlin. Ger. Pat. 303,311, 21.12.15.

THE yeast used for pitching worts prepared from sugar or molasses and mineral salts, is purified by a fermentation of the same nutrient solution, in which the mineral acid liberated in the course of fermentation is allowed to accumulate, calcium carbonate being added only when the yeast has been purified by the acid liberated. The acidity necessary for purification is about 0.125%.—J. H. L.

Mash filtration; Apparatus for —. J. G. Crossman, Watford. Eng. Pat. 140,946, 30.6.19. (Appl. 16,421/19.)

XIXA.—FOODS.

Proteins of barley, oats, rye, and wheat kernels; Nutritive value of the —. T. B. Osborne and L. B. Mendel. J. Biol. Chem., 1920, 41, 275—306.

THE proteins of the entire kernel of these grains are adequate for growth and more efficient than those of the endosperms.—J. C. D.

Corn [maize] gluten meal; Nutritive value of commercial —. C. O. Johns, A. J. Finks, and M. S. Paul. J. Biol. Chem., 1920, 41, 391—399.

COMMERCIAL maize gluten meal supplemented by dried brewers' yeast, whole ground yellow maize, or coconut press-cake furnishes the necessary protein for normal growth of rats. Eighteen per cent. of whole, ground, yellow maize-meal furnishes sufficient water-soluble vitamins for normal growth.

—J. C. D.

Corn [maize] and corn meal; Cause of deterioration and spoiling of —. J. S. McHargue. J. Ind. Eng. Chem., 1920, 12, 257—262.

WHOLE grain containing 12% of moisture can be kept in good condition for at least 12 months if absorption of further moisture is prevented. In moist air it absorbs water, and when it contains 15% moulds develop at the ordinary temperature and under conditions of restricted ventilation, and rapid deterioration ensues. Alcoholic and acetic fermentation takes place when the moisture-content amounts to 20%. Flour made from whole grains undergoes an increase of acidity when exposed to the air, but with a moisture-content of 12% it may be kept suitable for human consumption for 4—6 months, provided moisture and air are excluded. Dried flour undergoes little or no increase of acidity, especially at low temperatures. Flour made from grain previously freed from embryos, with a moisture-content of 12%, may be kept for 12 months when air and moisture are excluded, without acquiring any musty or sour flavour, the only change which occurs being a gradual increase in acidity, confined mainly to the oil, but the removal of the embryos greatly diminishes the nutritive value of the grain. Some particulars are given of the kinds of mould found growing on spoiled maize.—J. H. L.

Kennet powder; Preparation of —. O. Gratz. *Molkerei-Zeit.*, 1913, 29, 273—274, 279—280. *Chem. Zentr.*, 1920, 91, II., 299.

RENNET extracts were made either with 5% sodium chloride solution or with 0.2—0.3% hydrochloric acid, and were preserved with formaldehyde or toluene. The best precipitating agent was sodium chloride, which was added to the strongly acid extract almost to saturation. The slimy precipitate, of yellowish-grey colour, settled only slowly. Occasionally a portion floated as a fine scum. The product was dried at 30°—35° C. under reduced pressure. The organic portion was rich in chymosin. The powder could be re-dissolved in water, and its enzyme activity and solubility were increased by a second precipitation, although the yield was considerably reduced. In the first precipitation 15—20 g. of dry powder was obtained from 1000 g. of extract.—J. F. B.

Hydrocyanic acid content of Phaseolus lunatus beans. H. Lührig. *Chem.-Zeit.*, 1920, 44, 262.

THE enzyme occurring naturally in *Phaseolus lunatus* beans liberates the whole of the hydrocyanic acid when the beans are mashed with water (J., 1920, 277A); the addition of saliva does not increase the quantity (prolonged action decreases it), and no further quantity is obtained when the distillation residue is treated with emulsin, saliva, bile, or pancreatic juice.—W. P. S.

Fruit juices. Macmillan and Tingle. See XVIII.

Preservatives. Perry and Beal. See XIXB.

Action of chloropierin on wheat parasites. Piutti. See XIXB.

PATENTS.

Potatoes and the like vegetable substances; Process for the economical desiccation of —. Wauquier et Cie., Lille, France. Eng. Pat. 122,422, 15.1.19. (Appl. 1083/19.) Int. Conv., 15.1.18.

THE material, converted into a finely divided form by shredding, crushing, or otherwise, is freed from as much water as possible in centrifugal separators of the imperforate, non-filtering drum type, and finally completely dried by a known method. Four-fifths of the water present in fresh potatoes can be removed by the centrifugal treatment.—J. H. L.

Meat; Dry and soluble extract of raw —. A. Chalas, Paris. Eng. Pat. 129,639, 8.7.19. (Appl. 17,070/19.) Int. Conv., 10.7.18.

THE liquid plasma of meat is separated by pressure from the muscular fibrin, and evaporated in a high vacuum at a very low temperature, to exclude bacterial development. Evaporation may be effected in bulk, and if necessary in two successive evaporators, or by means of drying rollers provided with scrapers, inside the vacuum chamber, or by injecting the plasma into the chamber in a finely divided form, in which case electric lamps in the upper part of the chamber may be used to replace the heat lost by evaporation. Dehydrating agents, such as sulphuric acid, calcium chloride, or zinc chloride, are preferably used to absorb the moisture evaporated. Soluble products, such as albumoses, may be prepared from the muscular fibrin, e.g., by the action of pepsin or papain, and added to the plasma in the course of or after desiccation.—J. H. L.

Meat-like preparations; Manufacture of —. P. Hildebrandt, Hamburg, Germany. Eng. Pat. 139,525, 31.1.18. (Appl. 1816/18.)

ANIMAL or vegetable refuse containing proteins, e.g. fish waste, cartilage, horn, hair, oil-cake, etc., is treated with oxidising agents such as permanganates, chromates, ozone, hydrogen peroxide, or

per-salts, and afterwards hydrolysed by boiling with strong mineral acids for several hours.—J. H. L.

Meat juice preserves; Manufacture of —. Nobel und Co., Hamburg. Ger. Pat. 309,180, 6.12.17. Addition to 307,135 (J., 1920, 78A).

IN order to curtail the cooling process and to prevent the development of odours, hydrogen peroxide is added to the mixtures containing meat, salt, vegetables, and milk powder before the cooling process. The hydrogen peroxide is decomposed by enzymes with liberation of oxygen, which effects the conversion of the mixture more rapidly than air.—J. F. B.

Milk; Process of making a substitute for —. A. B. Moses, Seattle, Wash. U.S. Pat. 1,332,562, 2.3.20. Appl., 19.5.19.

A MIXTURE of flour derived from the seed of leguminous plants and water is heated at about 160° F. (71° C.), filtered, and then heated practically to the boiling point to precipitate proteins.—J. H. L.

Bran and the like; Apparatus for extracting from — their nutritious components. L. C. Reese, Saginaw, Mich. U. S. Pat. 1,334,552, 23.3.20. Appl., 16.12.18.

IN a battery of intercommunicating extractors adapted to be worked in series at different temperatures, each extractor is connected to means for heating its contents to the temperature required for the series worked at the higher temperature, and to means for cooling the extracting liquid on its passage to the series worked at the lower temperature, and means are also provided for maintaining the temperature uniform in this series.—J. H. L.

Egg substitute; Manufacture of —. H. S. Cullen and G. L. Fulton, Wellington, N.Z. Eng. Pat. 120,396, 24.10.18. (Appl. 17,399/18.) Int. Conv., 2.11.17.

Artificial milk; Manufacture of —. K. Erslev, Nijmegen, Holland. Eng. Pat. 121,133, 28.11.18. (Appl. 19,673/18.)

SEE U.S. Pat. 1,297,668 of 1919; J., 1919, 436 A.

Sauce. U.S. Pat. 1,332,448. See XVIII.

XIXB.—WATER PURIFICATION; SANITATION.

Water; Sterilisation of — by ultra-violet rays. W. L. Decker. Cleveland Eng. Soc., Jan., 1920. *Chem. and Met. Eng.*, 1920, 22, 639—645.

WITH a quartz lamp at 66 volts, 3.5 amps., the time necessary to destroy various germs was determined. Less than 20 seconds is sufficient for *B. coli*. To obviate interference by suspended matter, even after filtration, it is advantageous to agitate the water during exposure to the rays, for which purpose a special apparatus with baffles has been devised. A graph is given which is a continuous record, over a period of 3 months, of the bacteria in the untreated, filtered, and sterilised water; the turbidity of the untreated and filtered water; the efficiency of bacteria removal in the filters; the rate of filtration; the hourly and daily consumption of water; the number of men working; the oxygen consumed in the raw water; the presence or absence of pathogenic germs in the treated water. If fresh bacteria are added to water which has been treated with ultra-violet rays, 90% of these succumb in an hour.—W. J. W.

[Sewage] *sludges; Action of activated — on the ammonia of sewage and of ordinary water.* Diénert and Girault. *Comptes rend.*, 1920, 170, 899—901. (Cf. J., 1920, 347 A.)

THE activated sludges prepared as previously de-

scribed (*loc. cit.*) cause the disappearance of ammoniacal nitrogen in an almost identical manner from sewage and from ordinary water the ammoniacal nitrogen content of which has been made equal to that of the sewage. As the sludge gets older the ratio of ammonia oxidised to time of disappearance at first diminishes and then increases. Nitrous nitrogen only appears after the sludge has been used for several months.—W. G.

Preservatives; Quantities of — necessary to inhibit and prevent alcoholic fermentation and the growth of moulds. M. C. Perry and G. D. Beal. J. Ind. Eng. Chem., 1920, 12, 253—255.

TEST-TUBES containing sterilised 2% dextrose meat broth were inoculated with *Sach. cerevisiae* and *Penicillium glaucum*. The following table shows approximately the percentages of various antiseptics required to prevent visible fermentation or mould growth and also the percentages required to destroy the organisms completely. In the case of alcohol the results differed according as the tubes were closed by a cap to prevent evaporation or not. The action of sodium benzoate appears to be due solely to the antiseptic properties of the free acid.

	<i>Sach. cerevisiae.</i> No gas evolved.	Organisms destroyed.	<i>Penic. glaucum.</i> No visible growth.	Organisms destroyed
Alcohol:				
Without cap ..	16	20	8	17
With cap ..	11	15	8	14
Sodium salicylate	1	9	3	5
Sodium benzoate	0.5	3	0.25	5
Sodium sulphite ..	0.6	above 12	or less above 12	above 12
Sodium bisulphite	0.25 or less	10	0.25 or less	6
Formaldehyde ..	0.25 or less	0.25 or less	0.25 or less	0.4

—J. H. L.

Chloropicrin; Action of — on the parasites of wheat and on rats. A. Piutti. Comptes rend., 1920, 170, 854—856.

A CLAIM for priority over Bertrand and his co-workers (J., 1919, 336A, 437A). 20 c.c. of chloropicrin per cub. m. of space, at 15°—20° C., proved effective for the destruction of wheat parasites, and the flour and bread made therefrom were quite inoffensive and retained all their nutritive power. For the destruction of rats in ships, a much lower concentration of chloropicrin than that used by Bertrand (*loc. cit.*) is effective.—W. G.

Chloropicrin; Action of — on the higher plants. G. Bertrand. Comptes rend., 1920, 170, 858—860.

At moderately high concentrations of chloropicrin (200 g. per cb. m.) the leaves of the trees examined were rapidly killed but remained fixed on the branches. At much lower concentrations (20 g. per cb. m.) marked plasmolysis was noticeable and finally the leaves softened, turned brown, and fell off. This effect diminished as the concentration was further diminished. Young leaves are less sensitive than old leaves, and the terminal ends are very resistant.—W. G.

Absorptive power of charcoal. Winter and Baker. See VII.

PATENTS.

Cresol preparations; Production of clear —. P. Borinski, Berlin. Ger. Pat. 315,016, 5.8.17.

CRESOL is mixed with an emulsifying agent, *e.g.*, concentrated sulphite-cellulose waste lye, and as much of a salt of an aromatic hydroxycarboxylic acid, *e.g.*, sodium salicylate, as is necessary to pro-

duce a clear mixture. Preparations containing up to 90% of cresol can be obtained as permanently clear liquids.—H. J. H.

Emulsifying liquids. Ger. Pat. 314,017. See XII.

Tar soap. Ger. Pat. 316,258. See XII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Theobromine; Solubilities of —. R. V. Wadsworth. Analyst, 1920, 45, 133—131.

The high solubilities for theobromine recorded by Mitscherlich appear to have been due to the presence of caffeine as an impurity. The solubility of the pure alkaloid in various solvents was as follows: Ether at 15.5° C., 0.003; at b. pt., 0.003; trichloroethylene at 15.5° C., 0.020; at b. pt., 0.03; chloroform, 0.06 and 0.07; water, 0.06 and 0.70; and tetrachloroethane, 0.090 and 0.870%.—C. A. M.

Hexamethylenetetramine; Preparation of —. A. Sander. Z. angew. Chem., 1920, 33, 81.

THE formation of hexamethylenetetramine by cold treatment of an ammonium salt with excess of formaldehyde is not confined to ammonium carbonate (J., 1920, 279A), but occurs with most ammonium salts, and the reaction is, in fact, utilised for the estimation of ammonia. By the use of the carbonate, however, a purer product is more readily obtained.—W. J. W.

ββ'-Dichloroethyl sulphide. C. S. Gibson and W. J. Pope. Chem. Soc. Trans., 1920, 117, 271—278.

SULPHUR dichloride reacts with dry ethylene at 40°—50° C. according to the equation $2\text{CH}_2:\text{CH}_2 + \text{SCl}_2 = (\text{CH}_2\text{ClCH}_2)_2\text{S}$, but owing to the further rapid action of the dichloride on the product, the yield only amounts to about 50% of the weight of the sulphur dichloride taken, and a considerable proportion of impurity is present. A much purer, but otherwise identical, product is obtained in 90—98% yield by the action of ethylene on sulphur monochloride at temperatures up to 70° C., preferably at 55°—60° C. The absorption of the ethylene at the commencement is facilitated by adding about 5% of ββ'-dichloroethyl sulphide to the monochloride. When the absorption of ethylene abruptly ceases, the product is heated to 100° C. to redissolve the viscous deposit of sulphur which separates towards the end of the reaction. On subsequent cooling the sulphur is deposited in a compact crystalline form from which the dichloroethyl sulphide can readily be separated by decantation. Pure ββ'-dichloroethyl sulphide, colourless needles, m.p. 13°—13.5° C., is without action on aluminium, lead, brass, iron, bronze, zinc, or tin at ordinary temperatures, and on the first three metals at 100° C. It has sp. gr. 1.285 at 15°/4°, and $n_D^{15} = 1.53125$.—G. F. M.

Dichloroethyl sulphide; Action of water on —. C. Boulin and L. J. Simon. Comptes rend., 1920, 170, 815—818.

DICHLOROETHYL sulphide is slowly decomposed by cold water: $(\text{C}_2\text{H}_4\text{Cl})_2\text{S} + 2\text{H}_2\text{O} \rightleftharpoons [\text{C}_2\text{H}_4(\text{OH})]_2\text{S} + 2\text{HCl}$. The decomposition is accelerated by rise in temperature and increase in the amount of water present. It is retarded by the presence of acids and alkalis and by the products of decomposition. The solubility of dichloroethyl sulphide in cold water is 0.48 gm. per litre and it is increased by the presence of thiodiglycol.—W. G.

Liquids having constant boiling points; Certain binary and ternary mixtures of —. W. R. G. Atkins. Chem. Soc. Trans., 1920, 117, 218—220. THE undermentioned binary mixtures exhibit con-

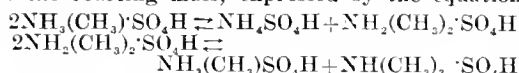
stant minimum boiling points: Acetone—carbon tetrachloride, 55.9° C. at 763 mm.; methyl ethyl ketone—carbon tetrachloride, 73.8° at 760 mm.; methyl ethyl ketone—*tert*-butyl alcohol, 79.0° at 762 mm.; methyl ethyl ketone—*isopropyl* alcohol, 77.3° at 759 mm.; methyl alcohol—carbon bisulphide, 37.1° at 751 mm.; carbon tetrachloride—*tert*-butyl alcohol, 71.0° C. at 768 mm. The ternary mixture methyl ethyl ketone—carbon tetrachloride—water has a constant boiling point at 65.7° C. at 760 mm. with the composition 22.2%, 74.8%, and 3% of its constituents respectively. The mixture carbon tetrachloride (85%)—*tert*-butyl alcohol (11.9%)—water (3.1%) boils constantly at 64.7° C. and 768 mm. pressure.—G. F. M.

Nitriles; Preparation of unsaturated — by catalysis. A. Mailhe. Bull. Soc. Chim., 1920, 27, 226—229.

UNSATURATED aliphatic or aromatic nitriles, such as crotononitrile, oleonitrile, elaidonitrile, and cinnamonitrile may be prepared by passing the vapours of the esters of the corresponding acids along with ammonia over alumina at 480°–500° C. as previously described (J., 1920, 349A).—W. G.

Methylamine; Preparation of — from ammonium methyl sulphate. W. S. Denham and L. F. Knapp. Chem. Soc. Trans., 1920, 117, 236—247.

THE product of the action of heat on ammonium methyl sulphate always contains dimethylamine and trimethylamine in addition to methylamine (cf. Werner, Chem. Soc. Trans., 1914, 105, 2762). Under no conditions is more than 25–30% of the ammonia converted into methylamine. Support for the view that a state of equilibrium is attained in the reacting mass, expressed by the equations



is derived from the fact that methylamine is formed when a mixture of the sulphates of ammonium, dimethylamine, and trimethylamine are heated together. Moreover, by increasing the reacting mass of ammonium sulphate by heating 2 equivalents of it, or preferably ammonium benzenesulphonate because of its greater fusibility, with ammonium methyl sulphate the yield of methylamine is increased to nearly 50% of the theoretical.—G. F. M.

Chlorhydrins and glycols; Preparation of ethylene and propylene — from oil-gas. B. T. Brooks. Chem. and Met. Eng., 1920, 22, 629—633.

IN the treatment of olefines with hypochlorous acid, it is unnecessary to use solutions of greater concentration than 0.1% total hypochlorite, and formation of chlorhydrin is accelerated by increasing the solution of the gas in the aqueous solution by agitation or moderate pressures. The author employed an apparatus of the liquid-circulating type, the free hydrogen chloride being neutralised by soda ash, sodium bicarbonate, or borax. The formation of chlorhydrin was fairly rapid, until the solution contained about 5% chlorhydrins, after which it proceeded more slowly. Propylene reacts more readily than ethylene. With a larger apparatus, using about 600 lb. of chlorine and 1000 galls. of solution, at a temperature of 15° C., and under 75 lb. pressure, yields of 4.55–7.75% chlorhydrin were obtained from oil-gas containing 34–36% total ethylene and propylene. Graphs are given indicating the behaviour on distillation of 24% ethylenechlorhydrin solution and of 24% and 70% ethylene- and propylene-chlorhydrins mixed. The effect of saturating a 24% solution of the chlorhydrins with common salt is also shown. Nearly theoretical yields of glycol are obtained from ethylenechlorhydrin. By treatment of a 20.8%

aqueous solution of ethylene- and propylene-chlorhydrins with sodium bicarbonate, the yield was 81.5% of the theoretical; when sodium carbonate was used, only 74.2% was obtained. (Cf. J., 1919, 923A).—W. J. W.

Marsh gas [methane]; Interaction of chlorine and — under the influence of light. Conversion of methyl chloride to methyl alcohol and methyl acetate. J. R. H. Whiston. Chem. Soc. Trans., 1920, 117, 183—190.

THE photochemical reaction between chlorine and methane never stops at the first stage to give methyl chloride exclusively, but a mixture of all the chloromethanes is produced in which methylene dichloride and chloroform predominate. In a mixture of 2½ vols. of methane to 1 vol. of chlorine only 25% of the chlorine forms methyl chloride, and with equal volumes only 10–13%. Methyl chloride can be completely converted into methyl acetate by passing it over porous sodium acetate at 290°–297° C., whilst slaked lime at 300° C. converts it almost quantitatively into methyl alcohol.

—G. F. M.

Acetylene; Action of nitric acid on —. K. J. P. Orton and P. V. McKie. Chem. Soc. Trans., 1920, 117, 283—296.

ACETYLENE is readily absorbed by absolute nitric acid or mixtures of nitric and sulphuric acids. The rate of absorption is decreased by diluting the nitric acid, and increased by certain catalysts, and by increase of temperature. The above-mentioned conditions also largely determine the nature and proportion of the products, the most important being nitroform and other substances which on further nitration yield tetranitromethane, carbon dioxide, and other substances, including oxalic acid which cannot be converted into tetranitromethane. High (100%) or low (70%) concentration of nitric acid, low temperature (0°–15° C.), and certain catalysts, notably platinum and silver, favour the production of the oxalic acid type of product, whilst 95% nitric acid, a temperature of about 30° C., and notably the use of mercury as a catalyst, cause nitroform and allied substances to predominate. The addition of 0.3–0.4% of mercuric nitrate to the nitric acid practically eliminates oxalic acid etc. from the product, and increases the percentage of the carbon in the acetylene employed converted into nitroform etc., to about 30%, the remaining 70% going to carbon dioxide. Nitroform in the product represents only 80–85% of the total substances, which on heating the reaction mixture with fuming sulphuric acid to 90°–95° for 4 hours are converted into tetranitromethane. These other substances are not very stable, and when the object is the preparation of tetranitromethane it is important to proceed with the further nitration, or at least to add the fuming sulphuric acid without delay. On cooling the nitrating mixture tetranitromethane separates as an oil, or sometimes as crystals, and is isolated by extraction with carbon tetrachloride or by distillation. It has m.p. 13.75° C., and sp. gr. 1.65009 at 15.8° C. The best yield observed was 22.3 g. from 4.6 litres of acetylene and 90 c.c. of absolute nitric acid diluted to 95% with water, the absorption being carried out at 30° C. in the presence of mercuric nitrate.—G. F. M.

Oxalates; Specific colour reaction of —. V. Macri. Boll. Chim. Farm., 1920, 59, 73.

THE colour reaction for oxalates described by Caron and Raquet (J., 1919, 662A) was published earlier by the author (J., 1917, 1176) as a reaction for detecting manganese salts. Addition of an oxidising agent is unnecessary, since agitation of a hot solution of a manganese salt with a little alkali

causes absorption of atmospheric oxygen and formation of higher oxides of manganese, these giving the red coloration with an oxalate. The reaction is not given in acid solution.—T. H. P.

Mercury; Determination of — in organic compounds. A. Wöber. Z. angew. Chem., 1920, 33, 63–64.

THE mercury may be obtained in solution, in a suitable condition for its determination by Rupp's iodometric method (J., 1908, 1179), by heating 0.5 g. of the substance with sulphuric acid at 60° C. in a flask fitted with a tapped funnel and a delivery tube connected with a Peligot tube which is cooled with water; 1 c.c. of concentrated hydrogen peroxide solution is added, drop by drop, to the contents of the flask, and the heating is continued until a clear solution results and sulphur trioxide fumes appear in the Peligot tube. The solution is then cooled, diluted, treated with 1 g. of sodium chloride, neutralised with sodium hydroxide solution (sp. gr. 1.3), mixed with the rinsings from the Peligot tube, and diluted to 100 c.c.—W. P. S.

Myricyl alcohol. Gascard. See XII.

PATENTS.

Amines; Process of substituting halogens for the amino groups in aliphatic —. Chem. Fabr. Flora, Dübendorf, Switzerland. Eng. Pat. 132,245, 25.8.19. (Appl. 20,863/19.) Int. Conv., 31.8.18.

THE amino-groups of aliphatic amines may be replaced by chlorine or bromine by treatment at 50°–100° C. with a mixture of nitric acid (sp. gr. 1.4) and concentrated hydrochloric or hydrobromic acid respectively. The reaction is accompanied by vigorous evolution of nitrogen, and proceeds without appreciable formation of by-products. It is particularly applicable technically to the aliphatic amino-acids or their salts; for example, *l*-leucine yields *l*- α -chloro (or bromo) isobutylic acid.

—G. F. M.

Ethyl alcohol and ethyl acetate; Synthetic manufacture of —. P. V. H. Pascal, Angoulême, Franco. Eng. Pat. 140,115, 31.5.18. (Appl. 9019/18.) Int. Conv., 3.4.17.

ETHYL alcohol is obtained by the electrolytic reduction of acetaldehyde or paraldehyde in acid medium, preferably 5–10% sulphuric acid, in a cell divided by a porous diaphragm, the aldehyde being introduced into the cathode chamber in two stages, so that the cathodic liquid contains initially about 10%, and finally 30% or more of organic material. The cathode may be of lead, antimonial lead, or mercury, and a current density of 2–3 amps. per sq. decm. is employed, which is sufficient to maintain the temperature at about 40° C. Instead of using aldehyde, acetylene may be converted into alcohol in one operation by introducing the gas into the acid electrolyte containing a mercury salt, whereby hydration and reduction occur simultaneously. By increasing the temperature, the density of the current, the percentage of acid or aldehyde, or the duration of reduction appreciable quantities of secondary products are formed, such as butanediol-1,3, crotonyl alcohols, butyl alcohol, ethyl ether, etc., and by suitable selection of conditions one or more of these may be made to predominate. Similarly by diminishing the efficiency of the diaphragm ethyl acetate is formed by combination of the alcohol formed at the cathode with acetic acid formed at the anode. Oxygen or ozonised oxygen is obtained as a by-product in all these operations.—G. F. M.

Pentaerythritol; Treatment of solutions containing crude —. Rheinisch-Westfälische Sprengstoff-A.-G., Cologne. Ger. Pat. 298,932, 21.6.14.

A solution containing crude pentaerythritol is neutralised and concentrated; the calcium formate which separates from the hot solution is removed, and pentaerythritol crystallises as the solution cools.—L. A. C.

Monobromoacetone; Manufacture of —. Manufacture of monobromoketones. J. D. Riedel, A.-G., Berlin-Britz. Ger. Pats. (A) 298,944, 15.9.15, and (n) 298,953, 15.10.16.

SOLUTIONS of bromine in bromide liquors are added at raised temperatures to bromide liquors containing (A) acetone and bromoacetone, or (n) a ketone or mixture of ketones and bromoketones, prepared by neutralising the aqueous solutions obtained in previous preparations of monobromoacetone and monobromoketones respectively.—L. A. C.

Glycol; Production of — from ethylene. Chem. Fabr. Griesheim-Elektron, Frankfurt. Ger. Pat. 300,122, 22.2.16.

By avoiding prolonged contact of ethylene with the oxidising agent (potassium permanganate), *e.g.*, by applying the principle of counter-currents, and by avoiding the accumulation of manganese dioxide mud and other materials which would tend to retain glycol, an almost quantitative yield of the latter is obtainable. Ethylene produced by the catalytic hydrogenation of acetylene can be advantageously used, in which case the accompanying ethane is obtained as a by-product.—D. F. T.

Succinylsuccinic acid esters; Manufacture of —. R. Willstätter, Munich. Ger. Pat. 300,672, 27.1.17.

SUCCINYLSUCCINIC acid esters, for use in the synthesis of tropine derivatives, are prepared by electrolysis of neutral or feebly acid solutions of acetonedicarboxylic acid esters. The secondary potassium salt of acetonedicarboxylic acid ester, produced by the action of concentrated aqueous or alcoholic potassium hydroxide on acetonedicarboxylic acid ester, yields succinylsuccinic acid diethyl ester (colourless needles, m. p. 46°–47° C.) on electrolysis; this gives pyrrole derivatives, *e.g.*, *N*-methylpyrrolediacetic acid ester (prisms, m. p. 164° C.) on treatment with ammonia or amines. Acetonedicarboxylic acid monomethyl ester yields less readily the corresponding succinylsuccinic acid methyl ester, from which *N*-methylpyrrolediacetic acid dimethyl ester (needles, m. p. 170°–171° C.) is obtained.—L. A. C.

Rectification of ether; Apparatus for —. Maschinenbau-A.-G. Golzern-Grimma, Grimma. Ger. Pat. 305,172, 19.9.16.

THE formation of by-products, losses due to evaporation or deficient conversion of alcohol, and increase of pressure due to too rapid supply of ether vapour are prevented by providing an intermediate or compensating column furnished with a condenser, through which the crude ether vapour passes on its way to the rectifying column.

—J. S. G. T.

Hydrocarbons and organic oxygen compounds; Preparation of — from the chlorinated products of mineral oils. F. Bergius, Hanover, and L. Landsberg, Nuremberg. Ger. Pats. (A) 309,281, 23.9.13, and (n) 309,282, 26.4.16.

(A) THE chlorinated products are heated under pressure with dilute aqueous alkalis, whereby the chlorine is completely removed. Chlorinated paraffin hydrocarbons of sp. gr. 0.790 and containing approximately 25% Cl yield an oily, insoluble mixture of terpene-like hydrocarbons and oxygen

compounds which may be used as a substitute for oil of turpentine; the aqueous liquid contains phenolic and acidic products. (b) By using alkali carbonates instead of dilute caustic alkali the formation of charred or tarry products is avoided; catalysts such as copper or copper salts may be simultaneously applied. The oily product has a higher specific gravity than the material originally chlorinated, and may be used as a solvent, *e.g.*, for the recrystallisation of trinitrotoluene.—D. F. T.

Germ-free virus; Process for obtaining —. Quarzlampen-Ges.m.b.H., Hanau. Ger. Pat. 315,101, 4.6.14.

VACCINE virus, obtained by the process described in Ger. Pat. 314,859 (this J., 1920, 312 A), is mixed with a very dilute solution of a photo-dynamical substance and subjected to the action of chemically active rays. Accompanying micro-organisms are destroyed by about 5 minutes' treatment without damage to the virus. Methylene Blue, Methylene Violet, Nile Blue, phenazine, Neutral Red, phenosafranine, Fluorescein and several of its derivatives, acridine, and chrysophanic acid are among the dyes mentioned as suitable photo-dynamic substances, as well as chlorophyll, uranium salts, and iron salts.—B. V. S.

Piperidine piperidylthiocarbamate; Preparation of —. Farbenfabr. vorm. F. Bayer und Co., Leverkusen. Ger. Pat. 316,009, 12.10.17.

PIPERIDINE piperidylthiocarbamate, for use as a vulcanisation catalyst, is prepared by the action of carbon bisulphide on an aqueous solution of piperidine at a low temperature; the presence of the water moderates the reaction.—D. F. T.

Hog-cholera serum; Process of producing refined —. J. Reichel, Assignor to H. K. Mulford Co., Philadelphia, Pa. U.S. Pat. 1,335,986, 6.4.20. Appl., 3.4.14.

SEE Eng. Pat. 6604 of 1915; J., 1916, 653.

Acids from wood tar etc. Ger. Pat. 315,417. See III.

Cymene. U.S. Pat. 1,336,694. See V.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photomicrography with simple apparatus. T. J. Ward. Analyst, 1920, 45, 130—133.

For magnifications up to about $\times 4$ the camera is fitted with a short-focus (10 to 12 cm.) single meniscus photographic lens, whilst by placing a large diameter lens on a support between the camera lens and the object magnifications up to about $\times 8$ may be obtained. For magnifications up to about $\times 75$ the camera lens is replaced by a microscope objective fitted into a cork. For most purposes "ordinary" plates are the most suitable.

—C. A. M.

Action of coal upon a photographic plate. Sinkinson. See IIa.

PATENTS.

Strong [photographic] prints from weak negatives; Process for producing —. G. Staess, Berlin-Steglitz. Ger. Pat. 316,087, 1.5.17.

A THIN film of transparent gelatin or celluloid, coloured red by means of potassium ferric oxalate and ammonium thiocyanate, is interposed between the negative and the printing paper. This is bleached as printing proceeds in inverse proportion to the density of the negative, and the effect of printing under a steeper negative is obtained. The bleached film regains its colour on storing in the dark. The amount of intensification obtained may be reduced by partial bleaching of the film before printing or increased by using two or more films.

—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Nitrocellulose from wood pulp. R. G. Woodbridge, jun. J. Ind. Eng. Chem., 1920, 12, 380—384.

DIFFICULTIES experienced in the nitration of wood pulp in crêpe paper form were that the nitrated material tended to gelatinise, and that this and the shortness of the fibre rendered it difficult to remove the acid from the nitrocellulose in the centrifugal wringer. The yield of nitrocellulose was very low, only about 1.2 lb. per lb. of wood cellulose, as compared with 1.52 lb. from cotton cellulose. The consumption of acid was high, and the material was difficult to dehydrate. The viscosity of wood pulp nitrocellulose was much lower than that of cotton nitrocellulose. It was found, however, that satisfactory results could be obtained by the use of mixtures of cotton fibre with at least 50% of wood pulp, the most suitable material being sulphite pulp from spruce or mildly bleached soda pulp from pine instead of a deciduous wood. The mixtures gave no material difficulty in nitration, the acid recovery was normal, and the yield of nitrocellulose about 1.50 lb. per lb. of cellulose, indicating a yield of about 1.48 lb. per lb. of wood cellulose. In preparing the nitrocellulose the dehydration required a somewhat longer time. For nitrating wood pulp alone additional purification would be essential, and would add to the cost of the product. (Cf. J., 1919, 555 A, 602 A.)—C. A. M.

Nitrocellulose and inorganic nitrates; Estimation of nitrogen in — with the nitrometer. E. G. Beckett. Chem. Soc. Trans., 1920, 117, 220—235.

NITROMETRIC estimation of nitrogen in nitrocelluloses and, to a lesser extent, in inorganic nitrates, always gives low results, and the conclusion previously reached (J., 1914, 628) that with 96% sulphuric acid the various errors compensate one another is now shown to be incorrect. The interval of time which is allowed to elapse between the introduction of the nitrate and sulphuric acid into the nitrometer and the shaking has a great influence on the results, particularly with the nitrocelluloses, the diminution in volume of crude nitric oxide obtained being the more pronounced the stronger the sulphuric acid and the longer the interval. As the result of analyses of the crude gas evolved under varying conditions and of the residual sulphuric acid, whereby the necessary correction was calculated, the best results were found to be obtained by using 15 c.c. of 92.5—94.0% sulphuric acid and by allowing $\frac{1}{2}$ to $\frac{3}{4}$ hour to elapse between the introduction of the nitrocellulose and acid into the nitrometer and the shaking. In this case the error is about -0.7% of the total nitrogen found.

—G. F. M.

PATENTS.

Explosive mixture. R. L. Hill, Tamaqua, Pa., Assignor to Atlas Powder Co., Wilmington, Del. U.S. Pat. 1,334,303, 23.3.20. Appl., 30.8.18.

AN explosive mixture consists of ammonium perchlorate, nitro-starch, and a metallic compound capable of combining with chlorine.—W. J. W.

Solvent from smokeless powder; Process for recovering —. E. du Pont, Wilmington, Del. U.S. Pat. 1,334,360, 23.3.20. Appl., 13.6.19.

IN recovering from smokeless powders a mixed solvent, the constituents of which have different boiling points, the solvent is slowly vaporised, a portion of the vapour is cooled to condense only the vapour of higher boiling point, the uncondensed vapour is returned to and passed through the material, and another portion of the vapour is cooled and condensed.—L. A. C.

Explosive compounds. Atlas Powder Co., Wilmington, Del., Assignees of R. L. Hill and A. J. Strane, Tamaqua, Pa., U.S.A. Eng. Pat. 134,523, 1.9.19. (Appl. 21,438/19.) Int. Conv., 1.11.18.

SEE U.S. Pat. 1,307,495 of 1919; J., 1919, 604 A.

Displacement of liquids in nitrocellulose. Ger. Pats. 305,512 and 307,701-2. See XI.

XXIII.—ANALYSIS.

Finger-prints; Detection of — on documents. C. A. Mitchell. Analyst, 1920, 45, 122—129.

For the mechanical development of finger-prints upon paper good results may be obtained by the use of various dry powders, such as graphite, red lead, cadmium iodide, methylene blue, etc. Forgeot's ink method is based upon the fact that there is normally a slight secretion of oil on the fingers, which repels the liquid from the parts of the paper on which the papillary ridges have rested. Variable results obtained by different workers are attributable to variations in the composition of the ink. A suitable ink for the purpose is obtained by dissolving pyrogallol in 1% osmium tetroxide solution, which behaves like iron salts in forming an ink with substances containing three hydroxyl groups in juxtaposition. The reaction also affords a sensitive test for osmium tetroxide. The most sensitive vapour-reagent for finger-prints is iodine, but mercuric iodide vapour gives more stable colorations. Osmium tetroxide vapour gives permanent colorations with recent finger-prints, but is less trustworthy than iodine vapour. Conversely, the reaction may be used for detecting osmium tetroxide, a slightly greasy finger-print being exposed to the vapour.—C. A. M.

Moisture in coal etc. Myhill. See II A.

Benzol in gas etc. Shuttleworth. See II A.

Hydrocarbons. Tausz and Peter. See II A.

Paraffin in ceresin. Smelkus. See II A.

Sulphuric ions in complex compounds. Job and Urbain. See VII.

Hydroxyl ion concentration. Fricke. See VII.

Hypiodous acid. Colman. See VII.

Boric acid. Van Liempt. See VII.

Carbon in iron. Yensen. See X.

Carbon in ferro-alloys. Rottmann. See X.

Soap. Weber. See XII.

Carbon black. Perrott and Thicssen. See XIII.

Glue jellies. Low. See XV.

Tensile strength of glue. Hopp. See XV.

Sucrose. Jackson and Gillis. See XVII.

Sucrose. Rolfo and Hoyt. See XVII.

Sucrose in molasses. Brodie. See XVII.

Red wines. Macmillan and Tingle. See XVIII.

Oxalic acid in vinegar. Bau. See XVIII.

Mercury in organic compounds. Wöber. See XX.

Oxalates. Maeri. See XX.

Nitrocellulose and nitrates. Beckett. See XXII.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Andrew, Miller, Trevelyan, and Armstrong, Whitworth and Co. Furnaces burning powdered fuel. 12,471. May 5.

Blagden, and Howards and Sons. Manufacture of catalysts for dehydrogenation, and application of such catalysts. 12,513. May 5.

De Laval Separator Co. Centrifugal separator. 12,631. May 6. (U.S., 31.8.17.)

Eustis. Recovery of gases. 12,368. May 4.

Fairweather (Lysekils Mekaniska Verkstads Aktiebolag). 11,661. See XIX.

Gröndal. Apparatus for lixiviating granular or pulverulent material. 11,927. Apr. 29. (Sweden, 29.4.19.)

Hamer. Producing high temperatures in heat-energy carrying fluids. 11,531. Apr. 26.

Harris. Drying apparatus. 12,009. Apr. 30.

Leigh and Little. 11,785. See II.

Merry. Evaporation of liquids. 12,454. May 5.

Merry. Utilisation of natural thermal energy. 12,601. May 6.

Nauerz. Condensing gases and gaseous acids. 11,676. Apr. 27. (France, 30.4.19.)

Sutton. Apparatus for effecting reactions between gases. 12,249. May 3.

Thornycroft. Apparatus for extraction of juice from vegetable substances. 11,822. Apr. 28.

Union Espagnole de Fabriques d'Engrais, de Produits Chimiques, et de Superphosphates. Roller crushing machines. 11,489. Apr. 26. (France, 9.3.14.)

Wade (Norton Co.). Grinding machines. 12,255. May 3.

Walpole. Extraction apparatus. 11,571. Apr. 26.

Weyers. Downdraught kilns, ovens, etc. 11,518. Apr. 26. (Holland, 2.5.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

2004 (1919). Ablett. Method of subjecting material to chemical treatment and grinding or/and mixing and apparatus therefor, also ball mill and combined grinding or/and mixing and vacuum drying apparatus. (142,176.) May 12.

3156 (1919). Arter. Grinding machines. (141,831.) May 5.

4959 (1919). Van Norman. Grinding machines. (141,851.) May 5.

5975 (1919). Laverack. Means or apparatus employed for drying excreta, mucilaginous, and other solid matters containing a large proportion of water. (141,861.) May 5.

7250 (1919). Davis. Apparatus for drying or cooling coal, limestone, and other substances or materials in granular form. (141,873.) May 5.

13,751 (1919). Durrans, and Boake, Roberts, and Co. Still-heads. (112,330.) May 12.

16,015 (1919). Conte. Process of and apparatus for separating solids from liquids. (128,941.) May 12.

25,018 (1919). Norsk Hydro-Elektrisk Kvaestof-aktieselskab. Effecting crystallisation from solution. (134,207.) May 12.

25,186 (1919). Gaillet. Combination of pyramidal surfaces for purification of liquids, vapours, and gases. (133,971.) May 12.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

- Barrett Co. 11,833. *See* XIII.
 Culver. Artificial fuel. 11,546. Apr. 26.
 De Laval Separator Co. Process for cleaning gasoline. 12,629. May 6. (U.S., 23.9.18.)
 Dobson and Pickard. Gas-producers. 12,271. May 4.
 Duckham, and Woodall, Duckham, and Jones. Gasification of coal etc. 11,802. Apr. 28.
 Engelke. Treating hydrocarbon oils. 11,679. Apr. 27.
 Fitzgerald. Utilisation of crude etc. petroleum oils etc. 11,581. Apr. 27.
 Lambert. Recovery of condensable gases or vapours from carbon. 12,709. May 7.
 Leigh and Little. Treatment of dust from gas-cleaning plants. 11,785. Apr. 28.
 Perry. Apparatus for distilling and briquetting carbonaceous material. 12,802. May 8.
 Twynam. Gasification of fuel. 12,741. May 7.
 Wilson. Method of treating and utilising carbonaceous materials with recovery of hydrocarbon oils contained therein. 12,500. May 5.

COMPLETE SPECIFICATIONS ACCEPTED.

- 21,021 (1918). Crush. Gas-producing plant. (141,771.) May 5.
 21,724 (1918). Christopher. Method of and apparatus for the distillation, carbonisation, or gasification of coal, carbonaceous material, oil shales, and the like. (142,163.) May 12.
 2706 (1919). Holt and Walker. Method of manufacturing gas from coal or like carbonaceous material. (141,828.) May 5.
 2825 (1919). Hack and Boughton. Apparatus for washing gas. (142,203.) May 12.
 2905 (1919). Mann. Process relating to the decomposition of hydrocarbons and other substances in the liquid and/or vapour phases. (142,206.) May 12.
 2974 (1919). Hurez. Coke ovens. (123,099.) May 12.
 20,002 (1919). Steinschneider. Distilling-boiler, more especially for the distillation of heavy hydrocarbons of the petroleum, lignite-tar, and coal-tar industry and the like. (142,376.) May 12.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

- Brégeat. Process for recovery of naphthalene and/or camphor from gaseous mixtures. 12,753. May 7.
 Drew and Morgan. Production of toluene derivatives. 12,470. May 5.
 Sutton. Manufacture of naphtholates and hydroxynaphthoic acids. 12,140. May 1.

COMPLETE SPECIFICATION ACCEPTED.

- 20,002 (1919). Steinschneider. *See* II.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

- Carpmael (Farbenfabr. vorm. F. Bayer and Co.). Manufacture of indigoid vat dyestuffs. 12,526. May 5.
 Carpmael (Farbenfabr. vorm. F. Bayer and Co.). Manufacture of azo dyes. 12,639. May 6.

COMPLETE SPECIFICATION ACCEPTED.

- 524 (1917). Vidal. Preparation of a black colouring-material. (141,759.) May 5.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

- Beaver and Claremont. Impregnating paper etc. with electrical insulating-compounds etc. 11,658. Apr. 27.
 Briggs, and British Cellulose and Chemical Manufacturing Co. Treatment of fibres and fabrics. 11,886. Apr. 29.
 Bronnert. Manufacture of artificial silk. 12,051 and 12,719. Apr. 30 and May 7.
 Burlin. Process of manufacture of paper etc. pulp from peat. 11,458. Apr. 26.
 Green. Cellulose acetate and nitrate compositions and light esters. 12,605. May 6.
 Summers. Process of retting flax etc. 12,106. May 1.

COMPLETE SPECIFICATIONS ACCEPTED.

- 14,688 (1919). Dreyfus. Manufacture of solutions of cellulose acetate and of celluloid-like masses, films, and other products or articles produced therewith. (128,215.) May 5.
 18,129 (1919). Robinson. Production of fibres from flax or the like. (141,982.) May 5.
 19,165 (1919). Duclaux. Process for the purification of nitrocellulose. (130,619.) May 5.
 27,209 (1919). Stulemeyer. Method and means for manufacture of artificial silk. (142,038.) May 5.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

- Euster and Olig. Machine for mercerising yarn. 12,382. May 4. (Germany, 10.7.19.)
 Pearce. Holders for dyeing cops etc. 12,298. May 4.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

- Busico. 12,750. *See* XV.
 Fairweather (Fredrikstad Elektrokemiske Fabriker Aktieselskabet). Process for manufacture of perborates by electrolysis. 12,701. May 7.
 Gibbs. Production of magnesium salts. 11,563. Apr. 26.
 Hauenschield. 11,814. *See* IX.
 Hoseason and Norris. Preparation of chlorine compounds, and methods of employing same. 12,655. May 7.
 Levy and Terrisse. Regenerating hydrochloric acid. 12,751. May 7. (Switz., 15.11.19.)
 Matheson. Manufacture of alum and sulphate of alumina. 12,628. May 6.
 Matheson. Manufacture of sulphuric acid. 12,630. May 6.
 Nauerz. 11,676. *See* I.
 Parrish, and South Metropolitan Gas Co. Manufacture of ammonium sulphate. 12,039. Apr. 30.
 Parrish, and South Metropolitan Gas Co. Manufacture of ammonium sulphides. 12,040. Apr. 30.

COMPLETE SPECIFICATIONS ACCEPTED.

- 1466 (1918). Soc. Industrielle de Produits Chimiques. Process for isolating or purifying alkali chromates. (119,647.) May 5.
 15,514 (1918). Soc. l'Air Liquide (Soc. Anon. pour l'Exploit. des Proc. G. Claude). Apparatus for the synthesis of ammonia at very high pressures. (142,150.) May 12.
 17,650 (1918). Heberlein. Method of and apparatus for electrolysing a solution of a nickel salt. (141,766.) May 5.

17,749 (1918). Maconochie and De Ros. Methods of and apparatus for the oxidation of tin and the production of tin oxide, the improvements, being also applicable for the treatment of zinc and zinc-containing materials for the production of zinc oxide. (142,157.) May 12.

693 (1919). Percival, and Edison Swan Electric Co. Production of argon. (111,783.) May 5.

1313 (1919). Linder and Lessing. Manufacture of sulphate of ammonia. (141,787.) May 5.

1427 (1919). South Metropolitan Gas Co., Evans, and Hollings. Manufacture of ammonium sulphate. (141,798.) May 5.

1428 (1919). South Metropolitan Gas Co., and Weight. Manufacture of sulphate of ammonia. (141,799.) May 5.

1744 (1919). South Metropolitan Gas Co., Parrish, and Vilon. Manufacture of neutral sulphate of ammonia. (141,819.) May 5.

2729 (1919). Norsk Hydro-Elektrisk Kvaestof-aktieselskab. Production of pure concentrated nitric acid and tetroxide of nitrogen. (124,191.) May 5.

6438 (1919). British Thomson-Houston Co. (General Electric Co.). Methods of and apparatus for fractionating air. (141,861.) May 5.

10,150 (1919). British Thomson-Houston Co. (General Electric Co.). Production of fluid halogen compounds of silicon, boron, or titanium. (141,908.) May 5.

11,793 (1919). Langwell. Manufacture of finely-subdivided barium carbonate. (141,925.) May 5.

13,009 (1919). Minton, and United Alkali Co. Apparatus for the manufacture of detached or flake caustic-soda or other matter required to be obtained in a state of division from a fused or molten mass. (141,942.) May 5.

16,359 (1919). Duchemin. *See* XVI.
1997 (1920). Soc. Industrielle de Produits Chimiques. Process for isolating or purifying alkali chromates. (138,111.) May 12.

VIII.—GLASS, CERAMICS.

APPLICATIONS.

Delannay and Dimitri. Process for manufacture of refractory and insulating products. 12,143 and 12,145. May 1. (France, 21.11.16 and 16.7.18.)

Lawley (Cambridge Glass Co.). Manufacture of glass. 12,111. May 5.

Norske Aktieselskab for Elektrokemisk Industri Norsk Industri Hypotekbank. Manufacture of porous material. 12,618. May 6. (Norway, 16.5.19.)

Somerville, and South Metropolitan Gas Co. Glass vessels containing wire to be heated to incandescence. 12,377. May 4.

COMPLETE SPECIFICATIONS ACCEPTED.

20,912 (1918) and 526 (1919). Adair. Drying of china clay, and arrangements connected therewith. (142,161.) May 12.

17,226 (1919). Langwell. Manufacture of glass. (142,365.) May 12.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Bakema. Blast furnaces for manufacture of cement etc. 12,827. May 8.

Hauenschild. Burning or calcining cement, magnesite, etc. 11,814. Apr. 28.

Henggeler and Hottinger. Process for production of hydraulic mortar. 12,316. May 4.

Stewart and Woolcock. Concrete products. 12,337. May 4.

Walton. Method of producing coloured effects on or in plaster of Paris, artificial stone, cements, etc. 12,200. May 3.

COMPLETE SPECIFICATIONS ACCEPTED.

5317 (1919). Sharratt. Brick-kilns. (142,237.) May 12.

18,772 (1919). Roy. Process and apparatus for rendering wood impermeable, and recovering liquids contained therein. (130,981.) May 12.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Amalgamated Zinc (de Bavay's), Ltd. Recovery of lead and silver from sulphide ores and metallurgical products. 11,831 and 12,589. Apr. 28 and May 6. (Australia, 17.5 and 6.5.19.)

Cowper-Coles. Electro-deposition of iron alloys. 12,662. May 7.

Cunningham. Process for coating aluminium etc. with nickel etc. 11,513. Apr. 26.

Electrolytic Zinc Co. Recovery of zinc by electrolysis. 11,934. Apr. 29. (Australia, 24.12.19.)

Emesco, Ltd. 12,456. *See* XI.

Kring and Stalhane. Coating metal objects with another metal. 12,214. May 3.

Walter. Alloys of metals of the ferro-chromium group and silicon or its compounds. 12,386. May 4. (Germany, 7.1.20.)

Walter. Method of producing alloys with metals of the ferro-chromium group and silicon or its compounds. 12,387. May 4. (Germany, 14.1.20.)

Walter. Method of producing alloys with metals of the ferro-chromium group and silicon or its compounds. 12,486. May 5. (Austria, 20.3.18.)

COMPLETE SPECIFICATIONS ACCEPTED.

17,749 (1918). Maconochie and De Ros. *See* VII.

1156 (1919). District Chemical Co., and Hoblyn. Mixtures for carburising steel during the case-hardening process. (141,802.) May 5.

6392 (1919). Gasche. Grading or concentration of ores and the like. (142,246.) May 12.

11,725 (1919). McKechnie, and McKechnie Bros. Method for separating or recovering nickel from cupro-nickel alloys, scrap, and the like. (142,310.) May 12.

11,868 (1919). Perez. Process for decomposing mercury, and obtaining radium and gold, which are contained in the formation of this metal. (126,961.) May 5.

12,372 (1919). Harris. Dezincing of lead. (142,315.) May 12.

14,161 (1919). Jarvis. Manufacture of steel. (141,956.) May 5.

16,849 (1919). Brettell (Snead and Co. Iron Works). Heat treatment of metals, and apparatus therefor. (142,358.) May 12.

25,316 (1919). Harris. Refining of lead. (142,398.) May 12.

29,038 (1919). Belais. Gold alloys. (142,018.) May 5.

29,302 (1919). Dorr Co. Treatment of fine ores. (135,844.) May 5.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Allen. Electrodes for secondary or storage batteries. 12,567. May 6.

British Thomson-Houston Co., and Ralph. Electric furnaces. 12,811. May 5.

British Thomson-Houston Co. (General Electric Co.). Devices for controlling electric discharges through gases and vapours, and methods of operating said devices. 12,225. May 3.

Cowper-Coles. 12,662. *See* X.

Delannay and Dimitri. 12,143 and 12,145. *See* VIII.

Electrolytic Zinc Co. 11,934. *See* X.
 Emesco, Ltd. Electro-chemical de-rusting and cleaning process. 12,456. May 5.
 Fairweather (Fredriksstad Elektrkemiske Fabriker Aktieselskabet). 12,701. *See* VII.
 Griffith. 12,156. *See* XIV.
 Hayward and Kluytmans. Electric resistance materials. 12,064. Apr. 30.
 Pouchain. Electrodes for electric batteries etc. 12,263. May 3.
 Pouchain. Negative electrode for electric accumulators. 12,264. May 3.
 Pouchain. Positive electrodes for electric accumulators. 12,833. May 8.
 Pouchain. Electric accumulators. 12,834. May 8.
 Soncini. Electric crucible ovens. 12,372. May 4. (Italy, 25.4.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

17,650 (1918). Heberlein. *See* VII.
 10,953 (1919). Marks (Armour Fertilizer Works). Polyphase electric-heating furnaces. (142,283.) May 12.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Busico. 12,750. *See* XV.
 Hey. Clarifying oil and grease solvents. 11,580. Apr. 27.
 Mattei. 11,914. *See* XIII.
 Tseng. Manufacture of soap. 12,066. May 7.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Barrett Co. Manufacture of resin from naphthas. 11,833. Apr. 28. (U.S., 5.5.19.)
 De Bruyn. Manufacture of paste, distemper, and colourwash. 11,495. Apr. 26.
 Griffith. 12,156. *See* XIV.
 Mattei. Solvent for varnishes, oils, stuccos, etc. 11,914. Apr. 29.
 Titan Co. Titanic oxide pigments, and production of same. 11,981. Apr. 30. (Norway, 1.8.19.)

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Griffith. Electrolytic manufacture of metallic sulphides for use as vulcanising pigments. 12,156. May 3.
 Heywood, and Isleworth Rubber Co. Process for acceleration of vulcanisation. 12,757. May 8.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Bamber and Owen. Tanning hides and skins. 12,067. May 1.
 Busico. Method of separating fat, ossein, and tricalcic phosphate of bones. 12,750. May 7. (Italy, 11.3.19.)
 Carmichael. Tanning. 11,969. Apr. 30.
 Schidrowitz. Adhesives. 11,669. Apr. 27.

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Abonos Graditicos Nitrificantes Soc. Anon., Gonzalez, Llanas, and Sanchez. Chemical manure. 12,507. May 5.
 Croft. 11,727. *See* XIX.
 Hamler. Fertiliser-dryer. 12,571. May 6. (U.S., 28.5.19.)

COMPLETE SPECIFICATION ACCEPTED.

16,359 (1919). Duchemin. Manufacture of cyanamide, and apparatus therefor. (132,354.) May 12.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

Classen. Manufacture of sugar and furfural from wood etc. 11,691. Apr. 27. (Norway, 18.3.19.)
 Levy and Terrisse. Obtaining glucose from wood. 12,609. May 6. (Switz., 13.5.19.)

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Campbell. Method of rendering alcohol undrinkable. 12,677. May 7.
 Classen. 11,691. *See* XVII.
 Levy and Terrisse. 12,609. *See* XVII.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Bell, Bell, and Rider. Preserving meat, fish, etc., and ventilating rooms. 11,726. Apr. 28.
 Bendixen. Method of making liquid milk from dried milk, and of making artificial milk from casein. 12,625. May 6.
 Bolton and Mills. Apparatus for aerating and circulating sewage etc. 12,165. May 3.
 Croft. Producing meal or flour from fish for use as food or fertiliser. 11,727. Apr. 28.
 Fairweather (Lysekils Mekaniska Verkstads Aktiebolag). Distilling water. 11,664. Apr. 27.
 Forget-me-not, Ltd., and Hutchinson. Process for ageing flour. 12,493. May 5.
 Fuessly, Hepworth, Wilton, and Wilton and Son. Method of treating and filtering water containing tarry matters. 12,650. May 7.
 Hort. Process for manufacture of sterilised milk etc. 11,656. Apr. 27.
 Kent-Jones, Watson, and Woodlands, Ltd. Manufacture of flour and bread. 12,260. 12,261. May 3.
 Lewin. Sewerage purification plant. 12,532. May 6.

COMPLETE SPECIFICATIONS ACCEPTED.

4096 (1918). Dick. Condensed-milk product. (141,763.) May 5.
 1837 (1919). Pape. Preserving organic materials. (142,169.) May 12.
 2382 (1919). Falk and Frankel. Method and apparatus for preserving animal and vegetable substances. (123,073.) May 12.
 13,819 (1919). Whitham. Process of and apparatus for manufacture of cocoa and chocolate. (141,953.) May 5.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Blagden, and Howards and Sons. 12,513. *See* I.
 Brégeat. 12,753. *See* III.
 Faber and Schaeffer. Manufacture of sulpho-thalassolic acid and its salts. 11,515. Apr. 26.

COMPLETE SPECIFICATION ACCEPTED.

4275 (1919). Selden Co., and Selden. Process of treating cymene. (142,226.) May 12.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATION.

Jeffreys. Photography. 12,564. May 6.

COMPLETE SPECIFICATIONS ACCEPTED.

11,417 (1919). Marks (Du Pont de Nemours and Co.). Composition for detonators. (142,303.) May 12.
 19,165 (1919). Duclaux. *See* V.

I.—GENERAL; PLANT; MACHINERY.

Metallic catalysts; Effect of surface oxidation on some — F. F. Rupert. J. Amer. Chem. Soc., 1920, 42, 402—411.

PLATINUM foil which has been used as an anode for the electrolysis of water acquires increased catalytic activity towards detonating gas (electrolytic hydrogen-oxygen mixtures), which is due to surface oxidation, and can be induced by chemical means, as well as by anodic polarisation. A similar effect was found in the case of palladium and gold. In the case of cobalt, a slight increase in activity was detected, but was probably due to an increase in the area of active surface by etching. The catalytic activity of smooth palladium towards the hydrogenation of cottonseed oil is increased by surface oxidation. Palladium is 50 times as active towards detonating gas as platinum, whilst platinum is 2000 times as active as gold, and 200 times as active as cobalt.—E. H. R.

Waste heat and its importance in future heat economy. E. Reutlinger. Z. Dampfkessel u. Maschinen-betrieb, 1919, 42, 377—378, 394—397, 401—404.

Production of low-temperature tar in steam power plants. Wilkens. See IIa.

PATENTS.

Heat treatment of chemical compounds [under pressure]; Apparatus for — and method for operation thereof. F. X. Govers. E.P. 129,962, 22.11.18. (Appl. 19,271/18.) Conv., 18.7.18.

TUBULAR heating apparatus is kept clean internally by means of balls which are fed continuously, one at a time, with the chemical under treatment, and withdrawn after the process is finished. By this means long, continuous tubes with no joints for cleaning purposes may be used. Should a catalyst be necessary for the desired reaction, the balls may be made of the catalyst. A suitable form of ball-feeding apparatus consists of two discs rotating above and below a distance piece, which is rather thicker than the diameter of the balls. Holes are made in each of the discs, but not opposite each other, also in the distance piece, through which one ball at a time can drop. To deliver the material to be heated, it is preferable to have a pump capable of giving a large excess pressure, so that if a ball comes to rest against an obstruction, the obstruction may be removed by the scouring action of the liquid or gas under treatment forced at high velocity between the ball and the wall of the tube.—B. M. V.

Roller [rod] mills. F. E. Marcy, Salt Lake City. E.P. 136,950, 5.2.19. (Appl. 2854/19.)

In a rod mill the discharge end is left open, permitting ready inspection and admission and withdrawal of the grinding rods, and devices are provided for retaining the grinding rods, while allowing free outlet of the ground material.

—B. M. V.

Vacuum filters; Continuous — A. E. Alexander. From United Filters Corp., Brooklyn, U.S.A. E.P. 141,001, 30.12.18. (Appl. 2581/20.)

To allow of withdrawing the filtrate and wash liquors separately, the central shaft supporting the segmental filter leaves of vacuum filters of the rotating leaf type is constructed of a number of tubes (parallel to the axis) communicating with the leaves by means of ports extending through segmental clamps and with the vacuum apparatus by means of a rotating valve.—B. M. V.

Filters; Continuous — A. E. Alexander. From United Filters Corp. E.P. 136,309, 30.12.18.

In a continuous vacuum filter with segmental filter leaves (U.S.P. 1,293,555; J., 1919, 276 A) means are provided for inflating the filter bags at an appropriate stage, and thus bringing them in contact with scrapers to remove the filter cakes.

Filter presses. P. W. Norman, Teddington, and The Aluminium Plant and Vessel Co., Ltd., Wandsworth. E.P. 140,715, 27.10.19. (Appl. 26,358/19.)

In filter-presses of the plate and distance frame type provided with a common longitudinal outlet duct formed by ports in the plates, the passages from the plate surfaces to the outlet duct are more easily cleaned if drilled on the slant, either a separate passage from each face, or one inclined passage extending from a larger transverse hole through the plate; this latter hole, being within the filtering area, may be partly filled by a suitable plug.

—B. M. V.

Filter-table; Rotary — E. L. Oliver, Oakland, Cal. U.S.P. 1,335,695, 30.3.20. Appl., 28.5.18.

A HORIZONTAL rotary pan is divided into a number of compartments, each having a filter bed, and discharge conduits lead from each compartment to a central valve plate. The valve is adjustable, so that suction may be applied to different groups of conduits in succession, and thus to filter beds, which are at different distances from the supply of unfiltered liquid.—W. F. F.

Filtration; Method of and apparatus for — O. J. Salisbury, Assignor to United Filters Corp., Salt Lake City. U.S.P. 1,336,444, 13.4.20. Appl., 29.8.17.

THE filter cake is dried *in situ* before removal from the filtering medium, by heat applied to it in the direction of the passage of the filtrate.—W. F. F.

Filtering machine [for gases]. H. W. Tuttle. U.S.P. 1,336,600, 13.4.20. Appl., 28.9.17.

Gas to be filtered is passed through a number of filtering devices which are agitated simultaneously. The filtering devices are cleaned by passing a reverse current of air through the chamber containing them, while the agitation is continued.

—W. F. F.

Filter. L. Wachenberg, New York. U.S.P. 1,337,170, 13.4.20. Appl., 5.4.19.

A HORIZONTAL filter drum is mounted on hollow trunnions, and liquid is delivered through one of the trunnions to the filtering elements within the drum. An annular conduit, surrounding the liquid conduit in the same trunnion, is connected to pipes supplying hot water and compressed air for cleaning the filtering elements. The cleaning fluid passes through the filtering elements in the reverse direction, and is also delivered simultaneously against the outer sides of the filtering elements. The drum is rotated during the cleaning operation.

—W. F. F.

Washing of granular filter material; Regulating the air supply in the intermittent — K. Morawe, Berlin-Friedenau. G.P. 315,270, 5.1.17. Addition to 311,593.

In the process originally described (J., 1919, 522 A) it is advantageous to be able to set the air valves automatically, as the duration of a wash is too short to admit of the regulation of an ordinary tap. Suitable regulating devices are described.

—H. J. H.

Rotary kilns, and method and means of heating same [by fluid or solid fuel]. H. Fenton, Batley, and A. W. H. Vivian, London. E.P. 140,514, 21.12.18. (Appl. 21,470/18.)

A COMBUSTION chamber, preferably of circular cross-section corresponding with the interior of the kiln, is built at the lower end of a rotary kiln, and a burner for oil or gas fuel extends into this chamber approximately in alignment with the axis of the kiln. In addition, passages on either side of the combustion chamber extend upwards from a pair of hearths for solid fuel.—B. M. V.

Furnaces for burning low grade fuel. F. Cotton, Sydney, N.S.W. E.P. 140,972, 12.9.19. (Appl. 22,444/19.)

THE fuel rests directly on the level front portion of the hearth of the furnace, the back portion of the hearth sloping steeply upwards to the fire-bridge. A deep bed of fuel is maintained, and air is supplied by induction by jets of superheated steam through conduits formed directly through the front of the furnace, also from the front along the thickness of the side walls, turning into the furnace at suitable places. The furnace may be divided into two or more compartments by feather walls which do not extend right up to the roof, and each of which carries outlets for the air blast on both of its faces.—B. M. V.

Tubular heating appliances of vacuum pans and other liquid heating apparatus. Fawcett, Preston and Co., Ltd. From H. W. Taylor, Mauritius. E.P. 140,635, 3.5.19. (Appl. 11,042/19.)

THE heating tubes in the bottom of the pan or vessel are bent into semi-circles and fastened into headers, which are provided with baffles to cause the steam or other heating medium to pass half round a circle and then back again several times. Small openings may be left for the direct passage of condensed water from the inlet at the top to the outlet at the bottom division of the header. Means of suspending the tubes are described.—B. M. V.

Cooling-towers or structures for cooling water and other liquids. F. E. Gill, Shipley, and The Davenport Engineering Co., Ltd., Bradford. E.P. 140,898, 24.3.19. (Appl. 7264/19.)

A WATER distributing apparatus for the tops of cooling-towers, that will not silt up, may consist of pipes or closed troughs with outlets in the upper parts of their circumference carrying rotary spraying nozzles, the water being supplied under pressure.—B. M. V.

Cooling gases; Apparatus for —. J. A. Reavell, and Kestner Evaporator and Engineering Co., Ltd. E.P. 141,097, 4.12.18. (Appl. 20,040/18.)

THE cooling liquid is passed through vertical U-tubes connected in groups to headers. A baffle is provided between the vertical limbs of the U, and the gas to be cooled is admitted around the top of one and exhausted at the top of the other limb, passing down and up along the tubes. Dust separated from the gas drops off the tubes to a chamber below the bend of the U, and its removal may be aided by rapping the tubes.—B. M. V.

Volatile solvents; Recovery of —. D. V. Plumbridge, Rugby. E.P. 141,210, 14.5.19. (Appl. 12,103/19.)

For the recovery of volatile solvents from a material such as fabric that has been treated with rubber solution, the drying apparatus is covered by a closely fitting hood, and air is circulated within the hood by one fan and the saturated air withdrawn to a condenser by the same or another fan. A cut-off valve is provided, which may be interconnected

with the means of starting and stopping the machine. The gases on the way to the condenser may be compressed, and the condenser may be provided with baffles to remove fog.—B. M. V.

Drying kiln; Vertical —. J. C. Brian, Belfast. E.P. 141,468, 1.3.19. (Appl. 5121/19.)

GRAIN or like material from a hopper is fed by a distributing cone on to a series of louvres so that the falling material forms the walls of a chamber, from which air is exhausted by a fan and delivered to a heating furnace either above or below the firebars. The air after reheating is returned to the outer chamber of the dryer which surrounds the falling material.—B. M. V.

Drying apparatus. F. V. Burman, Marine City. U.S.P. 1,336,422, 13.4.20. Appl., 25.6.19.

MATERIAL is fed into a horizontal rotating drum by a shoot at one end, and the heating medium is admitted into the drum at the same end. A heat-protecting wall is arranged behind the shoot.—W. F. F.

Crystallisation; Treatment of liquids to effect therefrom while the liquid is kept in motion. B. Bakke, Assignor to Norsk Hydro-Elektrisk Kvaestofaktieselskab, Christiania. U.S.P. 1,329,158, 27.1.20. Appl., 29.9.19.

THE solution is contained in a revolving tube, the top of which is kept at a relatively high temperature while the lower part is subjected to cooling.—A. J. H.

Vapours and gases; Method and apparatus for separating —. W. A. Patrick, B. F. Lovelace, and E. B. Miller, Baltimore. U.S.P. 1,335,348, 30.3.20. Appl., 28.12.18.

A MIXTURE of gases or vapours is passed into a porous material having pores similar in size to those of hydrated silica gel, and the gas or vapour retained in the material is afterwards liberated.—W. F. F.

Liquids; Apparatus for treating — with gases. W. Sieck, jun., and S. Drucker, Chicago. U.S.P. 1,335,398, 30.3.20. Appl., 16.6.19.

A CYLINDER with open ends is fixed, with its axis vertical, coaxially within a cylindrical container for the liquid. The gas is introduced into the liquid through a perforated pipe near the lower end of the open cylinder, and a helical blade is mounted on a rotating vertical shaft passing coaxially through the open cylinder so that a flow of liquid is produced in the cylinder in counter current to the flow of gas.—W. F. F.

Gas-vapour mixtures; Method of treating [liquefying] —. W. E. Lummas, Lynn, Mass. U.S.P. 1,336,360, 6.4.20. Appl., 5.5.16.

THE mixture of gas and vapour is passed into a water-cooled condenser, where part of the vapour is liquefied, and the liquid then passes through a water-cooled coil in a separate vessel. The cooled liquid and the uncondensed gas and vapour from the condenser are then passed in counter current through a column scrubber to recover any residual liquefiable vapour.—W. F. F.

Still and still-setting. H. Henderson, Port Arthur, Assignor to Gulf Refining Co., Pittsburgh. U.S.P. 1,335,438, 30.3.20. Appl., 14.3.16.

A STILL is supported on a framework which exposes the lower part to view, and is heated externally. Heat-insulating material is applied only on the exterior upper portion and the interior of the lower portion of the still, and an internal heating device is also provided.—W. F. F.

Surface condenser. S. M. Lillio, Philadelphia. U.S.P. 1,335,692, 30.3.20. Appl., 18.4.19.

A TUBULAR surface condenser is provided with additional tube-plates close to the main tube-plates and adjacent to the condensing space; all the tubes pass through holes in the additional tube-plates. Means are provided for withdrawing liquid and air from the narrow spaces between the two pairs of tube-plates.—W. F. F.

[Electrically] **classifying finely-divided material.** W. A. Schmidt, Assignor to International Precipitation Co., Los Angeles. U.S.P. 1,335,758, 6.4.20. Appl., 28.8.16.

PARTICLES of different size, suspended in a gas, are separated by passing the gas successively, at uniform velocity, through a series of electric fields of similar intensity and uniform cross-sectional area, so as to produce a succession of electrical actions on the gas. Particles, progressively smaller in size, are precipitated successively.—B. N.

Electrodes for use in electrical precipitation apparatus. Siemens-Schuckertwerke G.m.b.H. G.P. 316,703, 28.6.18.

GASES are passed through a chamber containing precipitation electrodes constructed of parallel sheets of net or gauze having a corrugated or zig-zag section, and high-potential electrodes arranged in the cavities formed by the corrugations of the sheets.—L. A. C.

Gases; Rotary apparatus for removing dust from —. Siemens-Schuckertwerke G.m.b.H. G.P. 316,901, 9.5.18.

GAS enters a vertical cylindrical chamber through an opening in the side, and is deflected by a plate which causes deposition of the coarser particles of dust; the gas is then drawn upwards through a rotating fan fitted with vanes of wire gauze, and passes out of the top of the chamber. The dust settles to the bottom of the chamber and is removed.—L. A. C.

Catalyst furnaces. H. C. Greenwood and W. R. Tate, London. U.S.P. 1,335,891, 6.4.20. Appl., 26.3.19.

A VERTICAL, cylindrical catalyst space is surrounded by a heat interchanger consisting of one or more annular partitions of heat-insulating material. The partitions are made of a cross section which tapers gradually from one end to the other to conform to the varying temperature gradient.—W. F. F.

Contact masses; Preparation of — for catalytic treatment of gases. Nitrogen-Ges.m.b.H. G.P. 312,726, 16.3.12.

THE catalyst is intimately incorporated with hydraulic cement, and the mass allowed to set without pressure; or the cement may be gauged, allowed to set, impregnated with a solution of the catalyst, and then dried. A contact mass composed of 85% cement, 10% copper oxide, and 5% alumina is effective in the oxidation of carbon monoxide at 210°–220° C. and most efficient at 360° C., whereas for contact masses with a clay or brick basis the corresponding temperatures are 650°–700° C. and 780°–825° C.—A. J. H.

Separating machine; Centrifugal —. R. E. Lapham, San Francisco. U.S.P. 1,336,968, 13.4.20. Appl., 8.6.16.

A ROTATING separating bowl is provided with a reservoir containing a separating medium which is caused to move over the inner surface of the bowl by its rotation. Pulp is supplied to the bowl, and is separated into its constituents according to their density. Separate outlets are provided for the pulp tailings and the separating medium, and they are

directed towards their respective outlets, after the separating operation, by a force acting in opposition to the outflow of the separating medium.—W. F. F.

Sedimentation apparatus. C. L. Peek, New York, Assignor to The Dorr Co. U.S.P. (A) 1,337,094 and (n) 1,337,095, 13.4.20. Appl., 5.10.18.

SETTLED sludge is removed from apparatus, such as a Dorr thickener, upwards through a vertical sleeve surrounding the central driving shaft. The material is continuously withdrawn from the sleeve or sludge well by means such as an air lift so that a hydrostatic head is maintained between the main part of the tank and the sludge well, and communication is effected between the two by means of (A) the open bottom of the sludge well or (n) a conduit extending from the closed bottom of the sludge well to near the bottom of the tank.—B. M. V.

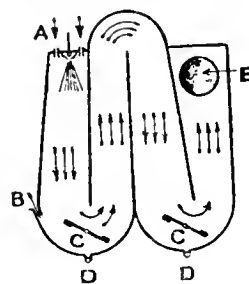
Refrigerating; Process of and apparatus for — F. W. Wolf, Assignor to The Isko Co., Chicago. U.S.P. 1,337,175, 13.4.20. Appl., 23.12.13.

THE apparatus includes in a closed circuit a compressor in which sulphur dioxide is compressed, an atmospherically cooled condenser for the compressed gas, and a chamber into which the condensed liquid is expanded.—W. F. F.

Tower for absorbing or concentrating gases. Chem. Fabr. Griesheim-Elektron. G.P. 305,122, 29.9.17.

A TOWER for absorbing gases, e.g., acids, in which the gas enters at the lower and passes out at the upper end and is washed by a counter-current of liquid, is constructed with double walls, the annular intermediate space being filled with packing, such as gravel, quartzite, etc., supported by a number of rings sloping towards the inner wall. In the annular chamber, which protects the outer walls of the tower and prevents loss of heat, the gases escaping from the inner chamber are again scrubbed by the liquid.—L. A. C.

Liquids; Chamber for drying —. L. Galland, Berlin-Wilmersdorf. G.P. 305,511, 21.10.17.



through D. The moist air escapes through E.—L. A. C.

Dehydration of mineral, vegetable, and animal products by means of electro-osmosis; Apparatus for —. Elektro-Osmose A.-G. G.P. 310,681, 4.7.17.

THE material is subjected to electro-osmotic treatment between a drum and an endless band, which serve as the respective electrodes. The drum and band rotate in the same direction, and the distance between the two steadily diminishes in the direction of motion. The band is pressed against the material under treatment by springs, and the distance between it and the drum is adjustable. The band may be made of brass webbing or of cloth overlaid with a deposit of metal. The band is cleaned by passage through water in an insulated trough.

—J. S. G. T.

Drying sludges of which the solid components are partly impermeable to water; Process for —. T. Steen, Charlottenburg. G.P. 314,595, 23.2.17. Addition to 314,043.

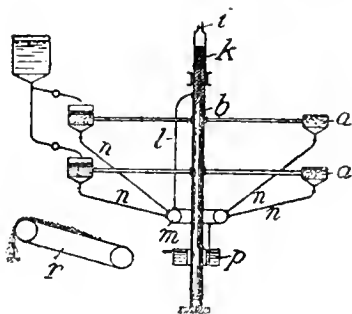
In the method previously described (J., 1919, 885 A) the impermeable material which penetrates into the permeable layer is removed from time to time by cuts of greater depth than usual. The filter bed is thus kept in working condition, without complete renewal, for a longer period.—L. A. C.

Sludge or the like; Apparatus for removing water from —. E. Techen, Leipzig. G.P. 315,312, 10.10.15. Addition to 278,258.

In the apparatus described in the chief patent (J., 1915, 213) the rotating drum is enclosed in a closed outer casing. Suction is applied as before through a contrivance within the drum, and filtration is assisted by application of pressure, e.g., by means of compressed air, to the annular space between the drum and the outer casing. Scrapers adjustable from the exterior are provided to remove the cake from the U-shaped trough, and the material is then forced by pressure through doors in the lower portion of the casing, which open and close automatically. By admitting water, steam, and hot air into the annular space the cake can be washed and dried before removal from the apparatus.—L. A. C.

Sludge; Apparatus for drying —. T. Steen, Charlottenburg. G.P. 315,553, 25.3.17. Addition to 314,043.

An apparatus for drying sludge by the process described in Ger. Pat. 314,043 (J., 1919, 885 A) consists of rotating filter beds, *a*, supported by a shaft,



b. The suction pipes, *n*, pass to a common holder, *m*, and suction is applied through *t*, *k*, and *i*; the holder, *m*, is connected to a water trap, *p*. The dried material from the filter bed falls on to an endless band, *r*, which conveys it away to be worked up for further use.—L. A. C.

Washing or chemical or mechanical treatment of materials. T. Steen, Charlottenburg. G.P. 315,780, 10.11.17.

The material is passed through a number of containers in which it is washed or treated with water or other liquid, the mixture passing from one container to the next through air-lift tubes. Overflows for liquid are provided between the separate containers, and these increase in cross-section and are arranged at progressively lower levels from the first onwards in the direction of motion of the liquid, to allow for the extra liquid introduced at different places in the series.—L. A. C.

Liquids; Method for agitating — by means of a magnetic field. G. W. Meyer, Jitschin. G.P. 316,499, 27.2.18.

A WHIRLING motion is imparted to liquids contained in an annular vessel by means of a rotating mag-

netic field set up between a primary inducer and a stationary or rotating body constructed of sheets of iron. The walls of the vessel are constructed of a material which does not interfere with the direction of the lines of magnetic force.—L. A. C.

Gaseous components; Preparation of solid chemical compounds [e.g., ammonium carbonate] from —. Badische Anilin und Soda Fabrik. G.P. 318,236, 9.7.15.

THE gases are led into chambers provided with a cooled rotating arrangement, and the solid substance produced is continuously removed by means of scrapers. To prevent separation of solid matter on the walls of the chambers, these may be lagged or, if necessary, heated. As an example, the preparation of ammonium carbonate and carbamate from ammonia, carbon dioxide, and steam is cited.—G. F. M.

Roller mill. F. E. Marcy. U.S.P. (A) 1,309,210, (b) 1,309,211, and (c) 1,309,212, 8.7.19. Appl., (A) 14.8.18, (b) 18.1.17, and (c) 26.9.17. (b) and (c) renewed 19.10.18.

SEE E.P. 136,950 of 1919; preceding.

Fractional distillation. G.P. 316,653. See III.

Centrifugal machine. U.S.P. 1,336,779. See XVII.

Separating liquids from solids. U.S.P. 1,308,918. See XIXB.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Peat; Determination of grade of —. G. Keppeler. Mitt. Ver. Förder. Moorkultur im Deuts. Reiche, 1920, [1]. Chem. Zentr., 1920, 91, II., 548.

THE degree of conversion to peat of mosses can be determined by decomposing the material by 72% sulphuric acid. The polysaccharides pass into solution, and the sugars can be estimated by titration with Fehling's solution. The dextrose estimated in this way, and calculated on the water- and ash-free peat, is termed the "total reduction." The maximum possible value is 68%, so that if the "total reduction" for any peat be *g*, then $100 - g \times 100 \div 68$ is a measure of the degree of conversion of the original vegetable matter into peat. This value is called the "degree of decomposition." The degree of conversion to peat may also be estimated from the residue after treatment with 72% sulphuric acid. From this residue (referred to 100 parts of organic matter) the ash and also the residuum left in the acid treatment of sphagnum moss (11%) are deducted in order to obtain the "degree of conversion." The newer moss peats generally show a low "degree of conversion," scarcely 30%, whilst the older ones give a much higher value, up to 50%.—A. G.

Tar and other by-products of coal distillation; Production of low-temperature — in steam-power plants. K. Wilkens. Mitt. Verein Elektrizitätswerke, 1919, 18, [241]. Chem. Zentr., 1920, 91, II., 384—385.

THE surplus heat of the steam boilers is utilised for the low-temperature distillation of coal. Between the boiler and the economiser a large number of retorts are built into which the coal is fed, and through which it is transported by screw conveyors, the blades of which are hollow and connected to a suction apparatus. After passing through a condenser the gas is led to a small gas-holder and is burned under the front end of the retorts. The coke is used in the boiler furnace.—G. F. M.

Combustion products or producer gas; Determination of the quantity of — from the analysis and from the calorific value of the coal. F. Cästek. Stahl u. Eisen, 1919, 39, 7—12.

THE author has elaborated a method of calculating the quantity of combustion products or water- or producer-gas in a mixture. By means of thermochemical equations for the complete combustion of pure carbon-hydrogen mixtures, the quantity of heat developed per cub. m. of burnt gases is expressed as a function of their carbon dioxide and oxygen content, and from this the amount of gas and its moisture content are calculated. Neglecting the sulphur and nitrogen present in coal, the pure coal substance can be represented as a carbon-hydrogen mixture, and the process is then applicable to coal. In the case of incomplete combustion (i.e., producer- or water-gas) the quantity of gas is calculated by considering that by complete combustion of the gases from 1 kg. of coal, the same amount of gaseous combustion product must be formed as if 1 kg. of coal were completely burnt direct, and the unburnt carbon in the ash, slag, coal dust, tar, and soot allowed for.—A. R. P.

Hydrocarbons; Catalytic oxidation of —. H. H. Franck. Chem.-Zeit., 1920, 44, 309—310.

By cracking aliphatic hydrocarbons in the presence of oxygen and a suitable catalyst carboxylic acids and other oxidation products are formed. Compounds of lead, manganese, mercury, vanadium, and chromium give good results as catalysts; alkalis and alkaline earths may also be used. For example, the oxidation of 0.5 kg. of hard paraffin wax in presence of 0.5—5% of a lead catalyst in an autoclave yielded, after 2 to 4 hours at 150° C., 460 g. of fatty acids. The mixed esters of these fatty acids yielded 18.75% of volatile (acetic to caproic) and 78.1% insoluble non-volatile acids (myristic to arachidic acids). A dark expressed residue of paraffin wax yielded 65% of fatty acids, the contents of the autoclave being bleached to pale yellow in the process. This product contained 40.7% of insoluble fatty acids in which there were only traces of stearic and arachidic acids. A sample of the mixed fatty acids esterified with glycol in the presence of 3% of sulphuric acid was converted into a solid fat with the odour of coconut oil. When refined this product was pale yellow and had the characters of a good edible fat.—C. A. M.

Spent oxide. See VII.

Heating steel furnaces with producer-gas and coke-oven gas. Schneider. See X.

PATENTS.

Fuel; Apparatus for treating —. W. L. McLaughlin, Decatur. U.S.P. 1,336,364, 6.4.20. Appl., 25.9.18.

COAL is crushed to pea size and dried by showering it through a hot gas zone, after which it is ground to an impalpable powder.—A. G.

Coke ovens and the like. Soc. de Fours à Coke et d'Entreprises Indus. E.P. 129,994, 16.7.19. (Appl. 17,805/19.) Conv., 16.7.18.

IN coke ovens having vertical flues in the walls and regenerators under the oven chambers the vertical flues are symmetrically divided in each heating wall into four groups, each pair being separated from the others by a transverse partition. The air in each of the two groups flows in an opposite direction at any given time, whilst short circuits and stagnant zones in the passages are avoided by dividing the regenerator under the oven chamber longitudinally into four sections, each communicating at the top with only one group of vertical flues, and being completely cut off from the other section.—A. G.

Peat; Carbonising oven for —. R. Schröter. G.P. 316,213, 30.3.16.

A HORIZONTAL carbonising chamber, heated from below, communicates with an adjacent shaft which extends vertically downwards, and in which the carbonised peat is saturated with tar and gaseous products carried by the gases passing down the shaft, so that little besides water vapour escapes into the chimney.—J. H. L.

Gas or gases; Manufacture of —. C. F. Broadhead, Brisbane. E.P. 122 179, 2.1.19. (Appl. 55/19.) Conv., 7.1.18.

A VERTICAL retort is superimposed on a water-gas generator, and coal is fed into the former by a gas-tight valve. Air and steam are blown alternately into the generator. During the air blow the gas valve is closed and the products of combustion pass through ports into an annular space surrounding the retort, the heat generated being transmitted through checker brickwork therein to the retort, and the waste gases passing through a snift valve to the atmosphere. During the steam blow the water-gas generated passes up through the vertical retort, and the heat of the water-gas, together with that transferred to the retort during the air blow, causes the coal to be subjected to low-temperature carbonisation.—A. G.

Water-gas; Production of — from coal carbonised in retorts. A. Peust, Berlin. G.P. 317,825, 9.2.19.

WHEN the evolution of gas from the coal is nearly completed, and whilst the coal is still incandescent, it is covered with a small charge of moistened, fine lignite. The temperature rises, and water-gas is produced without any external blast. The lignite is very soon gasified and carbonised.—J. H. L.

Illuminating gas and tar; Manufacture of —. H. Geyer, Marktredwitz. G.P. 317,977, 12.10.17.

COMMUNUTED coal is distilled in a rotary horizontal retort containing grinding bodies, e.g., balls of refractory material, and is subjected to the action of hot gases, poor in nitrogen. In this way local overheating is avoided.—A. J. H.

Purifiers employed in gas works. R. and J. Dempster, Ltd., and G. F. H. Beard, Manchester. E.P. 141,572, 30.6.19. (Appl. 16,363/19.)

IN gas purifiers employing conduits for directing the gas through the boxes (the conduits having walls forming part of those of the purifier), the valves are in open communication with the interior of the boxes and are situated within the boxes, adjacent to the conduits. (Cf. E.P. 844 of 1915; J., 1915, 897.)—A. G.

Waste liquor; Disposal of — in the recovery of ammonia from the gases produced in dry distillation etc. Phoenix A.-G. für Bergbau u. Hüttenbetrieb. G.P. 307,652, 19.4.14.

THE condensed liquor, after being freed from volatile ammonia, is concentrated in a tower through which a current of air is passed; part of the concentrated liquor is removed and the remainder is passed to the condensers to cool the hot crude gases, and is then returned to the concentrating tower. By this means the use of lime for recovering ammonia from the waste liquor and the use of clean water for cooling are avoided.—A. B. S.

Hydrocarbons; Rectification of —. De Bataafse Petroleum-Maatschappij, and J. H. C. de Brey, The Hague. E.P. 123,719, 7.11.18. (Appl. 18,270/18.) Conv., 1.3.18. Addition to 123,522 (J., 1920, 7A.)

MIXTURES of hydrocarbons are rectified under pressure (below 20 atm.), and the limits of temperature

may be selected so as to cause the elimination of worthless fractions from the liquid component and the removal of the last traces of valuable fractions from the gaseous component. For example, for compressor gasoline under 90 lb. pressure the maximum temperature may be 80° C. and the minimum -5° C. After this preliminary treatment the two components may be rectified.—A. E. D.

Liquid fuels; Production of —. H. Wade. From C. J. M. M. le Petit. E.P. 141,091. (Appls. 18,039, 4.11.18, and 19,134, 21.11.18.)

A MIXTURE of a formyl ester with one or more aliphatic amines is claimed for improving alcohol as a volatile fuel; e.g., such fuel may contain alcohol 65%, ether 34%, mono- or di-ethylamine 0.5%, and ethyl formate 0.5% by weight.—A. E. D.

Desulphurising sulphur-bearing hydrocarbons. Hall Motor Fuel, Ltd., and T. A. McCrea, London. E.P. 141,272, 8.9.19. (Appl. 22,094/19.)

THE vapour of the hydrocarbon is brought into contact with bauxite, clay, or the like at 150°–300° C. —A. E. D.

Lubricating composition; Process of making —. J. A. De Cew, Montreal. U.S.P. 1,336,207, 6.4.20. Appl., 9.10.17.

STABLE aqueous emulsions are prepared from mixtures of oils soluble and insoluble in water, containing a minimum of the former, by forcing the mixtures under pressure, in a fine stream, into hot water, and then forcing the resulting solution under pressure into cold water.—C. A. M.

Lubricating oil; Substitute for —. Tetralin G.m.b.H., Berlin. G.P. 309,178, 20.11.17.

THE lubricants claimed are obtained by polymerisation of hydrogenised naphthalene or naphthalene derivatives, or by combination of these with other organic substances. For example, tetrahydro-naphthalene condenses with benzyl chloride, chlorinated xylene, benzaldehyde, or trioxymethylene on heating or by means of condensing agents; the products are viscous oils of high boiling points. —J. H. L.

Lubricating and cooling agents soluble in water. Chem. Fabr. Flörsheim Dr. H. Noerdlinger. G.P. 316,720, 25.1.17.

AQUEOUS solutions of alkali compounds of the acid products formed by oxidation of wood tar (e.g., from beech, birch, or pine wood) are claimed. They may be added to other known lubricating and cooling agents, and are especially suitable for addition to the water in hydraulic plants, since besides their lubricating and cooling properties they act as disinfectants and tend to prevent rusting.—J. H. L.

Mineral oils; Apparatus for refining —. H. W. Jones, Assignor to The Kansas City Gasoline Co. U.S.P. 1,336,357, 6.4.20. Appl., 7.10.16.

A STILL or retort is composed of a number of vertical tubes close together, connected alternately at the top and bottom by short horizontal tubes so as to form a sinuous path for the circulation of oil. The vertical tubes project slightly beyond the connecting tubes at the top and bottom, the projecting ends being closed, those at the bottom by removable caps to retain deposits and heavy constituents out of the path of the circulating oil.—W. F. F.

Hydrocarbon oils and water; Apparatus for decomposing —. H. C. Wade, Assignor to Wadoll Corp., New York. U.S.P. 1,336,450, 13.4.20. Appl., 11.1.16.

OIL and water, suitably preheated, are heated in a spiral coil which contains "attenuated metal" at the outlet end of the coil.—A. E. D.

Liquid hydrocarbons; Process of converting —. W. C. McComb, Assignor to W. I. Patterson, New York. U.S.P. 1,337,144, 13.4.20. Appl., 15.10.18.

STEAM and the preheated oil are passed through a sinuous passageway of gradually increasing downward pitch towards a source of heat.—A. E. D.

Viscous oils; Process for the preparation of —. K. Börner. G.P. 318,034, 6.11.17.

HOT mineral oils are treated with an amount of chlorine sufficient to chlorinate any desired proportion of the oil, with or without the presence of a solvent; the product is heated above 200° C., air being drawn through at the same time.—B. V. S.

Feeding [powdered] fuel to combustion chambers; Means for —. V. Z. Caracristi, Assignor to Locomotive Pulverized Fuel Co., New York. U.S.P. 1,336,125, 6.4.20. Appl., 27.1.15.

Benzene hydrocarbons from coal-gas; Recovery of —. G. E. Darier. U.S.P. 1,336,893, 13.4.20. Appl., 10.7.16.

SEE F.P. 479,997 of 1915; J., 1917, 126.

Hydrocarbons; Treating — for production of lighter hydrocarbons. B. Andrews and W. C. Averill, jun. E.P. 141,223, 27.5.19. (Appl., 13,374/19.)

SEE U.S.P. 1,319,828 of 1919; J., 1919, 941A.

Sulphur dioxide from gases. E.P. 134,555. See VII.

Recovery of ammonia. E.P. 140,505. See VII.

Ammonium sulphate. E.P. 141,093. See VII.

Sludge acid. U.S.P. 1,309,206 and 1,337,141. See VII.

Nitric oxide in gas engines. G.P. 306,451 and 316,677. See VII.

Emulsifiable solid hydrocarbons. G.P. 308,442. See XII.

Carbon monoxide. G.P. 307,614. See XXIII.

IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Acetone, Losses in the preparation of — by the distillation of acetate of lime. L. F. Goodwin and E. T. Sterne. J. Ind. Eng. Chem., 1920, 12, 240–245.

THE results obtained by experimental distillations in an atmosphere of nitrogen indicate that the decomposition of calcium acetate is practically quantitative, and that the losses of acetone in practice must be attributed to defective apparatus. —C. A. M.

Charcoals; Some properties of —. H. E. Cude and G. A. Hulett. J. Amer. Chem. Soc., 1920, 42, 391–401.

WHEN the density of charcoal is determined by the immersion method in water or other liquid, a gradual increase in density is observed, due to the slow penetration of the liquid, the highest value, in water, being 1.900. (Cf. J.C.S., ii., 309.)—E. H. R.

Arc images in chemical analysis. Mott. See XXIII.

PATENTS.

Destructive distillation of wood, woody fibre, and similar carbonaceous substances. P. Poore, London. E.P. 141,417, 11.1.19. (Appl. 812/19.)

THE material is carbonised in a horizontal, rotary retort, mounted on a cradle in order to be easily

removed from the furnace for the removal of the residue and for recharging. A central vapour outlet pipe extends the length of the retort, and superheated steam is admitted through longitudinal pipes, adjacent to the walls of the retort.—A. G.

Destructive distillation; Process of —. O. F. Stafford, Eugene, U.S.A. E.P. 141,505, 14.4.19. (Appl. 9491/19.) Addition to 119,040.

Is addition to wood, other cellulosic or ligneous material, e.g. cottonseed hulls, rice hulls, peat, nut shells, fruit pips, straw, vegetable fibres, bagasse, moss, or the like may be carbonised (preferably in a finely divided state) by the process described in the main patent (J., 1919, 215A).—A. G.

Gas-mantle. L. Glaser. U.S.P. 1,335,831, 6.4.20. Appl., 11.10.17.

As automatic igniter consisting of pellets and bands composed of equal parts of platinum black and a silicious material is fused on to the body of an incandescence gas-mantle.—L. A. C.

III.—TAR AND TAR PRODUCTS.

Tar; Effect of superheating on the composition of —. M. Dolch. Montan. Rundsch., 1920, 12, 56–57, 78–79. Chem. Zentr., 1920, 91, 11., 545.

The properties of low-temperature tar produced from Kakani coal are compared with those of the same tar superheated by passing the tar vapours over glowing coke. The yield of tar diminished from 10.8% to 6.2%, the sp. gr. rose from 0.800 to 1.0374, and the free carbon content increased from 1.02% to 4.06%. The percentage of asphaltic matter rose from 31% to 57%, whereas the phenolic compounds decreased in quantity. The paraffins in the low-temperature tar amounted to over 6%, but there was practically no paraffin in the superheated tar. The yield of lubricating oils from the superheated tar amounted to 12%, as compared with 24% from the low-temperature tar, but the "neutral tar oils" increased in quantity from 15% to 24%. The asphaltic constituents of the heated tar were practically all liquid, and commenced to decompose at 348° C., whilst those from the low-temperature tar decomposed at 260°–270° C.—A. G.

Lignite; Uses of low-temperature tar oils from —. Harries. Verh. Vereins Förd. Gewerbell., 1920, 33–39. Chem. Zentr., 1920, 91, 11., 570.

HIGH-TEMPERATURE tars from lignite yield on extraction with liquid sulphur dioxide a highly-unsaturated extract, probably containing cyclic compounds, and a refined oil also containing unsaturated compounds. By ozonisation these products are converted into high-molecular fatty acids suitable for use in soap manufacture. Low-temperature tar yields similar products in the following proportions:—100 parts of lignite give 3 of tar, which can be separated into 1.4 of extract and 1.6 of a mixture of oil and paraffin. After removal of the paraffin, the expressed oil (0.64) yields 0.12 of crude fatty acids on ozonisation. (Cf. J., 1917, 539; 1919, 318 A).—L. A. C.

Coal-tar naphtha distillation test; Electric heater for the —. J. B. Hill and R. B. Chillas. J. Ind. Eng. Chem., 1920, 12, 280–281.

As iron pot is filled with solder to within a short distance of the top; two heating units are fitted at the bottom of the pot and are controlled by a double switch. One unit will heat the solder to 240°–280° C., whilst the two units together will maintain it at 345°–380° C. A ring of asbestos is floated on the surface of the solder, and on this is placed the distillation flask, the heat supplied to the contents

of the latter being regulated by the size of the hole in the asbestos ring and the depth to which the flask is immersed in the molten metal.—W. P. S.

Oxidation; Catalytic —. I. Benzene. J. M. Weiss and C. R. Downs. J. Ind. Eng. Chem., 1920, 12, 228–232.

The catalytic oxidation of benzene is discussed, particularly the formation of maleic acid by using vanadium dioxide as the catalyst (J., 1920, 9A, 44A); in this reaction, quinone is first formed and then oxidised to maleic anhydride and, finally, to carbon dioxide and water. The temperature appears to be the controlling factor in the oxidation.

—W. P. S.

Phenol; Manufacture of — in a continuous high-pressure autoclave. K. Brown. J. Ind. Eng. Chem., 1920, 12, 279–280.

CHLOROBENZENE and sodium hydroxide solution are pumped under a pressure of 2500–3000 lb. per sq. in. through a coil of hydraulic pipe immersed in a bath of melted sodium nitrate at 700° F. (about 370° C.); the resulting sodium phenoxide is discharged from the coil through a regulated needle-valve, condensed, acidified with hydrochloric acid, and the liberated phenol distilled. (Cf. Aylsworth, J., 1918, 229A).—W. P. S.

p-Dichlorobenzene; Some derivatives of —. J. H. Crowell and L. C. Raiford. J. Amer. Chem. Soc., 1920, 42, 145–152.

p-DICHLOROBENZENESULPHONIC acid is most conveniently prepared (yield 85–90%) by the action of sulphuric acid containing 10–12% SO₃ on p-dichlorobenzene at 140°–150° C.; 2,2',5,5'-tetrachlorodiphenylsulphone, (C₆H₂Cl₄)₂SO₂, m.p. 179° C., is obtained as by-product. 2,5-Dichloro-4-nitrobenzenesulphonic acid is obtained by nitrating the sulphonic acid with a mixture of fuming nitric acid (sp. gr. 1.6) and sulphuric acid (sp. gr. 1.84). Reduction of the nitro-acid in alkaline or weakly acidic solution yields sodium 2,2',5,5'-tetrachloroazobenzene-4,4'-disulphonate.—H. W.

Nitronaphthalenesulphonic acids; Reduction of —. H. E. Fierz and P. Weissenbach. Helv. Chim. Acta, 1920, 3, 305–311.

REDUCTION of 1,6- and 1,7-nitronaphthalenesulphonic acids, either electrolytically or by means of iron and ferrous sulphate, yields the corresponding hydroxylamines. Complete reduction is only effected in weakly acid solution, e.g. by the use of ferrous sulphate. The 1,5- and 1,8-acids, however, and also 1,3,6,8-nitronaphthalenetrisulphonic acid, may be reduced electrolytically to the amino-acids in 10% sulphuric acid solution, a current density of 200–600 amp. per sq. dm. being employed between lead electrodes. (Cf. J.C.S., i., 429.)

—J. K.

a-Naphthylaminesulphonic acids; Action of sodium hydroxide on —. H. E. Fierz. Helv. Chim. Acta, 1920, 3, 318–329.

The proportions of the various products obtained by treatment of a-naphthylaminesulphonic acids with varying strengths of sodium hydroxide solution at different temperatures were determined. The chief products obtained were as follow, the figures in brackets indicating the maximum proportion obtained, with the corresponding temperature and strength of alkali employed, the time of action in all cases being 3 hrs.:—From a-naphthylamine-4-sulphonic acid, a-naphthol (50%), a-naphthol-4-sulphonic acid (51%, 280°, 10%); from a-naphthylamine-5-sulphonic acid, 1,5-dihydroxynaphthalene (60.2%, 290°, 30 or 50%), 5-amino-a-naphthol (52.4%, 250°, 30%); from a-naphthylamine-6-sulphonic acid, a-naphthol-5(?)sulphonic acid (49%, 260°, 20%); from a-naphthylamine-7-sulphonic

acid, α -naphthol-7-sulphonic acid (40% (?), 260°, 50%), 8-amino- β -naphthol (37.7%, 280°, 50%); from α -naphthylamine-8-sulphonic acid, 1,8-dihydroxynaphthalene (46.6% [70% by very rapid heating], 300°, 10%), α -naphthylamine (32.5%). Some naphthalene is always produced when naphthalene- α - or β -sulphonic acid is fused with sodium hydroxide solution. (Cf. Willson and Meyer, J., 1915, 652.)—J. K.

Production of low-temperature tar in steam power plants. Wilkens. See IIa.

Chlorobenzenes. Bourion. See XX.

Indicators and their applications. Lubs. See XXIII.

PATENTS.

Still [; Tar —]. P. C. Reilly. U.S.P. 1,310,874, 22.7.19. Appl., 28.3.14.

A METAL still is mounted within and spaced from a furnace casing, so that products of combustion from the furnace have free access to all parts of the outer surface of the still.

Rectification of benzol. C. Otto und Co., G.m.b.H., Dahlhausen. G.P. 302,584, 22.6.17.

IN order to cool thoroughly the return system of the column and to separate the high-boiling from the low-boiling products, the dephlegmator is provided with a water-jet sucking and mixing device incorporated in the cold water conduit. Part of the warm water flowing out of the dephlegmator is injected into and mixed with the inflowing cold water. —A. G.

Tar-products and resinous substances; Separation of the low-boiling constituents of —. Chem. Fabr. Worms, A.-G. G.P. 310,171, 24.1.17.

THE material is heated with metal halides containing water of crystallisation, or with kieselguhr, with or without pressure. For example, creosote heated to 200° C. without a catalyst yields 43% of light oil, while with the addition of 25% of calcium, aluminium, or magnesium chloride, or of kieselguhr, the yield rises to 32%, 30%, 31%, and 41% respectively. The yield can be still further improved by re-distillation under pressure of an indifferent gas, or by ordinary distillation after heating for some hours under a reflux condenser or in an autoclave under pressure. —B. V. S.

Separating light oils from washing oil and regenerating the latter; Apparatus for —. H. Koppers, Essen, Assignor to The Koppers Co. U.S.P. 1,323,396, 2.12.19. Appl., 8.7.13.

AN apparatus for carrying out the process described in E.P. 15,965 of 1913 (J., 1914, 411) consists of a distilling column, with an attached condenser, and a distilling chamber. Means are provided for conveying products from a point of the distilling column to the distilling chamber, and for conveying vapour from the chamber to a vapour pipe connected with the outlet of the condenser.—L. A. C.

Fractional distillation of mixtures of liquids, e.g., wash oil saturated with benzene; Apparatus for the —. C. Weirauch, Birkenhain. G.P. 316,653, 30.6.16.

THE saturated wash-oil or the like is distilled in a series of vessels heated by a mixture of air and smoke-free furnace gases conveyed to each separate vessel by parallel flues. Each vessel is heated by gases at 400°–500° C., and is provided with a fractionating column which returns the condensed liquid to the following vessel.—L. A. C.

Benzene; Catalytic oxidation of —. The Barrett Co., New York, Assignees of J. M. Weiss and C. R. Downs. E.P. 122,167, 12.11.18. (Appl., 18,550/18.) Conv., 5.1.18.

BENZENE vapour mixed with air or oxygen is passed over a catalyst of fine pumice impregnated with vanadium oxide heated to 300°–700° C. The proportions of benzene and oxygen may be varied within wide limits, but as the reaction is probably

$C_6H_6 + 7O = (CH)_2(CO_2H)_2 + HCHO + CO_2$, the optimum proportions will be those indicated by this equation.—G. F. M.

Diphenyl; Production of —. J. M. Weiss and C. R. Downs, Assignors to The Barrett Co. U.S.P. 1,322,983, 25.11.19. Appl., 5.1.18.

A MIXTURE of benzene vapour and steam is heated sufficiently to produce diphenyl.—L. A. C.

Dinitrophenol; Manufacture of —. O. Silberrad. E.P. 140,955, 10.7.19. (Appl. 17,280/19.)

PHENOL is converted into *p*-nitrosophenol, which is then nitrated by suspending 100 pts. in a solution of 200 pts. of sodium nitrate in 350 pts. of water, and running the mixture into 480 pts. of 50% sulphuric acid at 70°–80° C.; the reaction mixture is finally heated to 95° C. for 6 hours, and the resulting 1,2,4-dinitrophenol filtered off. The yield amounts to 130% of the phenol originally employed. Nitric acid (sp. gr. 1.2) may be substituted for the sodium nitrate and sulphuric acid. —G. F. M.

Phthalic anhydride. C. E. Andrews, Assignor to The Selden Co., Pittsburgh. U.S.P. 1,336,182, 6.4.20. Appl., 14.10.19.

PURE phthalic anhydride, m.p. above 130° C. (J., 1920, 57A), is claimed as a manufactured product. —L. A. C.

Naphthalene; Purification of —. R. Kutschenreuter, Berlin. G.P. 317,634, 14.8.18.

NAPHTHALENE is recrystallised from hydro-naphthalenes, and is thereby obtained extremely pure. Naphthalene is miscible in all proportions with tetrahydronaphthalene at 30° C.; at 10° C. the solubility is 1 in 10, and below 6° C. it is almost insoluble.—G. F. M.

Tetrahydro- β -thionaphthol; Preparation of —. G. Schroeter and W. Schrauth, Berlin. G.P. 299,604, 18.5.16.

TETRAHYDRONAPHTHALENE- β -SULPHONIC chloride, m.p. 55°–56° C., b.p. 194°–195° C. at 14 mm., prepared from tetrahydronaphthalene and chlorosulphonic acid, is converted by reduction with zinc dust and sulphuric or hydrochloric acid into tetrahydro- β -thionaphthol, b.p. 153°–155° C. at 26 mm. This substance has only a slight mercaptan odour, gives a crystalline mercury salt, and with dimethyl sulphate a colourless methyl ether having an aromatic odour. Its solution in caustic soda gives with chloroacetic acid tetrahydronaphtholthioglycollic acid, $C_{10}H_{11}S \cdot CH_2 \cdot CO_2H$, m.p. 133°–135° C. By the action of air on an ammoniacal alcoholic solution of the thionaphthol the disulphide $(C_{10}H_{11})_2S_2$, m.p. 87°–89° C., is formed.—G. F. M.

p-Toluenesulphonic acids halogenated in the side chain; Preparation of salts of —. Ges. für Chem. Ind. in Basel. G.P. 312,959, 24.6.17. Conv., 19.1.16.

p-TOLUENESULPHONIC acid or its dry sodium salt is treated at a high temperature with chlorine or bromine, or with substances yielding chlorine or bromine, either with or without a halogen carrier, and, according to the amount of halogen employed and the reaction temperature, the *p*-sulphonic acids of benzyl chloride or bromide or benzal chloride or bromide of their salts are obtained.—G. F. M.

Lubricants. G.P. 309,178 and 316,720. See IIa.

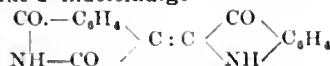
IV.—COLOURING MATTERS AND DYES.

Nitrous acid; Stabilisation of — in connexion with reactions carried out with this compound. Applications to diazotisation. E. Briner and R. Jonas. *Helv. Chim. Acta*, 1920, 3, 366—369.

Owing to the reversibility of the decomposition of nitrous acid into nitric acid, nitric oxide, and water, solutions of nitrous acid are stable in an atmosphere of nitric oxide under pressure. Di-nitraniline, tribromoaniline, and *p*-sulphobenzene-azo- α -naphthylamine, which are ordinarily difficult to diazotise, were successfully diazotised by condensing nitrogen trioxide (*cf.* Morgan and others; *Chem. Soc. Trans.*, 1917, 111, 187; 1918, 113, 588; *J.*, 1917, 449) in a cooled tube containing the amino compound and hydrochloric acid, and allowing the temperature to rise after sealing the tube. Picramide was not diazotised by this treatment. The value of the use of compressed air, proposed in the case of the last of the above three compounds (G.P. 143,450) is probably explicable in the same way.—J. K.

1.3-Dihydroxyisoquinoline [*o*-homophthalimide]; **Dyes derived from** —. W. Herzog. *Ber.*, 1920, 53, 564—567.

HOMOPHTHALIMIDE readily condenses with α -isatin-anilide in acetic anhydride solution to yield 1-oxy-4-isoquinoline-2'-indoleindigo



m.p. about 270° C., which is readily decomposed by dilute solutions of alkali hydroxides, but which can be converted into its leuco-compound by zinc dust and acetic acid; it gives dark violet shades on wool. Similarly, β -isatinanilide yields 1-oxy-4-isoquinoline-3'-indoleindigo, m.p. about 300° C., which is slowly decomposed by cold solutions of alkali hydroxide; it can be reduced by alkaline hydrosulphite or by zinc and acetic acid, but the vat is devoid of tinctorial properties. 1-Oxy-4-isoquinoline-2'-thionaphtheneindigo, from *o*-homophthalimide and thionaphthenequinoneanilide, has m.p. about 250° C. (decomp.). The leuco-compound has little affinity for fibres, giving fugitive cherry-red shades on wool and pale pink on cotton.—H. W.

Carminic acid. O. Dimroth and H. Kämmerer. *Ber.*, 1920, 53, 471—480.

THE close resemblance of carminic acid in absorption spectrum and tinctorial properties to kermes dye indicates that it, like the latter, is a hydroxyanthrapurpurin derivative (*J.*, 1916, 830). This is shown to be the case, since carminic acid is reduced by zinc dust and acetic acid to a leuco-compound, which is converted by air into desoxycarminic acid, and the latter can be oxidised to a diquinone, from which the parent substance is regenerated by acetic anhydride and sulphuric acid. The side-chain is considered to be $\text{C}_6\text{H}_4\text{O}_2$ and to contain four hydroxy-groups, and most probably has a sugar-like structure (although carminic acid is not a glucoside); it contains at least one asymmetric carbon atom, since carminic acid and its derivatives, but not kermesic or laccaic acids, are optically active. (*cf.* *J.C.S.*, i., 422.)—H. W.

Hydroxyanthraquinones; Acetylation of —, and **anthraquinones.** O. Dimroth, O. Friedemann, and H. Kämmerer. *Ber.*, 1920, 53, 481—487.

HYDROXYL groups in the β -position in hydroxyanthraquinones are more readily acetylated than similar groups in the α -position. β -Acetyl derivatives may be prepared by allowing calculated quantities of acetic anhydride and the hydroxy-compounds to react in cold pyridine solution; with an excess of anhydride, the α -hydroxy-groups are

also attacked. The acetylation of 1- and 2-hydroxyanthraquinones, of purpurin, Alizarin Bordeaux, and hydroxyanthrapurpurin has been investigated. Improved methods for the preparation of 1.4.9.10-anthradiquinone (Dimroth and Schultze, *J.*, 1916, 831) and its mono- and di-bromo-derivatives are described. (*cf.* *J.C.S.*, i., 443.)—H. W.

Anthocyanins present in young leaves of the grape vine. O. Rosenheim. *Biochem. J.*, 1920, 14, 178—188.

THE red pigment of the young leaves of *Vitis vinifera* is a free anthocyanidin, probably identical with cyanidin, the anthocyanidin of the purple grape. The pigment may occur in a colourless modification, termed a leuco-anthocyanin, in which form it appears to be combined with another substance, possibly a carbohydrate. The leuco-anthocyanin gives rise to anthocyanidin on treatment with strong acids.—J. C. D.

Rutin. Flavone pigment of *Eschscholtzia Californica*. C. E. Sando and H. H. Bartlett. *J. Biol. Chem.*, 1920, 41, 495—501.

THE presence of rutin in this flower has been established.—J. C. D.

Auramine as sensitiser. Bloch and Renwick. *See* XXI.

PATENTS.

Sulphur dye. C. Starobinetz, Jacksonville, Assignor to Barking Chemicals Co., Ltd., London. U.S.P. 1,318,022, 7.10.19. Appl., 8.8.18.

THE oily residue, containing various nitrotoluenes, obtained in the purification of trinitrotoluene, is fused with sodium sulphide and sulphur to obtain a brown sulphur dye.—A. J. H.

Arylaminoquinones; Preparation of mono- and di- —. M. Becke and W. and H. Suida, Vienna. G.P. 300,706, 12.6.15. (*cf.* U.S.P. 1,196,422; *J.*, 1916, 1256.)

MONOARYLAMINOQUINONES are obtained by the interaction in aqueous solution, or suspension, or by triturating together in the absence of any diluting medium, 1 mol. of a quinone of the benzene series with 1 mol. of a primary or secondary aromatic amine or a derivative thereof. By the further action of 1 mol. of the same, or another, aromatic amine diarylaminoquinones are obtained. Sulphonic acid derivatives of aromatic amines containing, in addition, an auxochrome or chromophore group, and aminoazo dyestuffs, are mentioned as suitable components. The reactions may be carried out on animal or vegetable fibres, and the arylaminoquinone then converted into its copper lake by treatment with copper acetate. The monoarylaminoquinones are soluble in ether and alcohol, only slightly soluble in water, and give yellow water-soluble bisulphite compounds. They dye wool in an acetic acid bath and on after-treatment with copper acetate give various shades of brown, fast to washing, alkali, and light. Their chief use is as intermediate products in the preparation of diarylaminoquinones, *e.g.*, 2.5-di-*p*-tolylamino-1.4-benzoquinone, which when dyed on wool and after-treated with copper acetate gives a very fast yellowish brown.—G. P. M.

Indigo derivatives; Preparation of —. Konsortium für Elektrochem. Ind. G.P. 310,197, 19.10.17.

ACETALDEHYDE or paraldehyde is allowed to react at a low temperature with a solution of indigo in concentrated sulphuric acid. The product is a greenish-blue vat dye for wool and cotton.

—A. J. H.

Monoazo dyestuffs; Manufacture of new —. Farbenfabr. vorm. F. Bayer und Co. E.P. 133,012, 22.11.17. (Appl. 17,225/17.) Conv., 25.7.16.
See G.P. 309,951 of 1917; J., 1919, 354A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Woods; Determination of cellulose in —. W. H. Dore. J. Ind. Eng. Chem., 1920, 12, 264—269.

ALL processes involving preliminary hydrolysis in the determination of cellulose by the chlorination method result in diminished yield of α -cellulose as well as of total cellulose and, therefore, are untrustworthy. The ratio of α -cellulose to total cellulose is practically the same whether or not preliminary hydrolysis is used, showing that the highest type of cellulose is as strongly attacked as are the lower types. During the treatments incidental to the chlorination process the hemicelluloses are hydrolysed and dissolved in the filtrates and washings, and preliminary treatment with the object of removing hemicelluloses is superfluous. The greater part of the furfural-yielding complex (probably oxycellulose) remains in the residue (total cellulose) practically unaffected by the hydrolytic treatments employed; the remainder (probably xylan) is hydrolysed and dissolved during the chlorination. The significance of the term "cellulose" as applied to wood products is discussed.—W. P. S.

Celloisobiose. Ost and Prosiogel. See XVII.

PATENTS.

Cellulose esters; Preparation of —. E. Wagner, Berlin. G.P. 299,181, 4.3.14.

PARTIALLY-ACETYLATED cellulose obtained by the action of acetic acid on cellulose is further acetylated by somewhat less than double its weight of acetic anhydride, whereby the insoluble acetyl compounds are converted into higher acetylated products, which are directly soluble in the usual solvents, e.g., acetone. By this means a saving of acetic anhydride and of the catalyst, where such is employed, is effected. An example is given of the preparation of a cellulose acetate containing about 61% of acetic acid.—G. F. M.

Washing and dehydration of cellulose and cellulose preparations; Continuous —. R. Müller. G.P. 307,078, 2.3.18.

A ROTARY suction machine is combined with a device for the counter-current washing of the material with alcohol of progressively increasing strength. A pulp containing, e.g., 10% nitrocellulose can be treated by this method.—B. V. S.

Cellulose derivatives; Increasing the softness and flexibility of products made from —. Farbenfabr. vorm. F. Bayer und Co. G.P. 307,125, 9.5.17.

β -NAPHTHOL amyl ether is used. It is liquid, insoluble in water, of high b.p., and is more stable to light than the naphthol ethers hitherto proposed as substitutes for camphor. It can be used with acetyl-, nitro-, ethyl-, propyl-, and benzyl-cellulose.

—J. H. L.

Paper; Preparation of textile fibres from raw plants and production of — therefrom. Nesselanbau G.m.b.H. G.P. 318,203, 2.2.18. (See also G.P. 307,144 and 312,381; J., 1920, 13A.)

THE raw material is treated with emulsions of mineral oils or halogen derivatives of hydrocarbons. The oils may be emulsified with soaps, sulphonated oils, etc.—J. H. L.

Paper yarn; Prevention of mould growth on —. M. Sarason. G.P. 318,307, 9.2.18.

THE yarn is impregnated with cellulose extract (Zellstoffextrakt), preferably after the latter has been fermented.—J. H. L.

Caustic alkali liquors. G.P. 316,147. See XVI.

Acidifying distillery mash. G.P. 317,998. See XVIII.

Material for nitration. G.P. 300,069. See XXII.

Cellulose derivatives. G.P. 309,260. See XXII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Aniline Black; Printing process for —. E. Jentsch. Färber-Zeit., 1920, 31, 75—76.

A GOOD black can be obtained by the following method. The padding mixture is formed of the following three solutions and is diluted to 100 kg.:— (1) 9 kg. wheat starch, 38½ kg. of water, 10½ kg. of gum tragacanth (6.5% aqueous solution), ½ kg. of Ultramarine BN are heated for one hour, cooled, and 390 g. of potassium chlorate is added. (2) 9890 g. of aniline salt, 580 g. of aniline, 9 kg. of water. (3) 4770 g. of potassium ferrocyanide, 14,930 g. of water. The fabric, prepared as usual, is printed, dried at 35°—38° C., and aged for 2—3 min. in the rapid ager, using steam as dry as possible. This is followed on the next day by chroming. The chrome bath holds 600 litres of a solution containing 1200 g. of potassium chromate and 1800 g. of sodium carbonate. For each piece of 30—38 m. is added 8 l. of a solution containing ½% potassium chromate and ¾% of sodium carbonate. After chroming at 40°—45° C. the fabric is washed, soaped, washed, and dried.—A. J. H.

Fading of dyes in daylight; Measurement of —. O. Meissner. Physik. Zeits., 1919, 20, 210—11. Chem. Zentr., 1919, 90, IV., 232.

MEASUREMENTS by Ostwald's method (J., 1919, 914A) of the effect of light on dyed fabrics showed that with dark colours during fading the proportion of white increased at the expense of the black, the purity and shade (colour tone) remaining approximately the same. In the case of light colours, the white and black both increased to approximately the same extent, and the purity was correspondingly decreased. In very light colours the black increased at the expense of the white.—A. J. H.

PATENTS.

Paper yarns and fabrics; Bleaching —. F. E. Jagenberg, Düsseldorf. G.P. 299,651, 1.9.16.

THE material is heated for 7 hrs. in a solution of a hydrosulphite, rinsed, chlorinated for 6 hrs., soured with a solution of sulphuric and hydrochloric acids, thoroughly rinsed with first warm and then hot sodium carbonate solutions and then rinsed in water. The treatment is repeated, and the product is obtained almost as white as linen or cotton, without its strength being affected. The finished yarn or fabric resembles cotton.—A. J. H.

Hanks of textile fabrics; Apparatus for treating — with liquids. W. Dürsteler, Assignor to Weidmann Dyeing Co., Ltd., Thalwil. U.S.P. 1,329,166, 27.1.20. Appl., 10.7.19.

IN a machine for the dyeing, bleaching, washing, weighting, etc. of fabrics in hank form, the hanks are carried by a frame mounted on a horizontal axis, around which it is periodically rotated through 180° in order to effect a change in the hank parts in contact with the carriers.

—A. J. H.

Fibre-treating machine. Fabric-treating machine.
H. M. Dudley, Philadelphia. U.S.P. (A) 1,335,097
and (a) 1,335,098, 30.3.20. Appl., 18.7 and
22.11.17.

(A) A DYEING machine consisting of a series of fibre chambers enclosed top and bottom by a series of perforated cones pointing inwards. The relative positions of the top and bottom can be varied. Below the bottom closure is a conical member within which are plates diverging upwards. A liquid can be forced upwards through the conical member and so through the fibre chambers. (a) A machine consisting of fabric chambers each containing a hollow perforated cylinder on which the fabric is wound. Means are provided for continuously forcing a liquid through the cylinders from the inside and so through the fabrics.—A. J. H.

Silk; Process for dyeing black and loading —
Ullnerwerk G.m.b.H. G.P. 316,751, 8.9.18.
Addition to 305,275 (J., 1918, 464 A).

"Bast soap" (boiled-off soap), artificial bast soap or a substitute therefor, is added to the loading bath in addition to water-glass.—J. H. L.

Boiling, dyeing, steaming, and washing textiles; Apparatus for — F. Gebauer, Maschinenfabr.
G.P. 315,869, 19.12.17.

A HORIZONTAL boiler is separated by partitions into a number of cylindrical compartments. Each compartment is provided with a manhole, a perforated false bottom, and valved inlet and outlet pipes at the top and bottom. Dye liquor, or the like, is circulated by means of a pump through a heater and into a main connected with each of the inlet pipes, and can thus be forced through any desired number of the compartments; the outlet pipes are also connected with a single main which returns the liquor to the pump.—L. A. C.

Printing pastes containing basic dyes; Preparation of — Farbenfabr. vorm. F. Bayer und Co.
G.P. 312,584, 28.3.16.

The printing paste is made of the colour, tannic acid, resorcinol, and a salt of a divalent metal capable of precipitating tannin. Suitable salts are acetates, formates, and lactates of zinc, cadmium, and manganese. Heavy shades can be obtained.

—A. J. H.

Hanks of yarn; Apparatus for treating — with liquids. G. S. Lord. E.P. 141,098, 5.12.18.
(Appl. 20,116/18.)

Arylaminoquinones. G.P. 300,706. See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric acid; Intensive manufacture of —
P. Pipereant. Monit. Sci., 1920, 10, 49—56.

VARIOUS systems of manufacture of sulphuric acid by the chamber process are critically examined, and a plant is described which is designed for the interaction of the sulphurous, nitrous, and aqueous vapours in a relatively small space. The constituents which have not entered into reaction are removed, passed through conduits wherein their characters (temperature, partial pressure of water vapour, etc.) are modified, and then introduced into a second reaction chamber, and so on, until reaction is complete.—W. J. W.

Ammonia synthesis; Electrically heated bomb for — R. O. E. Davis and H. Bryan. J. Ind. Eng. Chem., 1920, 12, 287—288.

A BOMB for the experimental synthesis of ammonia was made from a nickel-chrome iron gun forging

bored out to give a wall 1.5 in. thick, with an internal diameter of 4.25 in. and length of 15 in. A flange of the same material was screwed to the top of this shell, and to this was bolted a plate forming the head of the bomb, the closure being effected by means of recessed rings in the end of the cylinder wall and corresponding raised rings on the cap. The head plate carried the electrical heater, the catalyst tube, and inlet and outlet pipes. The sides of the bomb were protected by a fused quartz or glass tube. The heater consisted of two concentric alundum tubes wound in series with double strands of nichrome wire. The gases entered at the top of the bomb and passed down the outside of both tubes, then up through a steel tube (containing the catalyst), which slipped inside an alundum tube, and out at the top of the chamber. The catalyst tube could be withdrawn by loosening a nut; it was made gastight by means of a flange and copper washer.—C. A. M.

Gases for testing catalysts for ammonia synthesis; Purification of compressed — R. O. E. Davis.
J. Ind. Eng. Chem., 1920, 12, 289—290.

HYDROGEN and nitrogen prepared by decomposition of ammonia by an iron catalyst at 675° C. were freed from residual ammonia (about 0.04%) by scrubbing with water, and then passed through a steel-wool filter for catching oil and water spray, a palladium chamber to remove oxygen, a water trap, another steel-wool filter, two soda-lime and calcium chloride chambers, a phosphorus pentoxide chamber, and a sodamide chamber. The palladium chamber contained a central steel tube packed with steel wool mixed with 5% of palladised asbestos and fitted into an alundum tube wrapped with nichrome resistance wire. The gases were kept for about 12 secs. in contact with the palladium at 300°—400° C. The sodamide chamber, in which the last traces of carbon monoxide and water were removed, was heated to a little over 100° C.

—C. A. M.

Ammonia; Steam consumption in the manufacture of — from crude calcium cyanamide. J. Baumann. Chem.-Zeit., 1920, 44, 293—294.

Using an apparatus consisting of a horizontal autoclave, a Feldmann's column, and a locomotive boiler, it was found that 100 kg. of calcium cyanamide required 331 kg. of steam. The heat balance sheet showed a loss amounting to 7.7% of the heat supplied, which is probably to be attributed to the endothermic character of the reaction. The heat unaccounted for, in working up 13,200 kg. of calcium cyanamide, amounted to 2,366,800 cals. It is estimated that, using a plant such as that described in G.P. 251,934 (J., 1912, 1125), the steam consumption could be reduced to 126 kg. per 100 kg. of calcium cyanamide.—E. H. R.

Calcium and potassium sulphates; Formation of double salts of — at 100° C. E. Anderson and R. J. Nestell. J. Ind. Eng. Chem., 1920, 12, 243—246.

THE mean results of a series of determinations have shown that for the formation of potassium pentacalcium sulphate and of syngenite ($K_2SO_4 \cdot CaSO_4 \cdot H_2O$) at 100° C. (J., 1919, 320 A) the equilibrium concentrations are 1.05 mols. of potassium sulphate and 0.21 mol. of calcium sulphate, and 9.26 mols. of potassium sulphate and 0.142 mol. of calcium sulphate, respectively, per 1000 mols. of water. (Cf. J.C.S., June.)—C. A. M.

Calcium nitrate; Determination of nitrogen in — E. Platon. Chim. et Ind., 1920, 3, 310—312.

CALCIUM nitrate as manufactured in the Pyrenees by neutralising nitric acid, obtained from the air, with limestone, has the following average composition:— $Ca(NO_3)_2$, 76.15; $Ca(NO_3)_2$, 0.05; CaO , 0.15;

MgO, 0.30; CaCO₃, 0.55; MgCO₃, 0.35; Fe₂O₃, 0.10; Al₂O₃, 0.40; SiO₂, 0.50; and H₂O, 21.45%. Owing to its hygroscopic nature, a large sample should be taken, and 10–25 g. of this dissolved, and aliquot parts of the solution used. The best method of determining the nitrogen is by reducing the nitrate by means of Devarda's alloy in alkaline solution and distilling the ammonia.—C. A. M.

Hydrosulphite (Blankit, Redo); Evaluation of —. G. Bruhns. *Z. angew. Chem.*, 1920, 33, 92.

THE usual method of estimating hydrosulphite by titrating a solution of the salt with potassium ferricyanide, using ferrous sulphate as indicator, is subject to error and difficult to carry out on account of the rapidity with which the hydrosulphite solution is oxidised by atmospheric oxygen. The analysis can be carried out quickly and accurately in the following manner. In a porcelain dish are placed 20 c.c. of a standard solution of potassium ferricyanide, with a little ferrous sulphate sufficient to form a blue colloidal solution of Turnbull's blue, but not a precipitate. A slight excess of the hydrosulphite is weighed out in a scoop, and the salt is carefully dusted into the ferricyanide solution, with gentle stirring, until the end point is indicated by the change of colour from blue to bright reddish-yellow. The scoop is then weighed again to determine the weight of hydrosulphite used. The accuracy of the method depends on the skill in dusting the powdered salt into the ferricyanide solution. There is a small constant error due to the oxygen dissolved in the solution, but this is probably under 0.5%.

—E. H. R.

Potassium permanganate; Reaction between hydrochloric acid and —. F. P. Venable and D. H. Jackson. *J. Amer. Chem. Soc.*, 1920, 42, 237–239.

THE reaction between hydrochloric acid and potassium permanganate takes place in the two stages: $2\text{KMnO}_4 + 8\text{HCl} = 2\text{KCl} + 2\text{MnO}_2 + 4\text{H}_2\text{O} + 3\text{Cl}_2$; $\text{MnO}_2 + 4\text{HCl} = \text{MnCl}_2 + 2\text{H}_2\text{O} + \text{Cl}_2$. By regulating the amount of acid the reaction may be stopped at the first stage. Hydrobromic acid behaves similarly, but it will react at a concentration 0.00154N, whereas hydrochloric acid does not react if the concentration is below 0.002N.—J. F. S.

Spent oxide; Standard method of sampling and testing —. *Gas World*, 1920, 72, 320.

THE following methods have been drawn up by buyers of spent oxide, and have been approved by the National Gas Council. As the oxide is liable to change on exposure, samples sent for quotation purposes must be supplemented by samples taken during loading. On delivery, every cart or wagon must be sampled, individual samples being at once mixed, ground, and stored in air-tight receptacles, and then mixed and broken down immediately delivery is completed. The well-mixed oxide is poured several times on to a centre so as to cause even distribution of lumps and fines in the form of a cone. The cone is flattened and quartered, and two opposite quarters mixed, coned, and quartered until a convenient quantity (at least $\frac{1}{2}$ lb.) is obtained, which is then crushed until it passes entirely through a $\frac{1}{8}$ in. mesh sieve and packed in an airtight receptacle, a duplicate sample being kept for reference. For analysis the whole sample is again intimately mixed and reduced to 100 g., and then ground so that it will pass a 20-mesh sieve. Moisture is estimated by drying 5 g. for 3 hours at 100° C. in a water-oven. Sulphur and tar are determined by extracting the dried sample for 2 hours in a Soxhlet apparatus with carbon bisulphide. Air is then blown into the flask, and the extract dried for 2 hours at 100° C. in a water-oven. The flask is then heated on a sand-bath until the

sulphur just fuses, after which it is again blown out with air and weighed. Sulphur is estimated by treating the tar and sulphur residue with 15 c.c. of 95% sulphuric acid for 2 hours at 100° C. After cooling, the mixture is diluted, filtered, washed free from acid, and dried. The residue is then extracted with carbon bisulphide in a Soxhlet apparatus, fused as before, and weighed.—W. J. W.

Arsenate of calcium. I. Equilibrium in the system arsenic pentoxide, calcium oxide, and water at 35° C. (acid section). C. M. Smith. *J. Amer. Chem. Soc.*, 1920, 42, 259–265.

THE two compounds dicalcium orthoarsenate monohydrate, $\text{CaHAsO}_4 \cdot \text{H}_2\text{O}$, and monocalcium orthoarsenate, $\text{CaH}_2(\text{AsO}_4)_2$, respectively exist below and above an acid concentration corresponding to 27.5% As_2O_5 in the above-named three-component system.—J. F. S.

Arsenious acid; Reversible oxidation of —. C. Matignon and J. A. Lecanu. *Comptes rend.*, 1920 170, 941–943.

ARSENIOUS oxide, when heated with oxygen at 400°–480° C. under a pressure of 127–180 atm., is oxidised to an extent which increases with rise in temperature. A concentrated solution of arsenious acid in sodium hydroxide solution, when heated at 80° C. in oxygen under a pressure of 50 atm. for 5 hrs., is oxidised to the extent of 10.9%.—W. G.

Arsenic trichloride; Preparation of — from white arsenic and phosgene. L. H. Milligan, W. A. Baude, and H. G. Boyd. *J. Ind. Eng. Chem.*, 1920, 12, 221–223.

ARSENIC trichloride is formed almost quantitatively when phosgene is led over a mixture of arsenious oxide, 80, and carbon, 20%, heated at 200°–260° C. The reaction may be employed for the utilisation of the impure "tail gas" from phosgene manufacture, and the method of chlorination with phosgene in presence of carbon as catalyst will probably be of general application to inorganic compounds.

—W. P. S.

Mercuric oxide; Solubility of — in sodium hydroxide solutions. G. Fuseya. *J. Amer. Chem. Soc.*, 1920, 42, 368–371.

THE solubility at 25° C. increases with the concentration of sodium hydroxide, from 2.6 to 30.9 millimols. HgO per litre as the concentration of NaOH increases from 0.0096 to 2.09 mols. per litre. Hydrated mercuric oxide appears to function as a very weak acid.—E. H. R.

Rare earths. X. Purification and atomic weight of samarium. A. W. Owens, C. W. Balke, and H. C. Kremers. *J. Amer. Chem. Soc.*, 1920, 42, 515–522.

RARE earth residue from monazite sand, from which most of the cerium had been extracted, was converted into nitrate and treated with bromine to remove the remainder of the cerium. Lanthanum, praseodymium, and neodymium were removed by fractionation of the magnesium double nitrates, and finally europium and gadolinium by further fractionation with bismuth nitrate. The samarium was finally obtained as the pure anhydrous chloride. A mean of 18 determinations gave 150.43 as the atomic weight of samarium, silver being 107.88. (*Cf. J.C.S.*, ii., 316.)—E. H. R.

Carbonyl chloride; Detection and estimation of free chlorine in —. M. Delépine. *Bull. Soc. Chim.*, 1920, 27, 283–286.

A KNOWN weight of the carbonyl chloride is vaporised, the vapours are led into a 0.1% aqueous solution of potassium iodide, and the iodine liberated is titrated with standard thiosulphate solution. If the concentration of the iodide solu-

tion exceeds 0.1% there is likelihood of interaction between the oxychloride and the iodide with consequent liberation of iodine.—W. G.

Carbonyl chloride; Decomposition of — by water: estimation of escaped gases. M. Delépine, R. Douris, and L. Ville. *Bull. Soc. Chim.*, 1920, 27, 286–288.

CARBONYL chloride is only slowly decomposed by atmospheric moisture, but is rapidly decomposed by water, and less rapidly in presence of hydrochloric acid. To test the efficiency of the absorption towers used to remove the carbenyl chloride from the waste gases in its manufacture, a known volume of the air issuing from the absorption tower is bubbled through 10 c.c. of an aqueous solution containing 1 c.c. of $N/1$ sodium hydroxide and 5 c.c. of 95% alcohol. The liquid is then evaporated in a water bath to 2 c.c., 2 drops of acetic acid are added, and the evaporation is continued to dryness. The residue is treated with 2 c.c. of water, again evaporated to dryness, treated with 2 c.c. of water and the chloride present titrated with $N/40$ silver nitrate. By this means it is possible to estimate the chlorine to within 0.00005 g.—W. G.

Carbonyl chloride; Action of — on industrial iron containers. M. Delépine and L. Ville. *Bull. Soc. Chim.*, 1920, 27, 288–290.

CARBONYL chloride containing a little chlorine as impurity slowly attacks iron, forming ferric chloride. In the absence of chlorine the oxychloride will attack rust, and the ferric chloride present in commercial carbonyl chloride probably comes from this source.—W. G.

Carbonyl chloride in titanium chloride; Estimation of —. M. Delépine and J. Lafore. *Bull. Soc. Chim.*, 1920, 27, 290–292.

A MEASURED amount of the titanium chloride is decomposed by water, and the carbon dioxide resulting from the decomposition of any carbonyl chloride present is aspirated off and collected in barium hydroxide solution and then estimated volumetrically. A special arrangement of apparatus is described designed to prevent the choking of the mouth of the inlet tube by titanium oxide or oxychloride.—W. G.

Carbonyl chloride; Estimation of hydrogen chloride in —. M. Delépine, M. Monnot, H. Duval, and J. Lafore. *Bull. Soc. Chim.*, 1920, 27, 292–295.

FIVE grms. of dry finely-powdered mercury cyanide is placed in a dry flask fitted with a ground-in stopper carrying two tubes. The carbonyl chloride is introduced into the flask in a sealed bulb. The flask is evacuated and the bulb is broken. After 12 hrs. the hydrogen cyanide formed by the action of any hydrogen chloride present in the carbonyl chloride on the mercury cyanide is aspirated off and collected in 50 c.c. of $2N$ sodium hydroxide, and estimated by Denigès' method by titration with $N/20$ silver nitrate after addition of 5 c.c. of ammonia and 1 c.c. of 10% iodide solution.—W. G.

Electrical conductivity of gases during chemical reactions; Constancy of —. M. Trautz and F. A. Henglein. *Z. anorg. Chem.*, 1920, 110, 237–289.

EXPERIMENTS on the conductivity of gases during the reactions $2NO + Cl_2 \rightleftharpoons 2NOCl$; $2NO + Br_2 \rightleftharpoons 2NOBr$; $2HBr + Cl_2 \rightleftharpoons 2HCl + Br_2$, and $Cl_2 + H_2 \rightleftharpoons 2HCl$, failed to reveal evidence of the formation of free electrons.—E. H. R.

Hydrogen; Generation of — by the reaction between ferrosilicon and sodium hydroxide solution. E. R. Weaver. *J. Ind. Eng. Chem.*, 1920, 12, 232–240.

The process yields the most economical results when

about equal weights of sodium hydroxide (as 30% solution) and ferrosilicon (88% Si) are used. For a charge of 363 kg. of ferrosilicon, one-half of the sodium hydroxide solution and 28 kg. of ferrosilicon are placed in the generator (a cylindrical tank, 6 ft. 6 in. high and 6 ft. diam.), and when the temperature reaches 90° C., ferrosilicon is added at the rate of 5.6 kg. per min.; sodium hydroxide is at the same time added continuously at such a rate that the temperature remains constant. When all the solution has been added, the temperature is kept at 90° C. by the introduction of cold water until the solution contains about 20% of sodium hydroxide. The water supply is then stopped, and the reaction allowed to proceed.—W. P. S.

Iron and steam; The reversible reaction between —. E. Schreiner and F. B. Grimmes. *Z. anorg. Chem.*, 1920, 110, 311–331.

EXPERIMENTS on the equilibrium of the reaction $Fe + H_2O \rightleftharpoons FeO + H_2$ were made between 595° and 920° C., using an apparatus similar to that employed by Deville (*Comptes rend.*, 1870, 70, 1105, 1205). The values of the equilibrium constant $K = p_{H_2O}/p_{H_2}$ increase with increasing temperature. The values at lower temperatures agree fairly well with those calculated from thermodynamic principles, but at higher temperatures there is considerable discrepancy.—E. H. R.

Catalysts. Rupert. *See I.*

Stabilisation of nitrous acid. Briner and Jonas. *See IV.*

Tungsten oxides and water. Chaudron. *See X.*

Charcoal and chlorine. Bohart and Adams. *See XIX.*

Chlorine. Lamb and others. *See XXIII.*

PATENTS.

Contact process [for manufacture of sulphur trioxide]. H. E. Potts, Liverpool, Assignor to Simon-Carves, Ltd., Manchester. U.S.P., 1,335,257, 30.3.20. Appl., 17.6.18.

A STREAM of burner gas is treated with a solvent liquid, and the solution is subjected to the action of a current of air to produce a sulphur dioxide-air mixture (containing about the same proportion of sulphur dioxide as the burner gas), which is then dried and brought into contact with a catalyst to produce sulphur trioxide.—W. E. F. P.

Sulphuric acid; Catalytic manufacture of — from sulphur dioxide. G. Vortmann, Vienna. G.P. 316,858, 28.8.18. Conv., 7.9.16.

TIN oxide or tinstone, alone or mixed with inactive materials such as clay, pumice, asbestos, etc., constitutes an efficient catalyst.—W. J. W.

Sulphuric acid; Quantitative production of — from sulphates and sulphides. P. Jannasch and E. M. Alberts, Heidelberg. G.P. 318,221, 5.3.18.

A NON-REACTING material, such as quartz, is mixed with sulphates to prevent caking, and the mixture is subjected at a red heat to the action of a current of gaseous carbon tetrachloride, carbon dioxide being employed to drive the gas through the reaction chamber. In the case of sulphides, potassium or sodium chloride may be added. The sulphur is obtained in the form of sulphur chlorides, which are decomposed by water to yield sulphuric acid and hydrogen chloride. Hexachloroethane is obtained as a by-product. Suitable raw materials are gypsum, heavy spar, celestine, kainite, and pyrites, copper pyrites, and zinc blende.—W. J. W.

Sludge acid [from mineral oil refining]; Process of producing hydrochloric acid utilising —. H. M. Lasher, Assignor to Kansas City Refining Co. U.S.P. 1,337,141, 13.4.20. Appl., 12.2.19.

A SOLUTION of hydrochloric acid is produced by a counter current of water, the gas (evolved from the sludge acid) and water being brought into contact by a series of opposite periodic waves.—A. E. D.

Hydrochloric acid; Process for producing — from [acid] sludge [from treatment of mineral oils]. H. M. Lasher, Assignor to Kansas City Refining Co. U.S.P. 1,309,206, 8.7.19. Appl., 9.5.16.

THE acid sludge is mixed with sodium chloride and diluted with water until the asphaltic and tarry matters separate in the form of a scum on the surface of the liquid. The mixture is heated to a temperature not exceeding 284° C., and the hydrochloric acid vapours evolved are purified by passage through a mixture of sodium chloride and charcoal and then absorbed in water.

Hydrochloric acid; Apparatus for producing —. H. M. Lasher, Assignor to The Kansas City Refining Co. U.S.P. 1,335,743, 6.4.20. Appl., 13.7.17.

IN apparatus for producing hydrochloric acid, the gas is conducted from a still to a vertical scrubber, and thence into a horizontal absorber, provided with baffle plates, from which the solution is passed to a receiving chamber.—W. E. F. P.

Nitric acid; Process of making —. E. Thomson, Assignor to General Electric Co., New York. U.S.P. 1,337,106, 13.4.20. Appl., 6.12.18.

AS a step in the manufacture of nitric acid, nitric oxide is dissolved in nitric acid at a temperature of -40° C.—W. J. W.

Nitric acid; Method of concentrating —. Farbenfabr. vorm. F. Bayer und Co. G.P. 310,081, 7.9.18.

NITRIC acid is brought into contact with sulphuric acid in a reaction tower into which a counter-current of air is introduced to promote denitration. The sulphuric acid emanates from a concentrating plant immediately preceding the tower, and is at a sufficiently high temperature to bring about the reaction in the tower. With nitric acid of 40–50% strength, and sulphuric acid of such a concentration as to give an "outflow" acid of 70%, it is possible to produce nitric acid of 99.5% HNO₃.—W. J. W.

Nitrogen oxides; Manufacture of — from atmospheric air. E. Herman, Budapest. G.P. 305,124, 25.1.17.

NITROGEN oxides are obtained by burning carbon compounds with air by means of flameless surface combustion in a mass of aluminium nitride serving as a contact agent. The nitride may be mixed with iron oxide or nitride, copper, cobalt, nickel, manganese, chromium, calcium, barium, cerium, thorium, or titanium. The catalytic efficiency is proportional to the aluminium content of the nitride. From the combustion of 1 cb. m. of methane (8800 cal., 15°, 760 mm.) under a pressure of 10 atm., using superheated compressed air, without addition of oxygen, a yield of 380–490 g. nitric acid (100%) is obtained.—W. J. W.

Nitric oxide four-cycle gas engines; Increasing the yield from —. H. Nöh, Saarbrücken. G.P. 306,451, 29.8.16.

By washing out the exhaust gases by means of atmospheric air, the yield is increased. In a test with 70% excess of air, more than 125 g. nitric acid was obtained per cb. m. of gas.—W. J. W.

Nitric oxide; Production of — in two-cycle gas engines. H. Woll, Gersweiler - Ottenhausen. G.P. 316,677, 19.3.19.

IN order to obtain a whirling motion in the cylinder a jet of gas is introduced at the beginning of compression, followed by a jet of air as compression proceeds. The cylinder is provided with slots at its lower end for admission of air, and exhaust, and with gas and air valves at its upper end, in the compression chamber. By a uniform construction of the cylinder casing, exceptionally high temperatures are made utilisable, and the smooth surface of the walls of the compression chamber facilitates effective whirling with thorough mixing and rapid combustion.—W. J. W.

Nitrogen fixation; Process for —. K. Prinz zu Löwenstein, Neckargemünd, and F. Hauff, Stuttgart. G.P. 318,286, 24.7.15.

BARIUM carbonate is mixed with carbon, obtained free from slag by heating methane or other hydrocarbon, and the nitrogen, formed by combustion in air of the hydrogen of the hydrocarbon, is caused to combine with the carbon-barium carbonate mixture, with formation of cyanide and cyanamide.—W. J. W.

Cyanogen compounds, ammonia and the like; Production of —. G. Calvert, London. E.P. 140,484. (Appls. 17,167, 21.10.18, and 19,135, 21.11.18.)

A MIXTURE of alkali carbonate, carbon, and a catalyst (iron powder) is heated to 900°–1100° C. in the presence of nitrogen, and fresh surfaces of the heated mass are exposed to the nitrogen continuously by mechanical means arranged within the reaction chamber. The mass is removed from the chamber intermittently or continuously for the extraction of alkali cyanide, further quantities of alkali carbonate and carbon being supplied to the reaction chamber as required. Sufficient carbon is maintained in the reaction mixture to prevent sintering and welding of the catalyst, and sufficient alkali carbonate to ensure the suspension of the carbon and catalyst in the molten alkali. The alkali carbonate may be replaced by metallic sodium introduced in the molten or vaporous condition, and the carbon by carbon monoxide.—W. E. F. P.

Alkali cyanide; Method of separating — from alkali carbonate. F. J. Metzger, Assignor to Air Reduction Co., New York. U.S.P. 1,337,019, 13.4.20. Appl., 25.7.17.

Products containing alkali cyanides are extracted with a mixture of water and an organic solvent which retards hydrolytic decomposition of the cyanide, the temperature of the mass being kept below the point at which decomposition of the cyanide takes place.—W. J. W.

Nitrogen compounds from carbides; Conversion of — into ammonium compounds. R. Mewes, Berlin. G.P. 305,082, 24.3.15.

NITROGEN compounds from carbides are treated with a solution of one or more alkali salts, such as sodium or potassium chloride, or a magnesium salt, with or without heat, and the product is then heated to the sublimation temperature of the ammonium compound. The reacting substances may be subjected to the action of an electric current or, alternatively, heated to a temperature above 100° C. The process offers a convenient means of obtaining ammonium chloride and, simultaneously, sodium or potassium carbonate. At higher temperatures ammonia is produced and calcium chloride is formed.—W. J. W.

Ammonia and dicyanodiamide; Method of obtaining — from crude calcium cyanamide. Stickstoffwerke, G.m.b.H. G.P. 318,136, 18.10.18.

THE liquor obtained by treating crude calcium

cyanamido with water or mother-liquors is freed from mud, after which dicyanodiamide is recovered from it by crystallisation. From the liquor remaining after crystallisation, as well as the residual mud, ammonia is obtained. A nearly theoretical yield of nitrogen as ammonia results.—W. J. W.

Ammonia; Process of recovery of —. W. J. Chrystal, Glasgow. E.P. 140,505. (Appls. 19,940, 3.12.18, and 13,816, 31.5.19.)

RECOVERY of ammonia (from producer gas etc.), by direct absorption in sulphuric acid, is effected in two stages, the sulphate liquor being allowed to become completely neutral in the first stage, and kept at constant acidity in the second. As neutrality is attained in the first stage, the liquor is replenished by solution from the second.

—W. E. F. P.

Sulphate of ammonia; Production of —from distillation and like gases containing ammonia. E. C. R. Marks. From Soc. Franco-Belge de Fours à Coke, Brussels. E.P. 141,093, 28.11.18. (Appl. 19,677/18.)

DISTILLATION gases are cooled to 20°–25° C. to remove tar and water vapour, and are then mixed with hot ammoniacal gases obtained free from fog by passing the ammonia from the distillation of ammonia liquor through a rectifier, and the mixture is led into a closed saturator containing a solution of ammonium sulphate and sulphuric acid (33°–34° B., sp. gr. 1.296–1.308; 7% free acid), at 25°–40° C. The ammonium sulphate crystals are removed by a compressed air injector. Weak, impure sulphuric acid (50° B., sp. gr. 1.53) as well as pure acid (60° B., sp. gr. 1.71) may be used.

—W. J. W.

Ammonium sulphate; Process of raising — from saturators by means of compressed air. F. J. Collin, Dortmund. G.P. 316,596, 3.4.19.

BEFORE introducing the air it is heated, by which means incrustations are avoided.—W. J. W.

Sulphur dioxide; Process for the removal and recovery of — from gases which contain the same. Norddeutsche Hütte A.-G., and J. Behrens, Bremen. E.P. 134,555, 15.2.16. (Appl. 2280/16.)

Gas, free from ammonia and from which hydrogen sulphide has been removed by treatment with excess of sulphur dioxide, is washed with a cold, previously boiled solution of alkali sulphite, and sulphur dioxide is recovered by boiling the solution.

—W. E. F. P.

Electrolysers [for manufacture of alkali and chlorine]. G. G. Hepburn, Manchester. E.P. 141,305, 22.2.19. (Appl. 1475/20.)

An electrolyser, of the filter-press type, having mono- or bi-polar electrodes, for the manufacture of alkali and chlorine, is immersed in a non-conducting material (paraffin or carnauba wax, pitch, or naphthalene) which is not melted at the ordinary working temperature of the electrolyser, thus preventing the escape of gas or liquid. The material is melted for the insertion or removal of the electrolyser, or glycerol may be used as the immersing substance.—B. N.

Electrolytic [alkali] cells; Method of removing certain impurities from —. H. K. Moore, Assignor to Brown Co. U.S.P. 1,309,214, 8.7.19. Appl., 25.3.19.

GELATINOUS deposits of calcium and magnesium compounds formed on the porous diaphragms of electrolytic alkali cells are removed by treatment

with an acid solution, e.g., lactic acid, which reacts with the deposit to form soluble calcium and magnesium salts.

Electrolytic decomposition of chlorides. B. Cataldi, Turin. U.S.P. 1,336,281, 6.4.20. Appl., 12.5.19.

A solution of an alkali chloride is electrolysed between an insoluble anode and a mercury cathode separated from it mechanically, thus forming an alkali-metal amalgam and chlorine. Water is allowed to flow over the amalgam to form caustic soda and hydrogen, and the reaction at the surface of the amalgam is accelerated by local electrolytic action during the flow, the hydrogen and chlorine being collected.—B. N.

Titanium-oxygen compounds; Process of producing refined —. P. Farup, Assignor to Titan Co. A./S., Christiania. U.S.P. 1,325,561, 23.12.19. Appl., 21.11.16.

FERROTITANIFEROUS material is treated with hydrochloric acid to produce a solution containing the iron and an insoluble residue containing the titanium. The residue is then separated and treated with another reagent to dissolve the titanium compounds.—W. E. F. P.

Alkali-hydrate [caustic potash] solution. H. W. Charlton, New York, Assignor to American Potash Corp. U.S.P. 1,331,989, 30.3.20. Appl., 30.11.17.

A solution of crude caustic potash is obtained from greensand by a process of digestion. The product is practically free from sodium and aluminium compounds, and when dry contains 95–98% KOH, the remainder consisting of chloride, sulphate, and silicate of potassium with organic matter.

—W. E. F. P.

Soda-lime composition and method of preparing the same. R. E. Wilson, Washington, and C. P. McNeil, Astoria. U.S.P. 1,335,949, 6.4.20. Appl., 2.10.18.

A relatively slow setting cement is used in the preparation of a soda-lime reagent for the absorption of gases.—W. E. F. P.

Magnesium chloride; Method of making —. H. H. Dow and E. O. Barstow, Assignors to The Dow Chemical Co., Midland. U.S.P. 1,336,425, 13.4.20. Appl., 25.6.18.

BRINE containing a mixture of magnesium chloride and calcium chloride is treated with sodium sulphite to precipitate calcium sulphite, which is then removed.—W. F. F.

Alunite; Utilisation of native —. H. F. Chappell, New York. U.S.P. 1,336,508, 13.4.20. Appl., 11.6.18.

ALUNITE is treated with dilute sulphuric acid to dissolve the aluminium and potassium compounds, after which iron is precipitated with a cyanide, and ammonia is added to precipitate aluminium hydroxide, which is calcined to form alumina, the soluble potassium and ammonium salts being subsequently recovered in a solid form.—W. J. W.

Potassium salts; Separation of —from minerals, especially carnallite. Salzwerk Heilbronn, G. Kassel, and T. Lichtenberger. G.P. 289,746, 16.5.14.

By passing superheated steam through the molten potash mineral, the relatively heavier magnesium salt is deposited as magnesium oxide, and, in the case of carnallite, pure potassium chloride may be obtained. The separation may be effected during the decomposition of the mineral. The magnesium sulphate in kainite is completely converted into alkali sulphate by reaction with the chloride of potassium or sodium.—W. J. W.

Magnesium carbonate; Preparation of light basic —. "Lipsia," Chem. Fabr. G.P. 301,723, 7.3.16.

MAGNESIUM carbonate, $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$, is precipitated from potash end-liquors, filtered, mixed with water whilst moist to the consistency of a thin paste, and then heated to boiling. The product is completely free from potassium and chlorine, and is of a soft voluminous nature, similar to *magnesia alba*.

—W. J. W.

Absorbent for carbon dioxide; Manufacture of a highly-efficient —. R. von der Heide, Charlottenburg. G.P. 303,261, 16.1.17.

AN absorbent for carbon dioxide is prepared by adding not more than 25% (e.g., from 5 to 10%) of strongly-heated quicklime to molten anhydrous alkali hydroxide, and grinding the product to a fine powder.—L. A. C.

Alkali thiosulphates; Preparation of —. A. Clemm, Mannheim. G.P. 305,194, 18.11.17.

A SOLUTION of an alkali sulphide is allowed to flow over red-hot carbon, such as charcoal, arranged so as to offer a large surface, air or oxygen and carbon dioxide being simultaneously passed over the material. The carbonate is then separated from the thiosulphate by crystallisation. In the case of potassium salts, conversion of the carbonate into bicarbonate facilitates the separation.—W. J. W.

Hydrogen sulphide and magnesium hydroxide; Preparation of —. P. Beck, Nordhausen. G.P. (A) 307,041, 25.7.17, and (B) 307,042, 5.8.17.

(A) SULPHIDES or hydrosulphides of the alkaline earths are caused to react at normal temperature with magnesium sulphate. After separation of the alkaline-earth sulphates, pure magnesium hydroxide is obtained by decomposition of the magnesium sulphide or hydrosulphide. The alkaline-earth sulphates may be used for again producing the sulphides. (B) Alkaline-earth sulphides or hydrosulphides are decomposed with magnesium chloride, the alkaline-earth chloride produced being then caused to react with magnesium sulphate, thus reproducing magnesium chloride. The alkaline-earth sulphates so formed are reduced to sulphides by means of carbon.—W. J. W.

Calcium hydride; Manufacture of —. A. Kieselwalter, Frankfurt. G.P. 311,987, 30.7.18.

LIME or other calcium compound is fused in the electric arc in presence of hydrogen, the by-products formed being removed. The calcium hydride produced is then cooled in an atmosphere of hydrogen, which it continues to absorb during cooling.

—W. J. W.

Per-salts; Preparation of — containing a high percentage of active oxygen. Aschkenasi, Berlin. G.P. 316,997, 22.8.15. Addn. to 299,300.

THE salt to be peroxidised is evaporated repeatedly under reduced pressure with fresh quantities of hydrogen peroxide. By this means the active oxygen content of perborates can be increased from 6.2% to 13.4% and then to 19.6%, that of trisodium perphosphate from 8.2% to 13.7%, and that of disodium perphosphate from 6% to 12%.—A. R. P.

Perborates and disodium perphosphate; Manufacture of —. Aschkenasi, Berlin. G.P. 318,219, 23.11.18.

PHOSPHATES or borates are evaporated under reduced pressure with a concentrated solution of hydrogen peroxide. From sodium phosphate, disodium perphosphate with 8.8% of active oxygen is obtained, while sodium metaborate yields perborates with 10.72%, 21.7%, 22.2%, and 28.4% of active oxygen respectively.—W. J. W.

Hydrogen peroxide and the like; Process for stabilising —. M. Sarason, Berlin. G.P. (A) 318,134, (B) 318,135, and (C) 318,220, 3.7.18.

SOLUTIONS of hydrogen peroxide may be preserved at temperatures up to 70° C. by addition of 0.2% of (A) strontium hydroxide, (B) dextrose, or (C) aniline. The treatment is also effective with solutions of sodium peroxide, sodium perborate, and similar substances.—W. J. W.

Zinc compounds; Preparation of water-soluble — from roasted zinc ores. H. Reisenegger, Charlottenburg. G.P. 317,702, 14.10.17.

ROASTED zinc ores are heated with the waste liquors obtained by extracting burnt pyrites with water after the iron has been converted into ferric compounds. Zinc oxide goes into solution, with separation of the iron as hydrated oxide, and from the solution zinc salts may be obtained by evaporation and crystallisation, or metallic zinc by electrolysis, or zinc hydroxide by precipitation with a base. By using ammonia, ammonium salts are derived as by-products.—W. J. W.

Sulphurous acid or sulphur; Manufacture of — from magnesium sulphate. V. Erchenbrecher, Halle. G.P. 307,752, 16.10.17.

MAGNESIUM sulphate, in the form of a fine-grained powder, is treated with hydrogen sulphide in a rotary furnace at the minimum temperature necessary for the reaction. The decomposition yields either sulphur or sulphurous acid, dependent on the initial proportions of the reacting substances. The residual magnesia is readily hydrated and converted into carbonate.—W. J. W.

Sulphur; Process for obtaining — from sulphur dioxide and calcium sulphate. Akt.-Ges. für Anilinfabr. G.P. 300,715, 4.11.16.

SULPHUR dioxide is caused to react with carbon in a blast-furnace, the requisite temperature being maintained by combustion in oxygen of part of the carbon. The gases leaving the furnace contain free sulphur and volatile sulphur compounds, the latter being caused to react with calcium sulphate with formation of carbon dioxide, water, sulphur, and calcium sulphide. The calcium sulphate may be introduced into the furnace itself. Recovery of the sulphur from the calcium sulphate is facilitated by addition of iron oxide, alumina, clay, granite, porphyry, etc.—W. J. W.

Sulphur from crude potassium salts containing kieserite; Method of obtaining —. J. Kiermayer, and Hannoversche Kaliwerke A.-G. G.P. 310,072, 10.9.16.

SULPHUR may be obtained from potash salts by treating them, in a fused condition, with coke and wood charcoal or partly carbonised coal. Ordinary coal is not suitable on account of formation of decomposition products.—W. J. W.

Aluminium nitride; Process of producing —. Armour Fertilizer Works, Assignees of M. Shoeld. E.P. 122,829, 6.1.19. (Appl. 365/19.) Conv., 28.1.18.

SEE U.S.P. 1,274,797 of 1918; J., 1918, 662 A.

Boron derivative of pyrophosphoric acid; Manufacture of a —. B. Levin. U.S.P. 1,336,974, 13.4.20. Appl., 15.5.18.

SEE E.P. 116,735 of 1917; J., 1918, 416 A.

Contact masses. G.P. 312,726. See I.

Ammonium carbonate. G.P. 318,236. See I.

Calcium cyanamide. G.P. 309,173. See XVI.

VIII.—GLASS; CERAMICS.

Glass; Calculating the "hardness" or expansion of —. L. Springer. *Sprechsaal*, 1920, 53, 193—195.

In Germany, the term "hardness" is frequently used with reference to thermal expansion of a glass. Actually there is no connexion between "hardness" or refractoriness and expansion, as the addition of borax will reduce expansion and increase the fusibility and "softness" of the glass. The thermal expansion may be calculated from the ultimate analysis of a glass, as previously shown by Schott and Winkelmann and Mayer and Havas respectively (J., 1911, 543). The effect of various substituted oxides on the expansion of a glass may be calculated in a similar manner.—A. B. S.

Lime-soda glasses; Properties of —. J. H. Davidson and W. E. S. Turner. *J. Soc. Glass Tech.*, 1919, 3, 222—227.

ELEVEN glasses corresponding to the formula, $a\text{Na}_2\text{O}, b\text{CaO}, 6\text{SiO}_2$, and two commercial glasses were made, and their working properties in a molten state and also under the lamp were examined. Soda-lime glasses containing more than 1.5 Na₂O and less than 0.5 CaO should be avoided, but those containing 1.0—1.4 Na₂O and 1.0—0.6 CaO are durable and work well. In most cases the addition of alumina (as felspar, china clay, or ground brick) is desirable.—A. B. S.

Lime-soda glasses; Durability of —. J. D. Canwood, J. R. Clarke, C. M. Muirhead, and W. E. S. Turner. *J. Soc. Glass Tech.*, 1919, 3, 228—237.

EXPOSURE of the glasses mentioned in the preceding abstract to water, caustic alkali, sodium carbonate, and hydrochloric acid has shown that those containing less than 0.5 CaO are too readily attacked. The replacement of soda by lime increases the resistance of the glass. For common use in automatic or semi-automatic machines glasses corresponding to 1.2—1.4 Na₂O, 0.6—0.8 CaO, 6SiO₂ are recommended.—A. B. S.

Soda-lime glasses; Heat expansion of —. S. English and W. E. S. Turner. *J. Soc. Glass Tech.*, 1919, 3, 238—242.

THE graph showing the relation between the coefficient of linear expansion and the percentage of lime in a series of pure soda-lime glasses prepared by the authors is a straight line, the glasses richest in soda having the highest expansion. Schott's well-known factors for expansion (J., 1892, 817; *Ann. Physik.*, 1894, 51, 735) do not agree with the authors' experimental results. The view that strained glass has a higher coefficient of expansion is confirmed.—A. B. S.

Lime-soda glasses; Influence of lime on the value of Young's modulus of elasticity for —. J. R. Clarke and W. E. S. Turner. *J. Soc. Glass Tech.*, 1919, 3, 260—266.

YOUNG'S modulus of elasticity was measured in a series of lime-soda glasses (J., 1919, 722A) by observing the bending of a cylindrical glass rod supported horizontally at both ends and weighted in the middle. Increase in the lime, at the expense of the soda, was accompanied by a very marked increase in the elasticity of the glass. New factors are given for calculating the elasticity of glass from its composition; Winkelmann and Schott's factors give values differing widely from the experimental results.—A. B. S.

Magnesia-soda glasses. J. H. Davidson, F. W. Hodkin, and W. E. S. Turner. *J. Soc. Glass Tech.*, 1919, 3, 275—277.

A SERIES of magnesia-soda glasses, ranging from

2Na₂O, 6SiO₂ to 0.9Na₂O, 1.1MgO, 6SiO₂, was examined. Their working and lamp-working properties are similar to those of lime-soda glasses, though the magnesia glasses are rather more difficult to manipulate; they have a greater viscosity, a tendency to stringiness, retain heat longer, have a lower coefficient of expansion for heat, and a lower annealing temperature.—A. B. S.

Magnesia-soda glasses; Annealing temperatures of —. S. English and W. E. S. Turner. *J. Soc. Glass Tech.*, 1919, 3, 278—281.

IF the magnesia content of magnesia-soda glasses is increased beyond 10%, each added increment causes a great increase in the annealing temperature of the glass. Below 10% the increase in the annealing temperature with the lime content in lime-soda glasses, is greater than with increase of the magnesia content in magnesia-soda glasses. In glasses containing equimolecular proportions of magnesia or lime, those containing magnesia are easier to anneal than those containing lime, so that dolomite appears to be a desirable substitute for ordinary limestone in commercial glasses.—A. B. S.

Glass for lampworkers; Proposed standard formula for —. F. W. and F. H. Branson. *J. Soc. Glass Tech.*, 1919, 3, 249—253.

THE following batch mixture is recommended: Sand, 1104; potash felspar, 400; limespar, 222; potassium nitrate, 213; soda ash (95%), 410; manganese dioxide (87%), 3 parts. The glass made from this mixture heated at 1340° C. for 14 hrs. is free from appreciable colour and from tendency to devitrification; fuses readily on to glasses of various degrees of hardness and composition; does not appreciably darken in the blowpipe flame; and possesses a suitable viscosity, or good working properties, for lamp-blown work.—A. B. S.

Glass for table-working. M. W. Travers. *J. Soc. Glass Tech.*, 1919, 3, 253—256.

SOME glasses used for scientific apparatus devitrify and become useless when heated in the blowpipe. The defect is sometimes more noticeable if the glass has been in stock for some time. The best glasses for table-working appear to contain 67—69% SiO₂, 3—1% Al₂O₃, 6—8% CaO, 7—6% K₂O, and 13—12% Na₂O, but such glasses require high furnace temperatures and are costly. Much of the glass tubing now made would be improved if the glass were plained at a higher temperature in a gas-fired furnace. Experiments should be made to ascertain how much lime and alumina—the latter preferably as felspar—can be present in a glass which will work satisfactorily at the blowpipe table.—A. B. S.

Glass for lamp-working purposes. J. D. Canwood, J. H. Davidson, F. W. Hodkin, and W. E. S. Turner. *J. Soc. Glass Tech.*, 1919, 3, 266—274.

A NUMBER of foreign glasses used for making lamp-working tubing were analysed and other glasses of the same ultimate composition were prepared. The best lamp-working properties were obtained with a glass containing SiO₂ 69.10%, As₂O₃ 0.10%, Al₂O₃ 3.10%, CaO 6.89%, MgO 0.20%, K₂O 6.38%, Na₂O 1.5% and MnO 0.10%. A high lime content is not necessarily detrimental, but both arsenic and antimony oxides cause the glass to blacken when heated in the blowpipe, and should be omitted from the batch-mixture. In other tests a glass containing equimolecular proportions of soda and potash worked much better than one containing only one of these oxides.—A. B. S.

Glass; Apparatus for the detection of strain in —. S. English. *J. Soc. Glass Tech.*, 1919, 3, 258—260.

A TUBULAR wooden box is provided, near its lower end, with an opening through which light passes on

to a sheet of plate glass which acts as a polariser. The light reflected from the glass is passed through the specimen to be examined, then through a Nicol prism or analyser at the upper end of the box. If a specimen of strained or badly-annealed glass is examined, bright patches of light will appear. With well-annealed glass the field remains dark.—A. B. S.

Refractory materials; Reversible expansion of —.

H. J. Hodsman and J. W. Cobb. J. Soc. Glass Tech., 1919, 3, 201—222.

THE net change in volume of a refractory material which has been heated is due to the permanent after-contraction or -expansion and to a temporary change, solely dependent on the temperature. Specimens of refractory materials (usually 20 cm. by 2.5 cm.) were heated in a silica tube contained in a horizontal tubular electric furnace, and the changes in volume were measured by noting the relative movement of marks on a silica distance-piece sliding loosely in an extension of the silica tube and on the latter. Owing to abnormalities in the expansion of the silica tube and distance piece, the method cannot be used above 1000° C. Calcined alumina, magnesia brick, carborundum, calcined kaolin, and hard calcined ball clay expand regularly over the whole range of temperature (roughly $\frac{1}{2}\%$ between 0° and 1000° C. for most materials, but over 1% for magnesia bricks). The inversion of α -to β -quartz is accompanied by a very large expansion between 500° and 600° C. and is followed by a period of constant volume between 600° and 1000° C. Bricks made of equal parts of silica and fireclay behave like silica. The contraction of fireclay bricks is intermediate between kaolin and silica. On cooling, the contraction of both clay and silica just below 600° C. exceeds the corresponding expansion on heating; a rapid cooling of ware between 600° and 500° C. may, therefore, cause cracking.

—A. B. S.

Aluminous refractories; Load behaviour of —.

A. V. Bleining. J. Amer. Ceram. Soc., 1920, 3, 155—157.

HIGHLY-ALUMINOUS refractory materials are not inherently deficient in resisting pressure at high temperatures, the deformation which occurs depending largely on their previous thermal history. A refractory material which is practically constant in volume and resists a load of 50 lb. per sq. in. at 1400° C. was made by mixing calcined Pennsylvania flint clay with artificial sillimanite prepared from North Carolina kaolin 71.6% and anhydrous alumina 28.4%, the mixture being fired at cone 20 (1530° C.). The calcined materials were mixed with fireclay, to act as a bond, and were made into bricks which were fired at cone 14 or 16 (1410° or 1460° C.). The shaping of bricks by dry-pressing reduces the burning-shrinkage and the deformation under load at high temperatures.—A. B. S.

Quartzites and silica bricks. F. Wernicke. Stahl und Eisen, 1920, 40, 432—437.

FOR the manufacture of silica bricks in Germany, rocks of three different geological formations are employed, viz., felsquartzites—marine deposits belonging to the Devonian period—which have a dense structure of closely-interlocked crystals, the individual quartz grains being relatively large, with sharply-defined edges and a characteristic wavy extinction of colour; carboniferous sandstones, consisting of medium-sized quartz crystals, some having sharply-defined edges and closely interlocked, whilst in others the edges are corroded and the crystals are separated by a small quantity of cement-like material; and glacial or "amorphous" quartzites—fresh water deposits of Tertiary origin belonging to the period of lignite formation—consisting of very small grains of quartz with rounded edges distributed uniformly through an amorphous ground-

mass of silicious gel or "basaltic" cement. All these quartzites are very rich in silica; their refractoriness corresponds to that of cone 36 (1790° C.). In the manufacture of silica bricks it is not necessary to effect a complete conversion of quartz into tridymite provided the maximum expansion is attained. With glacial quartzites, this is reached by one firing at 1450° C. without excessively prolonged heating, but silica rocks from the coal measures require two or three firings before they attain their maximum expansion, and felsquartzites continue to expand after several prolonged firings. By examination under the microscope with a magnification of only 45 diameters using polarised light, useful quartzites can be distinguished from the useless ones, as the former contain minute crystals in a glassy ground-mass, whilst the latter consist almost wholly of larger crystals or sharply-defined fragments of such crystals. For the arches of electric and other metallurgical furnaces, silica bricks should be made of glacial or Tertiary quartzites. For glass-melting furnaces, where the temperature is not so high, silica bricks made from Coal Measure quartzites are satisfactory, and may be recommended on account of their greater purity and the lesser risk of spoiling the glass. Coal Measure quartzites may also be used for silica bricks employed in regenerators, coke ovens, etc. Mixtures of Tertiary and Coal Measure quartzites are also used. Coarse crystalline quartzites are only suitable for low-grade silica bricks used in annealing furnaces, etc.—A. B. S.

Terra-cotta slips, with reference to the use of asbestos and chlorite mica. H. Wilson. J. Amer. Ceram. Soc., 1920, 3, 114—133.

THE physical properties of the body (including the shapes and sizes of the grains of grog, the manner in which the particles are compressed together, and their behaviour during drying and finishing) are of great importance in producing good glazed ware. Particles of grog which are retained on a 65-mesh sieve are liable to cause surface cracks, as the clay between them shrinks whilst the grog does not. Addition of 10—15% of chlorite mica to the engobe or body slip makes the latter ink-proof when burned and decreases cracking. Addition of asbestos to a body slip renders it difficult to apply satisfactorily by spraying, but if applied by means of a spatula it produces a very resistant surface. Addition of 3% of asbestos to a glaze appears to render the use of body slips unnecessary in many cases.—A. B. S.

Boric acid frits; Solubility of —. G. Blumenthal, jun. J. Amer. Ceram. Soc., 1920, 3, 152—154.

TESTS of nine commercial frits containing boric acid, but otherwise of unknown composition, showed that all were slightly soluble in water—apparently on account of their not having been completely melted.—A. B. S.

Clay suspensions. Smith. See XIXb.

PATENTS.

Glass furnaces. J. S. Atkinson, Stein and Atkinson, Ltd., and T. C. Moorshead, London. E.P. 141,617, 11.10.19. (Appl. 24/884/19.)

A GLASS-MELTING furnace containing a rotating table is provided with a recuperator in which the incoming gas and air are heated by means of waste gases from the furnace.—A. B. S.

Kilns of tunnel and car type; Gas, coal, or oil-fired continuous —. *Gas- or coal-fired continuous kilns and dryers of the tunnel and car type.* F. Fidler, Wigan, and J. G. Maxwell, London. E.P. (A) 141,124, and (B) 141,125, 6.1.19. (Appls. 392 and 393/19.)

(A) HEAT absorbers, consisting of cast-iron panels with zig-zag flues or of arched castings forming

flues, are built in the walls, roof, etc., of the cooling zone of a tunnel kiln, so as to absorb heat radiated from the cooling goods. The heat from the absorbers is afterwards transmitted to air forced through them by means of a fan and then utilised for various purposes. (b) Goods are dried in one or more drying tunnels built alongside a tunnel and ear kiln by clean air heated by passing it through ducts in the walls and roof and beneath the floor of the cooling zone of the tunnel kiln or through heat absorbers as described under (a).—A. B. S.

Ceramic composition. O. Gerber, Witten, Assignor to The Chemical Foundation, Inc. U.S.P. 1,336,749, 13.4.20. Appl., 27.10.13. Renewed 3.9.19.

A CERAMIC composition consists of powdered glass, china, and earthenware moulded into shape and burned.—A. B. S.

Enamelling; Process for direct — by means of highly heated gases purified from dust and tar. F. Siemens, Berlin. G.P. 317,021, 5.2.18.

THE articles to be enamelled are exposed directly to flue gases for the purpose of melting down the enamel.—G. F. M.

Refractory articles; Manufacture of —. Carborundum Co. E.P. 133,014, 13.1.19. (Appl. 927/19.) Conv., 27.9.18.

SEE U.S.P. 1,296,715 of 1919; J., 1919, 365A.

Refractory materials. E.P. 140,835. See X.

Copper plating. G.P. 318,402. See X.

IX.—BUILDING MATERIALS.

"Iron" Portland cement, with a classification of cements made from blast-furnace slag. E. H. Lewis. Iron and Steel Inst., May, 1920. [Advance proof.]

MOST blast-furnace slags may be used for producing Portland cement, but only some of them are suitable for addition to Portland cement clinker to produce so-called "iron" Portland cement which is stronger than ordinary Portland cement. Failures in the use of blast-furnace slag as an aggregate for concrete have no relation to its suitability as a raw material for Portland cement or as an admixture to Portland cement clinker before grinding in order to make "iron" Portland cement. It is important to distinguish cement made from slags other than blast-furnace slag, as the former often contain an excess of sulphur are of poor quality.—A. B. S.

Preservation of railway sleepers; Effectiveness of sodium fluoride used as an antiseptic for the —. H. Devaux and H. Bouygues. Comptes rend., 1920, 170, 1006—1008.

ALTHOUGH sodium fluoride in 1% solution is an efficient antiseptic against such organisms as *Penicillium glaucum* and *Helicomyces fuliginosus*, it is futile to use it, by injection, for the preservation of railway sleepers, freely exposed to the weather, because it is very rapidly removed owing to the action of rain.—W. G.

PATENTS.

Waterproof cements, concretes, and mortars; Compositions for and production of —. H. C. Badder, S. F. Burrows, and H. L. P. Allender, London. E.P. 141,113, 3.1.19. (Appl. 180/19.)

A WATERPROOF concrete or mortar is made by adding to any suitable cement or concrete, burned clay saturated with a solution of silicate, such as sodium

or potassium silicate, and zinc, magnesium, calcium or equivalent chloride.—A. B. S.

[*Slag bricks.*] *Building material.* T. F. Hoare, Bradford. E.P. 111,390, 1.2.19. (Appl. 10,937/18.)

BRICKS, blocks, slabs, or the like may be made by running molten slag into hot moulds having double walls, the space between the walls being filled with a heat-insulating material; the moulds are then allowed to cool slowly.—A. B. S.

Wood; Process and apparatus for rendering — impermeable and recovering liquids contained therein. C. Roy. E.P. 140,361, 29.7.19. (Appl. 18,775/19.) Conv., 15.3.19. Addition to 130,981.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron ore; Rapid analysis of —. E. Little and W. L. Hult. J. Ind. Eng. Chem., 1920, 12, 269—273.

TO determine total iron, the hydrochloric acid solution of the ore, after reduction, is treated with an excess of standardised potassium bichromate solution, 5 g. of ammonium fluoride and 5 g. of potassium iodide are added, and the iodine liberated by the excess of bichromate is titrated with thiosulphate solution. The ammonium fluoride prevents reaction between the ferric compounds and the potassium iodide. Stannous chloride (followed by mercuric chloride) may be used to reduce the ferric salts, but reaction between the resulting mercurous chloride and iodine tends to make the results for iron too high; trustworthy results are obtained by using the Jones "reductor."—W. P. S.

Iron; Determination of — in iron ores by means of permanganate. R. Schwarz. Chem.-Zeit., 1920, 44, 310—311.

THE addition of silicic acid has no disturbing influence on the titration of iron in the method of Schwarz and Rolles (J., 1919, 179A, 821A) as was alleged by Brandt (J., 1919, 662A; 1920, 268A), nor does the simultaneous presence of oxygen affect the results. On the contrary, the presence of a silicic acid sol has a favourable influence.

—C. A. M.

Phosphorus in hematite iron; Determination of — and its commercial importance. N. D. Ridsdale. Cleveland Inst. Eng., Feb., 1920. Chem. News, 1920, 120, 219—221.

THE sample is dissolved in nitric and hydrochloric acids, silica is separated, arsenic is removed in the usual way, and the phosphoric acid and titanium are separated from the bulk of the iron by oxidising a small portion of the latter and precipitating it as basic ferric phosphate. The precipitate is dissolved in hydrochloric acid, oxidised with bromine, the titanium is separated by means of "cupferron," and the phosphoric acid is determined by the molybdate method. An alternative method consists in dissolving the iron in nitric acid with the addition of ammonium hydrogen fluoride, oxidising the filtered solution with permanganate, decomposing the excess of permanganate with ammonium oxalate, adding ammonium nitrate, ammonium chloride, and nitric acid, and precipitating the phosphoric acid (with more or less arsenic, silica, etc.) with molybdic acid reagent. The precipitate is collected, washed, dissolved in ammoniacal ammonium citrate solution, the phosphoric acid reprecipitated with molybdic acid reagent containing ferric nitrate, and the amount of phosphomolybdate precipitate determined volumetrically or gravimetrically.—W. P. S.

Phosphorus in iron, steel, ores, and slags; Critical examination of methods for the determination of —. H. Kinder. *Stahl u. Eisen*, 1920, 40, 381–387, 468–472.

MODIFICATIONS of the molybdate method and the magnesium pyrophosphate method were examined. In the molybdate method, the factor for converting the weight of the yellow precipitate into phosphorus is 1.64 when the precipitate is weighed after being dried at 105° C., and 1.723 when the precipitate is ignited at 450° C. Copper, nickel, and cobalt do not interfere with the molybdate method, chromium has but little if any effect, but silicon and tungsten must be removed previously; vanadic acid must be reduced previously with ferrous sulphate or sodium sulphite. The magnesium pyrophosphate method yields higher results than does the molybdate method; with a sample containing phosphorus equivalent to 15% P_2O_5 , the difference does not exceed 0.4%.

—W. P. S.

Iron; Crystallography of alpha and beta —. F. Giolitti. *Chem. and Met. Eng.*, 1920, 22, 585–589.

BELAIEV's work on Widmanstätten structure and other crystalline habits in slowly cooled steels (J., 1910, 954; 1912, 539) is discussed. The author's observations do not confirm Belaiev's explanation of his own results. The idea that ferrite is ejected to the periphery of homogeneous austenite on very slow cooling is not justified, as in the case of a uniform metal a Widmanstätten structure results. The suggestion that undercooling is partially responsible for Widmanstätten structure in slowly-cooled steels is not substantiated. The theory is advanced by the author that there are two distinct forms of ferrite crystals, one characteristic of regions with less than 0.4% carbon and the other of regions of higher carbon content. The former has the appearance of rounded grains separating from austenite as β -iron, and the latter are plate-like laminations forming as α -iron below 760° C. Confirmatory observations are cited to substantiate the theory, which explains the origin of all characteristic appearances of hypoeutectoid steels.

—T. H. Bu.

Blast-furnace practice; British —. F. Clements. *Iron and Steel Inst.*, May, 1920. [Advance proof.] 24 pages.

DATA supplied from seventeen furnaces working in various parts of the country are submitted and tabulated. The more important points dealt with are the chemical and thermal balance-sheets of the furnaces, the size of the various furnaces, the amounts, nature, and physical condition of the fuel or ore charged, the methods of charging, and the nature and treatment of the gas. For furnaces working under the same conditions of burden, coke, and blast, the larger furnaces show an advantage in fuel economy. Furnaces should be so worked that 50–60 lb. of carbon is burnt per sq. ft. of bosh per hour. A stack capacity of 0.75 cub. ft. per lb. of iron made per hour per sq. ft. of bosh should give effective results. The diameter of the furnace at the tuyeres should be designed to suit the bosh so that the zone of fusion coincides with the largest diameter of the furnace. Blowing plant and hot blast stoves should be of ample capacity to allow the furnace to be easily driven up to the maximum output conditions necessary to give economical results.—J. W. D.

Blast-furnace practice; Chemical and thermal conditions in —. H. E. Wright. *Iron and Steel Inst.*, May, 1920. [Advance proof.] 21 pages.

THE chemical and thermal reactions in blast-furnace practice are discussed. More attention should be

paid to the cleaning of coal for blast-furnace coke, and when poor fuel has to be used a richer ore ought to be mixed with it. The slag question in blast-furnaces, and the moisture content of the ore, require more consideration. To use oxygen in place of air in the blast is not considered advisable.—J. W. D.

Steel furnaces; Heating basic open-hearth — with a mixture of producer gas and coke oven gas. A. Schneider. *Stahl u. Eisen*, 1920, 40, 501–510.

A TILTING basic open-hearth furnace working on the production of carbon and special alloy steels has been heated by means of a mixture of producer-gas and coke-oven gas. An oxidising atmosphere is first produced over the bath by means of producer-gas so as to cause elimination of the carbon, then the coke-oven gas is introduced and an atmosphere poor in oxygen obtained, the bath being highly heated. A highly basic and reactive slag is formed which removes the phosphorus and sulphur, and this slag is continually removed by tilting the furnace so as to prevent oxidation of the added nickel, chromium, tungsten, or other elements.

—J. W. D.

Steel-making practice; Slag conditions in open-hearth basic —. J. F. Wilson. *Iron and Steel Inst.*, May, 1920. [Advance proof.] 22 pages.

HIGH slag volume retards the speed of heat transmission between the furnace and the bath, causes a blanketing effect, and thereby suppresses the "carbon boil" and prevents rapid elimination of the carbon, and also gives a high slag superheat. High slag viscosity exerts a blanketing effect. The rate of production is not only affected by these conditions, but the slow elimination of carbon tends to retain oxygen in the steel, and therefore affects the quality of the finished product. The condition of the slag influences the oxidation of the metalloids, especially the endothermic and high temperature carbon reaction, and consequently the efficiency of the furnace. A highly superheated slag gives a high furnace working temperature, and this affects the wear and tear of the furnace brickwork adversely.—J. W. D.

Silicon from slag; Reduction of — in the acid open-hearth process. B. Yaneske and G. A. Wood. *Iron and Steel Inst.*, May, 1920. [Advance proof.] 20 pages.

WHEN a highly silicious slag is in contact with the metal in the bath, reduction of silica takes place, as the slag contains more silica than is required to combine with the basic oxides present to preserve equilibrium. The formation of a highly silicious slag is facilitated by allowing the bath to remain too long at a very high temperature without additions of ore or lime. The reduction depends on the silica content of the slag and not on the temperature of the bath, and is effected by the excess of iron present, which is a far more powerful reducing agent than carbon.—J. W. D.

Iron; Blue-brittleness and ageing of —. Fettweis. *Stahl u. Eisen*, 1919, 39, 1–7, 34–40.

DRAWING of iron and steel causes the metal to age slowly at ordinary temperatures but rapidly at 300° C., thus giving rise to the phenomenon known as "blue brittleness." The author reviews recent work on this subject and draws the following conclusions. Iron drawn above 500° C. or at ordinary temperatures and then heated for a short time shows a maximum value for the tensile strength and a minimum value for the elastic limit and reduction in area between 200° and 300° C. The minimum value of the tensile strength occurs at 80° C., while the resistance in the repeated impact test is lowest between 200° and 300° C., and in the single impact

test between 400° and 500° C. If aged iron is annealed at different temperatures the mechanical properties will steadily vary with rising annealing temperature without attaining maximum or minimum values.—A. R. P.

Steel; Macrography of —. *Tests on corrosion*. H. Le Chatelier and B. Bogitch. *Rev. Mét.*, 1919, 16, 129—139.

PURE electrolytic iron was melted alone or with varying small quantities of sulphide or phosphide of iron either in magnesia crucibles exposed to the gas fumes and the air, or in silica tubes in an atmosphere of hydrogen. Specimens from each test were cut, polished, and etched with one of the following:—Stead's reagent (copper chloride and picric acid in alcohol), copper ammonium chloride in water, iodine solution, strong hydrochloric acid, 20% sulphuric acid. A series of photographs is given showing the different structures obtained. Stead's reagent colours those parts of the metal which contain least oxygen, while the other reagents attack the parts rich in oxygen. If no oxygen is present in the metal only the crystalline structure is developed by the etching agents. The authors consider that oxygen plays the same part in the macrostructure of steel as carbon does in the microstructure, and is the cause of the heterogeneity observed in slowly cooled or annealed metal, since it remains in solid solution in the portion of the metal that solidifies last. Sulphur may be detected in the metal by the production of black spots when it is treated with a piece of bromide paper moistened with sulphuric acid.—A. R. P.

Hypo-eutectic steels and alloys of similar constitution; Effect of annealing on micrographic structure of —. A. Portevin. *Rev. Mét.*, 1919, 16, 141—148.

ON rapidly quenching a mild steel from above Ac₃ nearly pure martensite is obtained; this, on annealing, gives a needle-like structure in the form of Widmanstätten figures, composed of complex ferrite-sorbite. If quenched between Ac₃ and Ac₁, however, irregular patches of martensite in a ground-mass of ferrite are formed; on annealing the former constituent is entirely replaced by sorbite without other alteration of the structure. Slower quenching results in the formation of troostite and martensite in a network of ferrite; after annealing the structure shows ferrite and sorbite, the parts which originally consisted of martensite having changed to complex ferrite-sorbite in the form of Widmanstätten figures. Similar results are obtained on annealing various bronzes and brasses.—A. R. P.

Ferromanganese as a deoxidising agent. A. Jung. *Stahl u. Eisen*, 1919, 39, 14—15.

THE statement that the addition of ferromanganese to steel in the converter in the presence of retained slag hinders the re-phosphorising of the metal is not quite correct. In a series of tests carried out on an iron containing 3% P a relatively large proportion of preheated solid ferromanganese was added to the converter. The slag contained 20—21% P₂O₅, and the lining of the converter was badly attacked. The phosphorus content of the metal in the converter increased by 0.01% P in the 5—7 min. which elapsed from the taking of the last dip sample until the ferromanganese had melted, and by 0.025% P from that point until the final sample was taken in the middle of pouring. By using the normal quantity of ferromanganese, a more resistant lining in the converter, and a lower temperature during the pouring, less phosphorus was taken up. Even when the ferromanganese was added in the liquid state, and the metal carefully skimmed, equally high results were obtained.—A. R. P.

Iron-nickel alloys; Forgeability of —. T. D. Yensen. *Amer. Inst. Min. Met. Eng.*, Feb., 1920. Blast Furnace and Steel Plant, 1920, 8, 231—234.

PURE iron-nickel alloys do not forge readily, if at all, at ordinary forging temperatures. Aluminium, carbon, magnesium, and silicon have little or no effect on the forgeability, but manganese and titanium in amounts of 2% make the alloys forgeable. The degree of forgeability of a material depends on the relative proportions of crystalline and intercrystalline or amorphous material, and the forgeability of an ingot or casting can only be ascertained by actually attempting to deform the material either by forging or rolling, or by a tensile test. The micro-structure gives no definite information. By the addition of manganese or titanium the amorphous material between the crystal grains is strengthened to such an extent that it becomes stronger than the crystalline material.—J. W. D.

Garnet; Electric furnace reduction of — [with production of ferrosilicon]. M. de K. Thompson and J. Davenport. *Chem. and Met. Eng.*, 1920, 22, 596.

ATTEMPTS were made to reduce garnet with coke in an electric furnace in order to remove the silicon and iron as ferrosilicon and produce a slag containing a higher percentage of alumina than the original garnet. Trials with red abrasive garnet containing 35% SiO₂, 27% Al₂O₃, 1.3% CaO, 2.2% MgO, and the remainder iron oxide, were encouraging, two runs averaging about 62% Al₂O₃ in the slag, whilst all the iron was removed and about half the silicon. The ferrosilicon contained 21—26% Si and 5—10% Al. An excess of 27% of coke gave as complete reduction as a 37% excess. Trials on a larger scale gave even better results. More silicon would probably be removed if the garnet and coke were ground finer than $\frac{1}{16}$ in. and briquetted with tar as a binder.—T. H. Bu.

Titanium alloys; Some electrical properties of —. M. A. Hunter and J. W. Bacon. *Trans. Amer. Electrochem. Soc.*, 1920, 391—401. [Advanced copy.]

NORWEGIAN iron, silicon-iron, nickel, copper, nickel-iron, and nickel-copper were alloyed with pure titanium, and magnetisation and hysteresis tests were made on the products. The titanium employed was prepared by reduction of titanium tetrachloride by sodium, and had m.pt. 1800° C. and sp. gr. 4.50. The magnetic quality of iron was improved by titanium so long as the latter functioned only as a cleansing agent; but when the iron itself contained more than a trace of titanium the magnetisation curve was lowered. Treatment of silicon-iron with titanium gave alloys having exceedingly high permeability and low hysteresis loss. The ageing of the titanium-treated iron and silicon-iron specimens was similar to but somewhat less than that of iron. The specific resistances of the remaining alloys were only moderately high for additions of titanium up to 5%. Beyond this point the alloys were difficult to draw and oxidised badly when run at a red heat.—W. E. F. P.

Copper; Determination of the coefficient of cubical expansion and the specific gravity of molten — and influence of the absorption of gas on the contraction of cast copper. W. Stahl. *Metall u. Erz*, 1919, 16, 443—444.

THE coefficient of cubical expansion of pure molten copper is 0.000054, whilst that of molten copper after refining by the poling process is 0.0000436. The difference is due to the absorption by the molten metal of hydrogen and carbon monoxide, which are partly evolved on cooling and partly remain in blowholes, thus causing the specific gravity to be

lowered from 8.95591 to between 8.3 and 8.6. The sp. gr. of pure copper at 1165° C. is given as 8.4322.

—A. R. P.

Copper-gold alloys; Hardness and resilience of —. A. Portevin and J. Durand. Rev. Mét., 1919, 16, 149—151.

THE Brinell hardness number of cast unquenched copper-gold alloys rises to a maximum, while the force required to break a notched bar falls to a minimum with the alloy containing 20% Cu. The fracture of this alloy shows a needle-like structure.

—A. R. P.

Zinc furnaces; Determination of the thermal conditions of —. O. Mühlhaeuser. Metall u. Erz, 1920, 17, 137—150.

TEN tables and twenty-two graphs compiled from a series of temperature measurements made with a Le Chatelier thermo-electric pyrometer are given. The figures were obtained in three furnaces of different construction, and show the variations observed between different tiers of retorts, different sections of the same furnace, and the front and rear walls of each section. The graphs illustrate the sudden changes in temperature occurring when the gas is turned off during the cleaning and recharging of the generator and during the emptying and refilling of the retorts.—A. R. P.

Tin in lead; Solubility in the solid state of —. N. Parravano and A. Scortecchi. Gazz. Chim. Ital., 1920, 50, i., 83—92.

THE electrical conductivity isotherms for the system tin-lead indicate the existence of solid solutions, the percentages of tin in the saturated solutions at different temperatures being as follows:—1.45 at 175° C., 13.5 at 170°, 12.5 at 162°, 10 at 150°, 6.5 at 100°, 5.0 at 75°, 3.0 at 50°, and 1.5 at 25° C. The conclusion that the recalcence observed with the solid alloys is due to simple supersaturation is confirmed. (Cf. J.C.S., June.)—T. H. P.

Anti-friction metal substitutes. Praetorius. Motor-wagon, 1920, 44—46. Chem.-Zeit., 1920, 44, Rep., 108.

LEAD-TIN alloys containing 13—80% Pb, 6—68% Sn, 13—15.4% Sb, 2.5—6% Cu, when tested in a 100 h.p. Mercedes motor, gave relatively good results up to a lead content of 33%, and also when the alloy contained 80% of lead.—A. R. P.

Mercury hydrosols; Mechanism of the formation and the properties of — prepared by various methods of dispersion. I. Nordlund. Koll.-Zeits., 1920, 26, 121—138.

By shaking mercury with aqueous solutions of potassium citrate ($2-7 \times 10^{-3}N$), strong, very stable sols of a reddish brown-grey colour were produced. Various other methods tried yielded sols of low stability. (Cf. J.C.S., June.)—J. F. S.

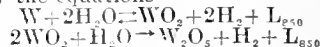
Molybdenite; Flotation of —. C. E. Oliver. Eng. and Min. J., 1920, 109, 840.

THE crushed ore (mainly quartz diorite, containing 0.5—0.75% MoS₂, distributed fairly uniformly through the rock, and iron pyrites which sometimes amounts to 3—5%) is ground in a Marcy ball mill with a mixture of pine oil and kerosene (coal oil), the mill being in closed circuit with a Dorr classifier, the overflow from which passes a 40-mesh screen. From the ball mill the pulp passes to a series of 6 Callow cells, in the first four of which ("roughing" cells) a concentrate containing 10—15% MoS₂ (and a similar proportion of pyrites) is obtained. This product passes to the two remaining ("cleaner") cells, where a concentrate containing 60—70% MoS₂ is produced. The latter is washed with water on an 80-mesh Callow screen to remove

the remaining pyrites and adhering gangue, the concentrate being removed from the under side of the screen by a spray of water and treated in a dewatering tank, from which it is passed continuously to a dryer. The final concentrate contains 85—95% MoS₂ and 2—3% FeS₂. The recovery of molybdenite rarely falls below 98% over a month's working, the oil consumed being 0.5 lb. pine oil and 1.0 lb. coal oil per ton of ore.—W. E. F. P.

Tungsten and its oxides; Reversible reactions of water on —. G. Chaudron. Comptes rend., 1920, 170, 1056—1058.

THE equilibrium of the system tungsten-water-oxides of tungsten-hydrogen at 850° C. may be expressed by the equations



The value of L_{850} for the first reaction is 2×4.4 Cals. and for the second 4 Cals.—W. G.

Iron and steam. Schreiner and Grimnes. See VII.

Cements from blast-furnace slag. Lewis. See IX.

PATENTS.

Alloy [not brittle at low temperatures]. Soc. Anon. de Commentry, Fourchambault et Decazeville, Paris. E.P. 127,243, 29.4.19. (Appl. 10,604/19.) Conv., 22.5.18.

AN alloy, suitable for use in liquefied gas machinery as it resists corrosion and retains good mechanical properties at a low temperature, is composed of Ni 40—70%, Mn 0.8 to 5%, iron the remainder. For part of the iron (5% at least must be left), and for part of the nickel (up to 7.5%) may be substituted all or any of C 0—1%, Cr 0—5%, Cu 0—10%, Co 0—10%, V 0.2—1%, Ti 0.1—0.5%.—B. M. V.

Alloy containing iron, nickel, and chromium. Soc. Anon. de Commentry, Fourchambault et Decazeville, Paris. E.P. 140,507, 17.12.18. (Appl. 21,068/18.) Conv., 19.12.17.

THE alloy contains 0.2—0.5% C, 1—2% Mn, 10—15% Cr, and 20—25% Ni, and the remainder iron, to which may be added 2—5% W. It is resistant to shock or high pressure, also to the action of strong acids, organic acids, and alkalis.—T. H. Bu.

Alloy comprising iron, nickel, chromium, tungsten (or molybdenum). Soc. Anon. de Commentry, Fourchambault et Decazeville, Paris. E.P. 140,508, 17.12.18. (Appl. 21,069/18.) Conv., 20.12.17.

THE alloy contains 0.3—0.6% C, 1—2% Mn, 10—15% Cr, 60—70% Ni, 2—5% W, and the remainder iron, to which may be added 0.2—1% V, 0.1—0.2% Ti, up to 10% Co, and 1—3% Mo to replace or partly replace the tungsten. The alloy possesses high mechanical strength at high temperatures, and is resistant to the action of chemicals.—T. H. Bu.

Alloy containing iron, nickel, and chromium. Soc. Anon. de Commentry, Fourchambault et Decazeville, Paris. E.P. 140,509, 17.12.18. (Appl. 21,070/18.) Conv., 21.12.17.

THE alloy contains 0.3—1% C, 0.5—1% Mn, 10—15% Cr, 25—40% Ni, and the remainder iron, to which may be added 2—5% W, 0.2—1% V, 0.1—0.2% Ti, up to 10% Co and 1—3% Mo. It is resistant to superheated vapours, and hot and moist gases, has good wearing properties, and its coefficient of expansion may be varied between 8 and 17×10^{-6} .

—T. H. Bu.

Steel and steel alloys; Process and apparatus for manufacturing —. W. E. Moore, Pittsburgh. U.S.P. 1,335,009, 30.3.20. Appl., 19.11.18.

STEEL is subjected to the action of an electric arc in the presence of a slag, and is then transferred to

a second furnace, where it is subjected to the heat of a shorter electric arc, in the presence of a slag of different character from that of the first slag.
—T. St.

Iron and steel; Method of carbonising — J. H. L. De Bats, Assignor to New Process Metals Corporation, Wilmington. U.S.P. 1,335,094, 30.3.20. Appl., 19.8.18.

PIECES of iron or steel to be carbonised are subjected to a preliminary heating to approximately 1100° C. to render them porous. They are then cooled, packed in carbonising material, and heated to a carbonising temperature.—T. St.

Cast-iron; Desulphurising — C. Ellis, Montclair, Assignor to Ellis-Foster Co. U.S.P. 1,335,370, 30.3.20. Appl., 9.10.17.

MOLTEN cast-iron is brought into contact with a mixture containing calcium cyanamide and calcium carbide.—T. H. Bu.

Mild steel; Desulphurisation of — H. Kaussen, Aix-La-Chapelle. G.P. 308,482, 12.3.16.

STEEL is desulphurised in the basic converter or open-hearth furnace by means of lime and iron carbide according to the equation $FeS + CaO + Fe_3C = 4Fe + CaS + CO$. The lime is derived from the excess contained in the fluid slag, and it is used with an admixture of fluid iron carbide collecting between the iron and the slag.—G. F. M.

Iron; Electrolytic extraction of metallic — directly from its ores. W. Pip, Darmstadt. G.P. 316,597, 26.6.18.

METALLIC iron is deposited electrolytically from a hot ferrous salt solution, and the ferric salt which is thereby continually formed is re-converted into ferrous salt by bringing the electrolyte into contact with spathic iron ore, whereby continuous cathodic deposition of the metal is made possible.—G. F. M.

Iron and iron alloys; Process for the preparation of pure — in powder form. Allgem. Elektrizitäts-Ges. G.P. 316,748, 24.5.16.

IRON or its alloys may be deposited electrolytically in a pulverulent form by the use of a high current density and low temperature in a bath consisting of a weak solution of a ferrous salt together with a volatile conducting salt (e.g. ammonium chloride). With a bath containing 3 pts. of ferrous chloride and 10 pts. of ammonium chloride in 100 pts. of water, and a current density of 10 amp. per sq. dm., at room temperature, a yield of 70–80% of the theoretical may be obtained. The iron deposit may be used as a catalyst or for the preparation of articles or salts of iron. If an anode of nickel-steel is employed a carbon-free, friable deposit of the alloy is obtained.—A. R. P.

Steel; Hardening bath for — E. Deisenhammer and K. Neudecker, Ratibor. G.P. 316,800, 3.7.18.

GLYCOLS, concentrated or dilute, may be used for hardening steel. The hardening effect of ethylene-glycol is increased by mixing the glycol with water.
—T. St.

Brass; Manufacture of — P. Dutoit and A. Boever, Lausanne. E.P. 132,520, 10.9.19. (Appl. 22,296/19.) Conv., 11.9.18.

BRASS made either from new metals or from skimmings, ashes, etc., is melted by submerging the raw material under a bath of molten salt of such gravity that slag will float but clean metal will sink. Barium chloride is the most suitable salt, and may be melted by an electric arc and kept molten by means of its resistance, the electrodes being so situated that the current passes through the slag and salt rather than through the molten metal.
—B. M. V.

Electrodes and welding and like rods used in soldering and depositing metals. E. H. Jones, and Alloy Welding Processes, Ltd., London. E.P. 140,568, 26.2.19. (Appl. 4848/19.)

Two or more metals or alloys are united by casting one of the metals or alloys in or about the others, or by brazing, soldering, or welding them together to form a welding rod, which is formed into an electrode by covering it with asbestos or other second-class conductor. (Cf. J., 1916, 1222; 1917, 1135; 1918, 705 A.)—B. N.

Crucible or melting pot furnaces; Tilting — Soc. des Alliages et Bronzes Forgeables, Paris. E.P. 140,709, 26.9.19. (Appl. 23,747/19.) Conv., 22.7.19.

THE tilting axis is tangent to the upper edge of the crucible over which the metal is poured, and part of the furnace body is detachable to allow the introduction of a sloping refractory channel piece through which the molten metal flows, so that a minimum surface is exposed to the atmosphere.
—T. H. Bu.

Metal smelting furnaces. A. Sinclair, Sunderland. E.P. 140,948, 2.7.19. (Appl. 16,588/19.)

A CUPOLA or similar furnace is provided with means for closing the top, and has an annular chamber surrounding the top to which air under pressure is delivered; pipes built into the walls of the furnace lead the air from this chamber to the fuel space of the furnace. The top and bottom of the cupola are also connected by passages controlled by damper slides and having in their lower parts ejectors supplied with air from the annular chamber by means of supply pipes.—J. W. D.

Furnaces for the production of mineral distillates of definite composition. W. J. Fadden, London. E.P. 141,089, 7.10.18. (Appl. 16,229/18.)

THE apparatus comprises a furnace, cooling and depositing chambers, and filters, and the draught through the cooling chambers and the filters is controlled from a position adjacent to the furnace. The furnace, into which the ore is fed by means of a conical hopper and rotating distributor, is divided into three zones, the upper zone being provided with means for admitting air for the oxidation of the distillate. The filter comprises a settling and admission chamber provided with means for removal of the deposit, a casing divided into a number of sections, each fitted with tubular filter bags, means for admitting gas and fume to each section independently, and gas outlets communicating with each filter section.—J. W. D.

Open-hearth furnaces. E. and W. Waring, Sheffield. E.P. 141,132, 8.1.19. (Appl. 532/19.)

IN an open-hearth furnace for the continuous production of steel direct from ore and scrap metal, three separate baths are maintained on different levels. The highest and largest bath is used for reducing the ore or scrap which is fed from a cupola stack over the bath; the middle and next largest bath is used for refining, while the lowest and smallest bath is in the form of an electric furnace with an undulating hearth, and is used for super-refining. Tuyères delivering to the respective baths lead from a common flue which is connected to a regenerative chamber.—J. W. D.

Furnace; Metal-heating — H. D. Williams, Pittsburgh, and W. Ahlen, Duquesne. U.S.P. 1,336,266, 6.4.20. Appl., 27.7.16.

THE furnace-heating chamber contains a number of large gas burners and, at one end, a number of small gas burners, all supplied with preheated air under pressure. The air is preheated by being passed, by means of a fan, successively through a heating chamber, a recuperator, and a second

heating chamber. A valve-controlled conduit connects the latter to the large burners, and a valve-controlled branch conduit connects the large and small burners.—T. St.

Gas crucible-furnacc. L. Tréfois, Brussels. G.P. 318,044, 15.12.17.

The heating chamber of the furnace is surrounded by two hollow-walled metal cylinders, leaving an annular space between them through which the hot burnt gases escape. The air and gas are preheated by passing them through the hollow walls of the two cylinders respectively, on their way to the burner, whilst the inner cylinder is further heated by radiation from the heating chamber itself. By this arrangement even inferior gas may be used for melting copper, nickel, silver, gold, alloys, etc.

—G. F. M.

Metals and refractory materials; Treatment of — and of tools or crucibles made thereof, and electric furnaces used therefor. G. Marsh, London. E.P. 140,885, 4.7.18. (Appl. 10,997/18.)

METALS or refractory materials, or tools or crucibles made therefrom, are heated by metal which has been melted by an electric arc, and are also electrically heated by a current passed through conductors connected to the refractory material and molten metal respectively. A pocket is made in the furnace wall to receive the metals or refractory materials.

—B. N.

Hard metal alloys, and methods of making same. G. H. Worrall, Missouri. E.P. 141,122, 6.1.19. (Appl. 374/19.)

A HARD lead alloy consisting approximately of Ba 1%, Ca 0.50%, Hg 0.25%, Sn 0.25%, Zn 0.10%, Al 0.02%, Pb 97.88% is prepared by alloying lead electrolytically with an alkali or alkaline-earth metal, increasing the hardness of this base alloy by adding a small percentage of mercury, then adding aluminium mixed with tin and zinc.—J. W. D.

Alloy; Metallic —. F. S. Hodson, Philadelphia. U.S.P. 1,335,284, 30.3.20. Appl., 12.6.18.

THE alloy contains 80–85% Zn, 0.001–2% Mn, and 0.0005–1% Ti, the remainder being mostly aluminium and copper in the ratio of about 9 to 1.

T. H. Bu.

Metal; Introducing [alloying] materials into molten —. A. Pacz, Assignor to General Electric Co., New York. U.S.P. 1,337,093, 13.4.20. Appl., 30.9.18.

IN alloying several metals very different in specific gravity with another metal, the former metals, in a finely divided condition, are made into briquettes and dropped into a molten bath of the latter metal.

—B. M. V.

Copper-bearing sands; Process of treating —. C. H. Benedict and H. C. Kenny, Lake Linden. U.S.P. 1,335,474, 30.3.20. Appl., 7.10.19.

THE sands are arranged in a bed or layer, and the copper is extracted by an ammoniacal solvent. The bed is then heated by means of steam, and water is vaporised in the heated bed under less than atmospheric pressure to recover absorbed ammonia.

—T. H. Bu.

Copper from lyes resulting from the treatment of cupriferous pyrites; Process for obtaining —. H. P. Soulié-Cottineau, Paris. E.P. 141,290, 31.10.19. (Appl. 26,819/19.)

COPPER is deposited electrolytically on iron in the form a film either by placing copper-coated iron

in a cupriferous solution or lye, or by causing the lye to fall on to the iron, when depolarisation takes place and a film of copper is deposited due to the presence of the copper-iron couple.—J. W. D.

Electrolytic process and anode. [Electrolytic deposition of copper.] F. L. Antisell, Perth Amboy. U.S.P. 1,313,246, 19.8.19. Appl., 17.8.18.

THE formation of ferric sulphate during electrolysis of solutions obtained by leaching low-grade copper ores with sulphuric acid, is reduced by dividing the anode surface into separate areas of active conductive material and of inactive non-conductive material. The anode may consist, for example, of a sheet of lead provided with a number of plugs of insulating material, the surface of the plugs being flush with the surface of the lead. The area of the active surfaces in the aggregate is appreciably smaller than the area of the cathode surface.

—B. N.

(A to D) *Electrolytic apparatus and method.* (E) *Electrolytic process and product.* M. M. Merritt, Assignor to Copper Products Co., Boston. U.S.P. (A) 1,335,174, (B) 1,335,175, (C) 1,335,176, (D) 1,335,177, 30.3.20, (E) 1,335,846, 6.4.20. Appl., (A to C) 2.8.18, (D) 18.9.18, (E) 2.11.17. (E) Renewed 4.12.19.

(A) METAL is deposited electrolytically on a stationary cathode, the level of the electrolyte being varied periodically to provide alternate immersion in and withdrawal of the cathode from the liquid without substantially changing the position of the cathode. (B) The cathode support has a provision for alternate immersion in, and withdrawal of the cathode from, the electrolyte. (C) Metal is deposited on segregated surfaces of objects by mounting them on a shaft, which is rotated during electro-deposition, so that a portion only of the surfaces to be plated is immersed in the electrolyte, and each surface is alternately immersed in and withdrawn from the liquid. (D) A mandrel, mould, or matrix is used as a cathode, and electrolytic deposition of metal is started in an alkaline electrolyte and continued in an acid electrolyte. (E) An aeroplane propeller is coated with an electrically conductive paint, and metal is deposited electrolytically upon it whilst rotating the propeller partially submerged in an electrolyte, the direction of rotation being periodically reversed.—B. N.

Copper plating; Process for —. M. Enderli, Karlsruhe. G.P. 318,402, 11.4.17.

A HOMOGENEOUS layer of copper formate, spread on the object on which a coating of copper is desired, is heated to a uniform temperature preferably in a non-oxidising atmosphere, pressure being applied either before or during this treatment. The process is designed for the production of a homogeneous deposit of copper on glass, porcelain, or metals, for the preparation of glazes in the ceramic industry, and of catalytic contact materials, and as a substitute for the electrolytic deposition of copper.

—G. F. M.

Electrolytic refining of metals or alloys containing a high percentage of foreign metal. Siemens und Halske A.-G. G.P. 317,146, 23.3.18.

SMALL quantities of acids, or their salts, of which the anions are capable of temporarily dissolving the foreign metal in the anode, are added to the electrolyte. By this means the layer of foreign metal which would otherwise form on the anode is loosened and its removal facilitated. In refining tin with a high content of lead or copper in a sulphuric acid electrolyte an addition of hydrofluosilicic acid is suitable. Hydrofluoboric acid, ethylsulphuric acid, or perchloric acid may also be used.—G. F. M.

Ores; Method of treating —. F. W. Wood, Baltimore. U.S.P. 1,331,981, 30.3.20. Appl., 24.6.14. Renewed 28.7.19.

PLASTIC ore containing 8% or more of alumina is dried to adjust its content of hygroscopic water to 14–20%, then subjected to a heavy pressure to form briquettes, which are charged into a smelting furnace. The drying prevents the ore from adhering to the compressing surfaces and facilitates the briquetting operation.—J. W. D.

Metalliferous materials; Process of treating —. Metallurgical process. H. B. Hoyland, Duluth, and G. B. Frankforter, Minneapolis. U.S.P. (a) 1,335,000 and (b) 1,335,001, 30.3.20. Appl., 15.8.13. (b) Renewed 23.10.16.

(A) THE material is treated with a solvent for the metal which is to be recovered, and the latter is then precipitated as sulphide in the presence of the gangue. The sulphide is subsequently separated from the gangue by flotation. (b) Metalliferous material is roasted to convert the metal to be recovered to oxide, and this is then, at least partially, converted to sulphide. The treated material is subsequently subjected to flotation treatment.—T. St.

Precious metals; Process for preventing distortion of — at temperatures higher than their fusing-points, and articles produced thereby. R. J. Peschko, Assignor to The Dentist's Supply Co., New York. U.S.P. 1,335,024, 30.3.20. Appl., 29.11.18.

THE precious metal or alloy is coated with a metal which is capable of being fused and oxidised at a relatively low temperature, and is then heated so that the coating metal partly fuses into the precious metal, and partly oxidises with the formation of an oxide skin. When, subsequently, the precious metal is heated to temperatures above its fusing point, the oxide skin suffices to sustain it against flow.—T. St.

Copper or copper-containing alloys; Prevention of selective corrosion of tubes and machinery parts of —. F. von Wursterberger, Assignor to Akt.-Ges. der Maschinenfabriken Escher Wyss und Co., Zürich. U.S.P. (a) 1,335,209 and (b) 1,335,210, 30.3.20. Appl., 7.6 and 11.6.19.

(A) TUBES or machinery parts of copper or copper-containing alloys in contact with water containing ions are protected by producing electrolytically in the water an easily soluble hydrolytically decomposable acid-reacting salt as anodic product, the cathodic product being carried away. (b) A salt having, upon hydrolysis, an acid reaction, is produced by electrolysis in a vessel separate from, but connected to the water supply of the machinery etc. to be protected.—T. St.

Zirconium and lead; Process for producing an alloy of —. N. Petinot, New York, Assignor to United States Ferro-Alloys Corp. U.S.P. 1,335,982, 6.4.20. Appl., 18.8.19.

ZIRCONIUM oxides and burnt lime are melted together in proportions to form calcium zirconate (CaZrO_3), sufficient carbon is added to reduce the zirconium oxide to metal and to form calcium carbide, and the mixture added to a molten bath of lead.—J. W. D.

Zirconium carbide; Process for producing alloys containing —. N. Petinot, New York, Assignor to United States Ferro-Alloys Corp. U.S.P. 1,335,983, 6.4.20. Appl., 18.8.19.

A MIXTURE of zirconium ore, iron, and carbon is melted, and then cooled rapidly, to obtain an iron alloy containing zirconium carbide or a double carbide of iron and zirconium.—J. W. D.

Alloy of iron, zirconium, and titanium, and process for the production thereof. Process for producing zirconium steel. H. C. Sicard, Buffalo, Assignor to United States Ferro-Alloys Corp. U.S.P. (a) 1,335,991 and (b) 1,335,992, 6.4.20. Appl., 18.8.19.

(A) AN alloy of iron, zirconium, titanium, and carbon is prepared by melting in an electric furnace a mixture of baddeleyite ore, rutile ore, scrap iron, and sufficient carbon both to reduce the oxides of zirconium and titanium and to combine with the zirconium, titanium, and iron to form a complex carbide in the finished alloy. (b) Zirconium steel is made by adding to molten steel an alloy prepared as described under (a).—J. W. D.

Aluminium; Solder for —. J. G. Kelly, Assignor to H. Hall, Sedalia. U.S.P. 1,336,081, 6.4.20. Appl., 27.9.18.

A SOLDER consisting of Pb 31, Sn 34, Zn 30, and Al 2%.—J. W. D.

Aluminium; Process for hardening —. H. M. Buluckian, Cleveland. U.S.P. 1,336,420, 13.4.20. Appl., 31.5.19.

ALUMINIUM is hardened by adding gum-glutin, oyster shell, and gum arabic to the molten pure metal.—W. F. F.

Aluminium; Process for increasing the resistance of — towards acid and alkaline liquids. Metallhütte Baer und Co., Kommandit-Ges., Abt. der Metallind. Schiele und Bruchsalser. G.P. 318,141, 9.3.19.

THE aluminium is covered with a layer of powdered calcium carbonate, heated to about 550° C., and the coating then united with the metal by hammering, rolling, or pressing. Aluminium thus treated is suitable for packing preserves.—G. F. M.

Zinc-aluminium alloys; Process for improving the mechanical properties of —. Hedderheimer Kupferwerk und Süddeutsche Kabelwerke A.-G. G.P. 318,346, 18.1.17.

ZINC-ALUMINIUM alloys containing 10–60% Al are submitted to mechanical working (pressing, rolling, hammering, etc.) at temperatures above that which is most suitable for rolling the zinc (120°–150° C.), and the metal is then heated to temperatures lying between 150° C. and the temperature of the conversion of the compound Al_2Zn_3 into a mixture of α - and γ -mixed crystals (e.g., 256° C. for the binary system aluminium-zinc). Alternatively, for this thermal after-treatment temperatures above the conversion point of the compound Al_2Zn_3 may be employed, but care must then be taken that the subsequent cooling occurs very slowly, otherwise, although the hardness and density may be increased, the toughness and ductility may under certain circumstances sink to very low values.—G. F. M.

Cobalt; Process of recovering —. M. J. Udy, Assignor to Haynes Stellite Co., Kokomo. U.S.P. 1,336,765, 13.4.20. Appl., 14.8.19.

ORES containing cobalt are subjected to a sulphating roast in the presence of sulphuric acid, the soluble sulphates are extracted with water, precipitated with calcium carbonate, copper removed by deposition, and sufficient lime added to form a basic insoluble sulphate. The sulphate bath is then electrolysed with an insoluble anode so that proportional quantities of the basic sulphate are dissolved as the acidity of the bath tends to rise above the normal.—J. W. D.

Metallic surfaces; Testing — for variations in composition. Allgem. Elektrizitäts-Ges. G.P. 317,603, 4.10.18.

IN order to detect local currents and potential

differences which may exist between different metals, or different portions of the surface of the same metal, particularly in connection with machines which come into contact with fluids and where electrolytic disturbances may consequently be set up resulting in the gradual destruction of the more electropositive portions of the metallic surface, the surface to be tested is covered with a suitable electrolyte kept in position in any desired spot by means of members of porous non-conducting material, the capillaries of which are filled with the electrolyte, and are provided with two electrodes which are connected on the one hand through a potentiometer and, on the other, make contact with the surface of the metal, whereby the potential difference, if any, of the galvanic element thus formed can be determined.—G. F. M.

Coating steel or iron with white metal; Process for —. E. W. Riley. E.P. 141,092, 19.11.18. (Appl. 18,982/18.)

Annealing [malleable iron castings]. A. Howgate. E.P. 141,200. (Appls. 10,637, 30.4. and 25,975, 23.10.19.)

Furnaces for drying foundry cores, annealing tin-plates and for other like purposes. W. E. Brown, W. G. Beeston, and J. J. Stevenson. E.P. 141,100, 6.12.18. (Appl. 20,231/18.)

Furnaces [for heat treatment of metal bars, etc.]. H. Fuller and R. A. Bedford. E.P. 141,403, 12.12.18. (Appl. 20,734/18.)

Metallurgical and like furnaces; Means for facilitating the repair of —. F. Hardy. E.P. 141,556, 12.6.19. (Appl. 14,813/19.)

Gold alloys. D. Belais. E.P. 141,246, 26.6.19. (Appl. 16,133/19.)

SEE U.S.P. 1,330,231 of 1920; J., 1920, 270 A.

Zinc compounds. G.P. 317,702. See VII.

Leather charcoal. G.P. 305,555. See XV.

XI.—ELECTRO-CHEMISTRY.

Lead accumulator; The reactions of the —. D. A. MacInnes, L. Adler, and D. B. Jonbert. Trans. Amer. Electrochem. Soc., 1920, 383—390. [Advance copy.]

AN experimental study of the amount of sulphuric acid consumed during the discharge of a lead accumulator gave results intermediate between those corresponding to the reactions $\text{PbO}_2 + \text{Pb} + 2\text{H}_2\text{SO}_4 \rightleftharpoons 2\text{PbSO}_4 + 2\text{H}_2\text{O}$, and $\text{Pb}_2\text{O}_3 + \text{Pb} + \text{H}_2\text{SO}_4 \rightleftharpoons \text{PbSO}_4 + 2\text{PbO} + \text{H}_2\text{O}$, respectively (Pb_2O_3 represents a state of oxidation higher than PbO_2). Careful chemical analyses of the oxide formed during charging have shown it to be PbO_2 . The chemical potential of the oxide on the positive plate is identical with that of chemically prepared or electrically precipitated PbO_2 . It is suggested that intermediate oxides, such as Pb_2O_3 , or basic compounds, such as $(\text{PbO})_x\text{PbSO}_4$, may be formed during discharge.—B. N.

Electric heater for distillation test. Hill and Chillas. See III.

Reduction of nitronaphthalenesulphonic acids. Fierz and Weissenbach. See III.

Platinised glass electrodes. Meillère. See XXIII.

Arc images in chemical analysis. Mott. See XXIII.

PATENTS.

Storage-battery plates; Process and apparatus for treating —. H. M. Martin, Assignor to Philadelphia Storage Battery Co. U.S.P. 1,301,871, 22.7.19. Appl., 14.11.14.

NEGATIVE storage-battery plates with their active material reduced to the metallic condition are dried in an atmosphere of a gas, e.g., hydrogen and/or nitrogen, which is chemically inert with respect to the active material. The dried plates may be kept for a long period without deterioration provided moisture is excluded.

Electric furnace. M. Ruthenburg, Buffalo. U.S.P. 1,335,199, 30.3.20. Appl., 4.5.18.

THE shell of the furnace is made of heat-transmitting material, and is surrounded by a resistance element substantially co-extensive with the outer surface of the shell, the element comprising a number of members individually removable for replacement.—B. N.

Electric furnace. L. Almagia and E. Guerreschi, Assignors to Soc. Anon. Ital. Gio. Ansaldo & Co., Cornigliano Ligure. U.S.P. 1,336,807, 13.4.20. Appl., 23.9.18.

ELECTRODES in the hearth and in the cover of the furnace are connected to a transformer by conducting wires. A second transformer is interposed in series in the conducting wires leading to the hearth.—B. N.

Electric furnace. S. Barfoed, Berkeley. U.S.P. 1,336,939, 13.4.20. Appl., 25.9.16.

THE furnace comprises a long water-jacketed tube with an electrode in each end. Air or gas admitted at one end is directed through the sphere of action of the electric arc, and the other end of the tube is provided with means for rapidly cooling the air or gas after it leaves the arc.—B. N.

Electric furnace. J. A. Pickens. U.S.P. 1,337,156, 13.4.20. Appl., 14.6.19.

THE furnace comprises a combustion chamber equipped with a pair of electrodes, one of which is solid, whilst the other has an aperture and a chambered opening longitudinally through its central portion. The electrodes are insulated from the surfaces of the combustion chamber.—B. N.

[*Electrical*] *ozonator.* M. W. Franklin, Assignor to General Electric Co. U.S.P. 1,337,062, 13.4.20. Appl., 3.8.16.

THE apparatus comprises a receptacle in which a cooling medium may be received, and a conducting tube passing through the receptacle and sealed in the walls. An ozone unit, arranged in the tube, comprises a dielectric (glass) tube having a closely adhering metal coating on its outer surface electrically connected to the conducting tube and a metal electrode within the glass tube, and separated from it to form a space through which an electrical discharge may be passed.—B. N.

Carbon monoxide; Electromotive activation of —. K. Hofmann, Charlottenburg. G.P. 310,782, 29.5.18.

GASES containing carbon monoxide, e.g. producer gas, are brought into contact with an electric conducting surface containing copper, such as copper gauze moistened with an alkaline electrolyte. Separated from this by the electrolyte, consisting of 5—25% caustic soda solution, is an oxygen (air) or other oxidising electrode such as manganese dioxide or nickel peroxide, or a copper plate covered with hydrated copper oxide. The electrolyte is maintained by the addition of slaked lime and the further addition of 5—15% of sodium chloride and 0.5% of sodium thiosulphate or sulphide is advantageous.

The cell gives at ordinary temperatures an E.M.F. of 0.9 volt, and by the interposition of a copper gauze to prevent the diffusion of oxygen a maximum of 1.4 volts is obtainable. (Cf. J., 1918, 765 A.)
—G. F. M.

Electric furnace. E. L. Crosby. U.S.P. 1,336,820, 13.4.20. Appl., 3.5.18.

SEE E.P. 132,879 of 1918; J., 1919, 870 A.

Electrically classifying finely-divided material. U.S.P. 1,335,758. See I.

Electro-osmosis. G.P. 310,681. See I.

Electrical precipitation. G.P. 316,703. See I.

Electrolysers. E.P. 141,365, and U.S.P. 1,309,214 and 1,336,281. See VII.

Calcium hydride. G.P. 311,987. See VII.

Electrodes. E.P. 140,568. See X.

Electrolytic apparatus and methods. U.S.P. 1,335,174-7 and 1,335,846. See X.

Testing metallic surfaces. G.P. 317,603. See X.

Denatured alcohol. E.P. 140,527. See XX.

Chlorhydrins. E.P. 140,831. See XX.

Pinacone. G.P. 306,304. See XX.

Mellitic acid. G.P. 318,200. See XX.

XII.—FATS; OILS; WAXES.

Linseed oil; Determination of the hexabromide value of —. H. Wolff. *Farben-Zeit.*, 1920, 25, 1213—1214.

THE yield of hexabromido and amount of unsaponifiable matter in linseed oil are determined in one operation:—5 g. of the oil is boiled for 15 mins. beneath a reflux condenser with 30 c.c. of 5% alcoholic potassium hydroxide solution, the mass dissolved in 50 c.c. of hot water, and the soap solution rapidly cooled and transferred to a separating funnel, the flask being twice rinsed with 5 c.c. of water. It is then shaken with about 100 c.c. of petroleum spirit. The lower layer is transferred to a second separating funnel, acidified with a slight excess of hydrochloric acid, and, after the addition of about 5 g. of sodium chloride, shaken with ether. The ethereal extract is separated, dried with anhydrous sodium sulphate and filtered into a weighed flask. The solvent is evaporated on the water-bath, while a current of dry carbon dioxide is passed through the flask, and the residue of fatty acids dissolved in 50 c.c. of ether. 10 c.c. of this solution is acidified with 1 drop of glacial acetic acid in a weighed centrifugal bottle, and then with 1.0 to 1.2 c.c. of bromine, added drop by drop at -10° to 0° C. After standing for 2 hours in the freezing mixture the ethereal solution is centrifuged, the ether decanted, the precipitate stirred with about 2 c.c. of chilled ether and again centrifuged. This process is repeated, and the washed precipitate is dried at 110° C. and weighed. The amounts of hexabromide yielded by different linseed oils may vary up to 8%, and the method is therefore preferable to that of Steele and Washburn (J., 1920, 197A), in which the yields are about 3% lower, but fairly constant for different oils.—C. A. M.

Hubbard squash [Cucurbita maxima] seed oil; Composition of —. W. F. Baughman and G. S. Jamieson. J. Amer. Chem. Soc., 1920, 42, 152—157.

A SAMPLE of the cold-pressed oil had sp. gr. 0.9179 at $25^{\circ}/25^{\circ}$ C.; n_D^{25} 1.4714, iodine value (Hanus)

121.0 (liquid acids 151.2, solid acids 3.8); saponif. value 191.5 (insoluble acids 201.8, liquid acids 201.7, solid acids 210.3); mean molecular weight, insoluble acids 278.0, liquid acids 278.1, solid acids 266.8; Reichert-Meißl value, 0.37; Polonske value, 0.39; acetyl value, 27.8; acid value, 0.50; unsaponifiable matter, 1.06; soluble acids, 0.33%; insoluble acids, 91.66; unsaturated acids, 76.45; saturated acids, 18.37; solidif. pt. of insoluble acids, 29.8° — 29.85° C. The oil contains the glycerides of palmitic acid (13%), stearic acid (6%), arachidic acid (about 0.04%), oleic acid (37%), and linolic acid (44%).
—H. W.

Okra [Abelmoschus esculentus] seed oil. G. S. Jamieson and W. F. Baughman. J. Amer. Chem. Soc., 1920, 42, 166—170.

FOUR samples of cold-pressed okra seed oil have been examined; the oils vary somewhat in composition. One of them was found to contain the glycerides of palmitic acid (27.23%), stearic acid (2.75%), arachidic acid (0.05%), oleic acid (43.74%), linolic acid (26.62%), and unsaponifiable matter (0.37%). Okra seed oil, as well as oils obtained from other members of the *Malvaceae*, give the Halphen colour test.—H. W.

Fat; Extraction of — from small animals. P. Lindner. Z. Tech. Biol., 1919, 7, 213—220.

THE author investigated the possibility of recovering fat from insects and small animals, e.g. worms, larvae, weevils, etc., but the working costs appeared prohibitive. In Argentina 16—18% of fat has been obtained from cockroaches by drying, grinding, and extracting with a solvent. A further suggestion by the author was to convert faeces, on a large scale, into fat and protein in the bodies of the larvae of flies. The offspring of 14 female flies would in four months produce sufficient larvae to work up the daily faeces of 70 million human beings in a few days. Arrangements for the cultivation of the larvae are described.—J. H. L.

Cholesterol and higher fatty acids; Nephelometric values of —. F. A. Csonka. J. Biol. Chem., 1920, 41, 243—249.

THE values are influenced by the saponification process and by the presence of other substances.
—J. C. D.

Catalysts. Rupert. See I.

Oxidation of hydrocarbons. Franek. See IIa.

Lignite tar oils. Harries. See III.

PATENTS.

Oil extraction apparatus. H. Engel, Hatch End. E.P. 140,513, 20.12.18. (Appl. 21,410/18.)

MEAL or other material is extracted in a combined heating and stirring apparatus containing hollow propellers mounted on a shaft and separated by distance pieces. Steam or other heating agent is passed through the propeller blades, passing from one propeller to the next through annular passages in the distance pieces.—C. A. M.

Fat; Production of — by cultivation of mould-fungi. Kriegsausschuss für pflanzliche und tierische Öle und Fette, Berlin. G.P. 306,365, 11.5.15.

MOULD fungi and yeasts, e.g., *Oidium*, *Sachsisia*, or *Endomyces* species are cultivated in liquids containing carbohydrates and nutrient salts, e.g., molasses or beer wort, the liquids having a large surface exposed to the air. After the vegetative period formation of fat is induced by strong aeration and restricted access of moisture whilst the organisms are deprived of nutriment.—J. H. L.

Hydrocarbons; Method of making solid — emulsifiable with water. St.-Rochus-Ges.m.b.H., Charlottenburg. G.P. 308,442, 25.1.17.

SOLID hydrocarbons are mixed with wool fat and/or waxes, and the mixture heated with concentrated caustic alkali solution to above 160° C. under a pressure of not less than 5 atm. Oxidising materials, such as sodium peroxide, percarbonate, perborate, etc., or organic peroxides, may be added. Solutions of the product in liquid hydrocarbons set to pasty masses, which may be mixed with medicaments for use as ointments, etc., or may be used in the preparation of solidified oils etc.—T. St.

Oxygen-yielding detergent; Production of an —. E. Möhring, Halle. Ger. Pat. 316,753, 21.2.18.

The product is prepared by compressing a mixture containing anhydrous alkali carbonate, sodium peroxide, and sufficient anhydrous magnesium chloride to neutralise the alkali hydroxide formed by the action of water on the peroxide. Other detergents may also be incorporated. In use, the magnesium salt interacts with the sodium peroxide, forming magnesium hydroxide and sodium chloride, whilst oxygen is liberated.—J. H. L.

Catalysts for use in hydrogenation processes; Production of —. W. P. Schueck. E.P. 122,192, 6.1.19. (Appl. 353/19.) Conv., 9.1.18.

SEE U.S.P. 1,305,173 of 1919; J., 1919, 589 A.

Lubricating composition. U.S.P. 1,336,207. See IIA.

Adhesives. E.P. 140,911. See XV.

Leather charcoal. G.P. 305,555. See XV.

Oil cakes. E.P. 128,216. See XIXA.

Bassia seeds. G.P. 318,413. See XIXA.

Fatty compounds. G.P. 318,222. See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Distilled colophony. L. Gerstacker. Farben-Zeit., 1920, 25, 1170—1171.

THE residue left in the partial dry distillation of colophony, known as "distilled" colophony, is used in the manufacture of brewers' pitch and varnishes. Products obtained up to 320° C. are viscous or hard, sticky masses, whilst those obtained at higher temperatures are all hard resins. The acid and saponification values are progressively lower than those of the original colophony. The distillation products are only partially soluble in absolute alcohol and acetone, but dissolve readily in ether, benzene, chloroform, and carbon tetrachloride. This partial insolubility in acetone interferes with the test for mineral oil in rosin oil by means of a mixture of alcohol and chloroform, but on allowing the liquid to stand any turbidity due to distilled colophony adheres to the sides of the vessel in drops, whereas mineral oil rises to the surface. When saponified with alcoholic potassium hydroxide distilled colophony gives a yellow turbid solution on dilution. Rosin oil in varnishes may be detected in the unsaponifiable portion, after removal of the rosin soap, by the Liebermann-Storch reaction (acetic anhydride and sulphuric acid), but the distinctive coloration is not given by the unsaponifiable matter of distilled colophony. When varnishes are distilled pinolin and rosin oils may be detected even in the lower fractions of the distillate (220°—280° C.), but if rosin oil only appears in the higher fractions (above 280° C.) the presence of distilled colophony is indicated.—C. A. M.

Colorimeter. Gamble. See XXIII.

PATENT.

Resinous substances. G.P. 310,171. See III.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Caoutchouc; Hot vulcanisation of —. F. Kirchhoff. Koll.-Zeits., 1920, 26, 168—173.

VULCANISATION of rubber may be effected by lead oxide or lead sulphide mixed with small quantities of sulphur and a small quantity of a sulphur-free organic catalyst.—J. F. S.

Rubber goods; Determining permanent set of —. E. L. Davies. India-rubber J., 1920, 59, 833—834.

THE average elongation at break is calculated from the results with ten sample pieces; ten other samples are then stretched to 60% of this elongation, and released after ten minutes, this operation being repeated thrice more with an interval of ten minutes each time for recovery before measurement and re-stretching. The permanent set is recorded after each of the four extensions, but the main effect is produced in the first.—D. F. T.

PATENT.

Recovering solvents. E.P. 141,210. See I.

XV.—LEATHER; BONE; HORN; GLUE.

Deliming [of limed pelt]; Velocity of —. G. Grasser. Collegium, 1919, 309, 3, 12. Chem. Zentr., 1920, 91, II., 351.

COMPARATIVE tests with 0.5% solutions of sulphuric acid, sulphuric acid plus sodium chloride, sulphuric acid plus sodium bisulphite, hydrochloric acid, ammonium chloride, hydrochloric acid plus sodium chloride, sodium bisulphate, lactic acid, acetic acid, sodium bisulphite, Neradol ND, molasses, and distilled water showed that sulphuric acid had the quickest deliming effect whilst the others followed in the order cited above. Further experiments with ammonium chloride, lactic acid, sulphuric acid plus sodium chloride, and sodium bisulphate on pieces of pelt limed in fresh limes and in limes sharpened with sodium sulphide or arsenic sulphide, showed that the rate of deliming is influenced less by the mode of liming than by the particular deliming agent employed.—D. W.

[Tannery] wetwork; Lyotropic influence and adsorption in the theory of —. H. G. Bennett. J. Soc. Leather Trades Chem., 1920, 4, 75—86.

THE view previously expressed (J., 1918, 313A, 314A) that the volume of gelatin and collagen gels is mainly determined by the net electric charge on the disperse phase resulting from ionic adsorptions and the lyotropic influence of dissolved substances in the continuous phase is further developed and the operation of these factors in the processes of soaking, liming, deliming, puering, and bating and drenching is discussed.

Sugar in tanning extracts and leather; Detection of —. G. Grasser. Collegium, 1919, 341—347. Chem. Zentr., 1920, 91, II., 352.

THE *o*-nitrophenylpropionic acid test for sugar was applied to various tanning extracts. A positive reaction was given by oak galls, knopperrn, gambier, and even by quebracho, although this contains very little sugar, and also by technical tannin. A commercial solid quebracho extract gave no reaction. The positive reaction given by quebracho and tannin was not caused by the splitting off of sugar from the tannin molecules, but by sugar actually

present as such. Catechol, pyrogallol, gallic acid, cellulose extract, formaldehyde, and synthetic tannins, such as Neradol D and ND, Ordoval G, failed to give the reaction. It is concluded that a positive reaction by tanning extracts with the reagent cannot be taken as an indication of adulteration with sugar unless there is additional evidence, and in the case of leather only when a distinctly deep blue colour is obtained with the reagent.—D. W.

Gelatin; Swelling of — in acid and alkaline solution. D. J. Lloyd. *Biochem. J.*, 1920, 14, 147—170.

THE properties of gelatin reacting as an amphoteric colloid are discussed. It is held that the structure of gels prepared from gelatin is probably that of a framework of neutral gelatin containing an interstitial fluid consisting of a solution of an ionisable gelatin salt. (*Cf. J.C.S.*, June.)—J. C. D.

PATENTS.

Tanned sponge. W. H. Allen, Detroit. U.S.P. 1,336,806, 13.4.20. Appl., 13.9.19.

SPONGE is tanned by treatment with a soluble chromium salt.—B. N.

Leather; Process for manufacturing waterproof, wear-resisting —. W. Reebberg, Hersfeld. G.P. 317,118, 3.11.18.

VEGETABLE-TANNED leather is treated with solutions of pyridine bases and impregnating materials insoluble in water, e.g. resin, asphalt, petroleum pitch, bitumen, or rubber dissolved in benzene. Leather tanned by other processes is impregnated in the same way after re-tanning with vegetable tanning materials.—D. W.

Tannin substitutes; Manufacture of —. C. Graf, Cologne. G.P. 317,462, 31.10.16.

GLAUBER'S salt, common salt, or Epsom salts are added to a mixture of sulphite-cellulose extract with an aqueous decoction of wood tar. Leather tanned with this mixture resembles that produced by a pure vegetable tannage, is plump, and has a bright colour with a dark cut.—D. W.

Leather charcoal; Preparation of high-grade — for hardening steel and iron, with simultaneous extraction of fat. M. Lindner, Dresden. G.P. 305,555, 14.4.17. Addition to 287,665 (J., 1916, 258.)

THE leather is subjected to a fat extraction process at 140°—280° C. and under 10—30 atm. pressure. By this means the leather is disintegrated and the extraction of the fat facilitated. The nitrogen content of the leather charcoal is considerably increased by this preliminary fat extraction.

—G. F. M.

Adhesives; Manufacture of —. M. O'Gorman and S. B. Schryver, London. E.P. 140,911, 17.4.19. (Appl. 9874/19.)

PROTEINS or alkali-proteins are isolated from the residues from the extraction or expression of oil from oil-seeds, especially hemp seed or castor beans, and treated with suitable bases or mixtures of bases, e.g., alkaline-earth hydroxides or magnesia, and with an alkali salt (sodium fluoride or arsenate), which will react slowly with the bases and form alkali hydroxide, and the resulting product is mixed with dilute alkali hydroxide solution.

—C. A. M.

Condensation product [tanning agent]; Soluble — and process of making it. O. Schmidt, Assignor to The Chemical Foundation, Inc. U.S.P. 1,336,759, 13.4.20. Appl., 4.12.13. Renewed 3.9.19.

SEE F.P. 462,635 of 1913; J., 1914, 326.

Leather substitutes. G. Miller. E.P. 141,385, 3.7.16. (Appl. 9364/16.)

XVI.—SOILS; FERTILISERS.

Soil acidity and alkalinity; Determination of — by indicators in the field. E. T. Wherry. J. Wash. Acad. Sci., 1920, 10, 217—223.

THE scale of values for the acidity and alkalinity of soil extracts (J., 1919, 548 A) is used in a method of determining the reaction of the soil in field tests. The following series of indicators is used: bromophenol blue, methyl red, bromocresol purple, bromothymol blue, phenol red, and phenolphthalein. About 1—2 g. of the soil is shaken with 5 c.c. of neutral salt-free water, the solid matter allowed to subside, and the clear supernatant liquid decanted and tested with the successive indicators until an intermediate colour is obtained with one indicator or opposing extremes with two overlapping indicators. The specific acidity or alkalinity in terms of p_H values may then be read from a table. (*Cf. J.C.S.*, June.)—C. A. M.

Lime requirement of a muck soil; Effect of aeration and other factors on the —. S. S. Walker. Soil Sci., 1920, 9, 77—81. (*Cf. Conner, J.*, 1919, 112 A).

THE lime requirement of a typical black muck soil was increased by air-drying, but the air-dried sample decreased in acidity during storage. When the soil was kept moist and frequently stirred for eight months the increase in acidity was decidedly less than when it was kept moist under anaerobic conditions. When the soil was neutralised with calcium carbonate and then kept moist for 8 months under either aerobic or anaerobic conditions it developed much more acidity than did the unneutralised soil under the same conditions.—W. G.

Soil; Reaction of the — as influenced by the decomposition of green manures. L. P. Howard. Soil Sci., 1920, 9, 27—39.

THERE is no indication that any acidity results from the use of rye as a cover crop during 25 years. Leguminous crops, however, during the same time considerably increased the lime requirements. On the other hand, from the treatment of uncropped soils in the laboratory with equal weights of green rye and clover it was found that the rye increased the lime requirement about twice as much as the clover.—W. G.

Chlorides in soil; Some factors influencing the quantitative determination of —. C. T. Hirst and J. E. Greaves. Soil Sci., 1920, 9, 41—51.

FOR the estimation of chlorides in soil 100 g. of the soil is shaken for 5 mins. with 500 c.c. of water, and the solution is clarified either with 2 g. of alum or by filtration through a Pasteur-Chamberland filter. Twenty c.c. portions of the clear filtrate are transferred to stoppered bottles and an excess of N/10 silver nitrate is added. After coagulation of the silver chloride by shaking in the presence of 10 c.c. of 95% alcohol, the excess silver nitrate is titrated with ammonium thiocyanate.—W. G.

Nitrifying [soil] bacteria; Isolation and study of —. W. M. Gibbs. Soil Sci., 1919, 8, 427—481.

BOTH *Nitrosomonas* and *Nitrobacter* develop readily on plates of washed agar or silicic acid gel, the latter medium being the more satisfactory for isolating the organisms. Pure cultures of either form of bacteria produce no visible growth when inoculated into bouillon. The enrichment process with both *Nitrosomonas* and *Nitrobacter* can be continued for an indefinite period of time without the slightest loss of activity of the organisms. Soil ex-

tracts used to prepare the nutrient solutions for the cultivation of *Nitrosomonas* and *Nitrobacter* did not prove toxic in either case. Sodium chloride in a concentration of 1.0% was very toxic towards *Nitrosomonas*.—W. G.

Molasses; Use of — as fertiliser in Mauritius. P. de Sornay. Bull. Assoc. Chim. Sucr., 1919, 37, 223—234.

THE application of molasses to the soil for cane cultivation has been very generally practised in Mauritius since before 1910. The results both here and in Reunion have been very satisfactory. The sugar disappears completely within about a fortnight. The beneficial effects of molasses fertilisation are most marked on plant canes, and diminish with successive ratoon crops. (Cf. Peck, J., 1910, 1467; Tempamy, J., 1911, 1463.)—J. H. L.

PATENTS.

Superphosphate manures; Apparatus for manufacture of —. H. Jones, and E. Newell and Co., Ltd., Misterton. E.P. 140,915, 3.5.19. (Appl. 11,019/19.)

HORIZONTAL, cylindrical superphosphate dens are arranged in pairs, and have each a longitudinal discharge opening in the lower portion of one side, provided with a door which hangs away from the opening when opened. The openings in two adjoining chambers are adjacent and above a common channel in which a conveyor for transporting the superphosphate operates. A rotary excavator is mounted on a carriage running on rails through the den, and the cutters of the excavator are provided with extensions leading back from the cutting edges to form sweeps or carriers. The cutters operate in a direction from the lower to the upper edge of the den opening.—W. J. W.

Calcium cyanamide; Means of emptying the furnace used in manufacture of crude —. P. Diemann, Piesteritz. G.P. 309,173, 14.2.18.

WHEN decomposition is complete, the filled, conical reaction chamber, while still red hot, is inverted so that the cover is at the bottom, and allowed to cool in this position, i.e., with the wider end downwards. By this means any burning on of the material on to the sheet-iron of the appliance is prevented.—W. J. W.

Caustic alkali liquors employed for treatment of straw, etc.; Utilisation of —. W. Colman, Charlottenburg. G.P. 316,147, 6.8.16.

THE black liquor, obtained in the treatment of straw with alkalis, is mixed with agricultural waste products and exposed to the atmosphere, by which means a stable fertiliser is produced. The caustic liquors remaining in the original material, together with the wash-waters, can be utilised for irrigation purposes.—W. J. W.

XVII.—SUGARS; STARCHES; GUMS.

Molasses; Determination of fine grain [sugar] in —. E. O. von Lippmann. Z. Ver. deut. Zuckerind., 1919, 44, 527. Chem. Zentr., 1920, 91, II., 498.

KALSHOVEN's method for determining the quantity of fine sugar crystals in molasses (J., 1920, 126 A) was applied to 15 final beet molasses; the results indicated that 5 contained between 1 and 5%, 7 between 5 and 10%, and 3 between 10 and 13% of sugar crystals.—J. H. L.

Fermentation; Spontaneous alcoholic — of concentrated sugar solutions [by Zygosaccharomyces]. J. Satava. Z. Zuckerind. Czechoslov., 1919, 44, 93—97.

THREE *Zygosaccharomyces* capable of fermenting

55—66% solutions of sucrose have been isolated. Organism K was found in a beet factory covering liquor ("Klärseil") of 66° Brix; its cells are elliptical or round; it does not liquefy gelatin; and in dilute solutions it ferments dextrose rapidly, and maltose and sucrose slowly. Organism S was obtained from beet factory syrup of 55° Brix; its cells are mostly oval or elongated and very unequal in size; it liquefies gelatin; and ferments dextrose rapidly, maltose slowly, and sucrose very slowly. Organism M was separated from several samples of marmalades; its cells are round; it liquefies gelatin; it ferments all the three sugars named vigorously in dilute solution; and it imparts a pleasant aroma and taste to the product, a property which may be utilised for improving the flavour of beet syrup. Pending further examination of these yeasts, they are regarded as variations of *Zygosaccharomyces Barkeri* (J., 1901, 918), which has also been found by the author to be capable of fermenting concentrated sucrose solutions.—J. P. O.

Caramel; Determination of the content of — as a means of estimating the sugar lost by superheating in the refinery. T. Koydl. Oesterr.-Ungar. Zeits. Zuckerind., 1918, 47, 16—39.

THE author proposes to estimate the amount of sugar lost during boiling by observing the increase in the colour of the product due to formation of caramel. Ehrlich's saccharan (J., 1910, 506), produced by heating sucrose to 220° C. *in vacuo*, is used as a standard, and the solutions are examined in a Stammer colorimeter. A solution of saccharan containing 10 mg. in 100 c.c. reads 2.5° Stammer; and in the production of 1 part of saccharan 4 parts of sugar is destroyed. On applying the method to the working of three massecuites obtained in the manufacture of white sugar, of raw sugar, and of after-product sugar, the amount of sugar destroyed was found to be 0.058, 0.037, and 0.833% of the dry substance respectively.—J. P. O.

Sugar factory waste water; Fermentation of — by yeast. A. Kraisy. Z. Ver. deut. Zuckerind., 1920, 163—176.

THE waste water, freed from pulp, is passed through a series of seven vessels each of about 20 cb. m. capacity. Each vessel is enclosed in a wooden casing, the top of which is provided with a sliding panel for regulating the rate of cooling. Each vessel is also provided with an air injector for agitating and aerating the contents. The first vessel is filled with waste water at 30°—36° C., and by regulating the rate of cooling, the water passes to the second vessel at 30° C. When the second vessel is one-third filled its contents are pitched with a yeast culture from a 1.5% solution of molasses containing lactic acid. The fermenting liquor passes through each succeeding vessel, 7—10 hours being required to pass through the whole series. When the whole series of vessels is in action, the second vessel, or at a later stage, the third vessel, is pitched with fermenting liquor from the seventh vessel at a rate of about 20 litres per min. The first, and subsequently the first and second vessels, serve for sedimentation of sand. The fermented liquor passes from the seventh vessel to settling tanks, and the sediment of yeast etc. is pumped to filter presses, the pressed yeast fodder being kiln-dried and ground. In the Einbeck factory 2500 kg. of yeast fodder is obtained weekly from 2500 cb. m. of waste water. The process effects a satisfactory purification of the waste water; practically all the sugar (about 0.23%) and about 90% of the total dissolved solid matter are removed. The yeast fodder contains rather a high proportion of ash (over 30%), but is richer in nitrogen than the pulp or dried slices; it is estimated to contain 5.8% of yeast.—L. E.

Melzitose; Occurrence of — in honey. C. S. Hudson and S. F. Sherwood. J. Amer. Chem. Soc., 1920, 42, 116—125.

A MANNA that forms on Douglas fir trees in British Columbia during summer droughts contains melzitose (J., 1918, 710 A), and under certain conditions this manna is collected by the bees and subsequently melzitose appears in the honey, sometimes in considerable amount. Melzitose has also been detected in other samples of honey. When a high proportion of melzitose is present in honey, it readily separates in the crystalline state; otherwise the readiest method of isolating the trisaccharide depends on fermenting away the other sugars with baker's yeast.—H. W.

Celloisobiose. H. Ost and R. Prosiegel. Z. angew. Chem., 1920, 33, 100.

A NEW biose, celloisobiose, $C_{12}H_{22}O_{11}$, has been isolated from the product of acetylation of cellulose in presence of considerable quantities of sulphuric acid at 15°—30° C. It forms fine needles with a slightly sweet taste which melt indefinitely at 165°—190° C. with evolution of gas; $[\alpha] = +23^\circ$ in aqueous solution. Its reducing power is 99% of that of maltose. It is not fermented by top-fermentation yeast, but readily yields dextrose when treated with hydrochloric acid. (Cf. J.C.S., i., 423.)—J. K.

Starch; Supposed fission of — by formaldehyde. M. Jacoby, W. von Kaufmann, A. Lewite, and H. Sallinger. Ber., 1920, 53, 681—685.

A REPLY to Woker's recent criticisms (J., 1920, 77 A). The simple explanation of the supposed diastatic action of formaldehyde lies in the fact that the latter forms a loose addition compound with starch (which does not give a blue coloration with iodine), and also modifies the physical properties of the colloid; precipitation with alcohol, or with numerous electrolytes which behave similarly, yields unchanged starch which under no conditions has suffered diastatic degradation.—H. W.

Molasses as fertiliser. De Sornay. See XVI.

PATENTS.

Centrifugal machine [for sugar]. A. H. Gibson, Salt Lake City. U.S.P. 1,336,779, 13.4.20. Appl., 21.4.15. Renewed 25.6.19.

A CENTRIFUGAL basket is carried by a vertical rotating shaft, and is provided with a bottom movable vertically, which is normally closed. Means are provided above the basket to raise and lower the bottom by the differential rotation of the basket and the said means. The bottom carries scrapers projecting upwards into the basket which engage and remove the deposit of sugar on the side walls when the bottom is lowered.—W. F. F.

Beet juices; Process for the clarification of —. E. Psenicka. G.P. 318,651, 19.3.18. Conv., 30.4.17.

RAW beet juice is mixed with sufficient saturation scum to impart a neutral or only slightly acid or alkaline reaction, and the mixture heated, a flocculent precipitate thus forming which can readily be filtered off. Subsequently the juice is treated with the necessary amount of lime, and carbonated as usual. Advantages claimed are that the separation of proteins and other non-sugars is more complete, and the amount of incrustation in the heaters and evaporators is less, than in the ordinary procedure. (Cf. J., 1919, 592 A.)—J. P. O.

Sugar cane and like crushing mills. J. Miller, and G. Fletcher and Co., Ltd. E.P. 111,078, 8.8.16. (Appl. 11,167/16.)

XVIII.—FERMENTATION INDUSTRIES.

Wine; Influence of various substances on the saturation of — with calcium sulphate. A. Bornträger. Staz. Sperim. Agrar. Ital., 1919, 52, 319—360. Chem. Zentr., 1920, 91, II., 455.

THE amount of calcium sulphate dissolved varies with the quantity of marc present, but the influence of the stalks alone is quite small. Addition of 1% of calcium sulphate was always found sufficient for saturation. The amount dissolved increases with rise in temperature to a certain degree. Saturation is attained in a short time by frequent stirring. The alcohol formed during fermentation causes the separation of a part of the dissolved calcium sulphate.—J. H. L.

Formic acid; Production of — by yeasts in media containing amides. P. Thomas. Ann. Inst. Pasteur, 1920, 34, 162—176.

THE volatile acidity produced during the culture of yeast on media containing acetamide (J., 1920, 77 A) is due partly to formic acid and partly to acetic acid formed during the fermentation. The total volatile acidity and the relative proportions of the two acids vary with the conditions. (Cf. J.C.S., June.)—W. G.

Acetone and n-butyl alcohol; Intermediate substances produced in fermentation method for preparation of —. J. Reilly, W. J. Hickinbottom, F. R. Henley, and A. C. Thaysen. Biochem. J., 1920, 14, 229—251.

THE chief intermediate products appear to be acetic and butyric acids. The possible mechanism of the formation of acetone and n-butyl alcohol during this fermentation process is discussed. Lactic acid may be produced in an infected mash. (Cf. J.C.S., June.)—J. C. D.

Soya bean urcase. A. W. Dox. Amer. J. Pharm., 1920, 92, 153—157.

THE urcase activity of soya beans of different varieties and crops was determined by mixing 1 g. of the powdered seed with 15 c.c. of water, and 10 c.c. of 1% urea solution, and titrating the liberated ammonia after 30 mins. at 40° C. The amounts of N/10 hydrochloric acid required by 37 samples ranged from 11.1 to 20.2 c.c. The differences in the urcase activity shown by different varieties of the beans did not stand in any relationship to the protein content or germinating power of the seed.—C. A. M.

Alcoholometric tables applicable to alcohol denatured with wood spirit and naphtha. O. Chénéau. Bull. Assoc. Chim. Sucr., 1919, 37, 213—222.

Fermentation of concentrated sugar solutions. Satava. See XVII.

Sugar factory waste water. Kraisy. See XVII.

Starch and formaldehyde. Jacoby and others. See XVII.

Iron in grape must. Mathieu. See XXIII.

PATENTS.

Distillery mashes; Acidification of —. H. Kaserer. G.P. 317,398, 30.10.17. Conv., 2.11.16.

SULPHITE-CELLULOSE waste liquors are employed for acidification, and fermentation is conducted as usual.—J. H. L.

Fat from mould-fungi. G.P. 306,365. See XII.

XIXA.—FOODS.

Baking properties of flours. T. von Fellenberg. Mitt. Lebensmittellunters. u. Hyg., 1919, 10, 229—260. Chem. Zentr., 1920, 91, 11., 412—413.

A STUDY was made of the baking properties of artificial flour mixtures made up of 10% of a protein (gliadin, glutenin, egg albumin, casein, or gelatin), 86% of starch (from wheat, rye, maize, rice, or potatoes), and the balance of sugar and mineral salts. In working them into dough 1.5% of sodium chloride and 2% of yeast were added. The dough was allowed to ferment for 3 hours at 32° C. and then baked at 130°—135° C. Comparative experiments were also made on commercial flours, and the effect of baking powders was investigated. The following conclusions are drawn:—The baking property of flour is chiefly due to gliadin, which alone determines the increase in volume. Differences in the gliadin-content account for the superiority of wheat flour over rye flour and of this over barley flour. Substitution of egg albumin or gelatin for gliadin gives fluid doughs which cannot be kneaded; with egg-albumin the dough rises well, but not with gelatin. Casein gives a dense bread, and with pectin the dough does not rise. The baking property of flour is also affected by the kind of starch present, wheat starch being the best. Addition of lecithin increases the volume of the loaf, but soluble polysaccharides impair the baking properties. Two stages may be distinguished in bread-making, *viz.*, the fermentation of the dough and the baking process proper. The larger the proportion of bran in a flour, the higher will as a rule be the acidity, the protein content, and the proteolytic activity of the flour; with increase in these factors the gluten becomes more fluid during fermentation of the dough, the duration of fermentation is lessened, and the volume of the loaf increased. In the case of bread made with the aid of baking powders, without fermentation of the dough, the volume varies directly with the elasticity and extensibility of the gluten and the capacity of the flour for retaining water, and inversely with the stiffness of the dough. It depends upon the content of proteins, especially gliadin, and is unfavourably affected by soluble polysaccharides. The capacity of the flour for retaining water depends on the presence of substances which dissolve and swell in water, *viz.*, proteins, soluble carbohydrates, husk constituents, and mineral salts.—J. H. L.

Potatoes; Melanin-value of —. H. Haehn. Z. Spiritusind., 1920, 43, 90—91, 104, 111—112.

THE rapid darkening which occurs in the juice of sliced or pulped potatoes is mainly due to the formation of melanin by the action of tyrosinase on tyrosine. A summary of the literature of the subject is given. The darkening is much more pronounced with some kinds of potatoes than with others. It may be expressed quantitatively by the melanin-number determined as follows:—5lb. of potatoes is washed, peeled, and pulped, and the juice is separated by a hand-press and freed from suspended matter and starch granules by centrifuging. About 1 hour after pulping the juice is slightly acidified with acetic acid, heated to boiling, and filtered from coagulated proteins. Ten c.c. is diluted to 100 c.c., and 10 c.c. of the diluted juice is treated with 1 c.c. of 10% sulphuric acid and oxidised in the warm with N/500 permanganate solution until decolorised. The number of c.c. of the permanganate solution required to decolorise 10 c.c. of the diluted juice is the melanin-number of the fresh juice. If the fresh juice is treated with 0.3% of sodium fluoride and left to stand for 24 hours the proportion of amino-acid nitrogen present increases owing to autolysis, and the melanin-number is increased.

The difference between the melanin-numbers before and after this 24 hours' autolysis is designated the autolytic value. For 16 different strains of potatoes tested the melanin-numbers of the fresh juices ranged from 5.8 to 17.5, and of the autolysed juices from 8.7 to 21.3, the autolytic values ranging from 1.6 to 13.1. After storage for 6 months potatoes show much higher autolytic values; the proportion of amino-acids increases during storage, whilst that of tyrosinase diminishes. If the fresh juice is rendered slightly alkaline before the 24 hours' autolysis, the melanin-number found after autolysis is appreciably higher than when the reaction of the juice remains acid, especially in the case of potatoes which have been stored.—J. H. L.

Foodstuffs; Estimation of water in —. K. Scheringa. Pharm. Weekblad, 1920, 57, 398—403.

TESTS made with meal (potato, wheat, and bone), milk powder, apples, carrots, jam, bread, cheese, etc., showed, in certain cases, slightly higher results when the water was determined by drying at 110° or 120° C. than when it was determined by distillation with xylol (b.p. up to 140° C.).—W. J. W.

Vitamine; Water-soluble — in milk. T. B. Osborne and L. B. Mendel. J. Biol. Chem., 1920, 41, 515—523.

THE authors claim that larger quantities of milk (16 c.c. per day) are necessary to supply the vitamins for the growth of rats than was found by Hopkins (J. Physiol., 1912, 44, 425).—J. C. D.

Vitamine; Fat-soluble — in plant tissues. T. B. Osborne and L. B. Mendel. J. Biol. Chem., 1920, 41, 549—565.

THE value of certain dried vegetables as sources of fat-soluble A is as high as that of butter-fat. Thus normal growth in rats may be obtained when the A vitamin is supplied in the form of 0.1 g. of dried tomato daily. The liability of fat-soluble A to destruction by heat reported by Steenbock and others (J., 1918, 712 A) and Drummond (J., 1919, 435 A) is not confirmed.—J. C. D.

Fruit juices; Antiscorbutic properties of concentrated —. A. Harden and R. Robison. Biochem. J., 1920, 14, 171—177.

ORANGE juice may be concentrated to dryness by a vacuum process at low temperature without any appreciable reduction in its antiscorbutic property. This method might be employed commercially for the preparation of a stable product containing a high concentration of the antiscorbutic vitamin. —J. C. D.

Vegetable and fruit juices; Effect of heat on antiscorbutic value of —. E. M. Delf. Biochem. J., 1920, 14, 211—228.

THE destruction of the antiscorbutic accessory factor by heat is not so rapid as was previously believed. Autoclaving the juice of cabbages, swedes, and oranges at temperatures up to 130° C. causes a corresponding loss of the vitamin, but in the case of orange juice it is not serious. The stability of the antiscorbutic factor in orange juice is remarkable, and does not appear to be entirely due to the presence of a high percentage of free acid. It is probable that destruction of the antiscorbutic property of fruit and vegetable juices by heat is much less when air or oxygen is absent. These observations have an important bearing upon the processes employed in the preservation of fruit and vegetables.—J. C. D.

Vegetables; Losses incurred in cooking of —. II. Green vegetables. H. Masters and P. Garbutt. Biochem. J., 1920, 14, 75—90.

THE addition of a small quantity of alkali to the

water employed in cooking green vegetables has not only a marked effect upon the colour, but also decreases both the time required for cooking and the losses incurred. No such effects were observed when salt was added. The addition of a small amount of ammonium carbonate to the water employed for steaming vegetables has a beneficial influence on the colour.—J. C. D.

Caffeine in mixtures of coffee and coffee substitutes and in caffeine-free coffee; Determination of —. E. Vautier. Mitt. Lebensmittelunters. u. Hyg., 1919, 10, 273–277. Chem. Zentr., 1920, 91, II., 414.

THE method previously described (J., 1918, 712 A) is not applicable when the amount of caffeine present is very small. In such cases it is advisable to decompose the crude caffeine obtained by ethereal extraction, by Kjeldahl's method (1 c.c. of N/10 acid=0.00485 g. of anhydrous or 0.00530 g. of hydrated caffeine). If necessary the crude caffeine may first be partially or completely purified, e.g., after removal of fats (*loc. cit.*) its aqueous solution is treated with 0.1–0.2 g. of sodium carbonate to fix the humic acids, and after evaporation and complete drying the caffeine is extracted in the pure state by chloroform.—J. H. L.

Sugar factory waste water. Kraisy. See XVII.

Precipitant for proteins. Marie. See XXIII.

Amino-acids. Tague. See XXIII.

Crude fibre. Clemens. See XXIII.

PATENTS.

Oil cakes and the like; Process and apparatus for adapting — for human food. K. Erslev, Nijmegen. E.P. 128,216, 11.6.19. (Appl. 14,729/19.) Conv., 1.8.17.

FINELY-GROUND, dried oil cake or the like is extracted with a fat solvent, the residue is ground, dried and extracted with absolute alcohol. The solvents are recovered for further use by distillation, and fats, carbohydrates, lecithin, etc., may be recovered from the extracts. The extractions are carried out by treating the material with a counter-current of the solvent, and two types of suitable apparatus are described.—L. A. C.

Cellulosic material; Transformation of — into easily digestible fodder. F. Lehmann, Göttingen. G.P. 307,616, 16.7.16.

AFTER the material has been decomposed by heating with alkalis, a part of the cellulose is oxidised to acids for the purpose of neutralising the alkali. Oxidation may be effected by means of air or oxidising agents such as sodium peroxide or potassium nitrate, and oxygen-carriers such as iron or manganese salts may be used. Palatable fodder is thus produced even when 8–10% of soda is used.—J. H. L.

Cellulosic plant materials; Production of foodstuffs or fodder from —. P. Strahl, Berlin. G.P. 317,111, 20.7.17.

AFTER the material, e.g., straw, husks, etc., has been decomposed by alkali hydroxides, alkaline earths, or mixtures thereof, the alkali is neutralised by addition of a vegetable or amylaceous material or juice which has been subjected to lactic fermentation.—J. H. L.

Milk; Preventing the souring of —. R. Marcus, Frankfort. G.P. 317,874, 13.4.17.

MILK is treated with pure, sterilised, inert colloids, such as silicic acid (5–10%), clay or alumina, in the form of powder or lumps.—J. H. L.

Bassia seeds; Manufacture of edible products from — and especially from compressed residues of the same. Halstein-Oelwerke G.m.b.H., Altona. G.P. 318,413, 26.9.16. Addition to 250,144 (U.S.P. 1,030,674, and E.P. 23,595 of 1911; J., 1912, 697, 1092).

THE comminuted seeds or pressed cake, mixed with water and slightly acidified, are heated, if necessary under pressure, to decompose and precipitate the sapotoxins and bitter substances and saccharify the starch. The material is then dried and extracted, first with dilute cold alcohol and then with strong warm alcohol. After the heating the liquid may be separated from the solid residue, and converted into syrup, caramel, or coffee substitute, whilst the residue, extracted as described above, can be used as fodder. The process can be applied also to horse-chestnuts, acorns, beans, etc.—J. H. L.

XIXB.—WATER PURIFICATION; SANITATION.

Boiler-feed water; Substitutes for sodium carbonate for softening —. C. Braungard. Chem.-Zeit., 1920, 44, 334–335.

THE procedure to be adopted when using sodium or potassium hydroxide, potassium carbonate, or barium hydroxide as substitutes for sodium carbonate in the treatment of various types of boiler-feed water is described.—C. A. M.

Clay suspensions and silicic acid; Coagulation of —. O. M. Smith. J. Amer. Chem. Soc., 1920, 42, 460–472.

MANY natural surface waters in the Mississippi valley contain colloidal clay and silicic acid, and experiments were made on the precipitation of such colloidal solutions and suspensions. Silicic acid is not precipitated, from a solution containing 184 pts. SiO_2 per million, by 0.07N concentration of various alkali salts, magnesium bicarbonate and sulphate, barium, calcium, ferric and aluminium chlorides, aluminium, ferrous and ammonium sulphates, but is precipitated by sodium hydroxide 0.099N, calcium hydroxide and barium hydroxide 0.0019N, and colloidal iron 0.0045N. For the precipitation of silicic acid by alkali in the presence of aluminium salts the optimum hydrogen ion concentration is 1×10^{-4} . If the alkali is added all at once, the silicic acid prevents the precipitation of aluminium hydroxide, apparently by acting as a protective colloid. The presence of divalent cations destroys this protective power. For the coagulation of clay suspensions, the order of efficiency of the electrolytes examined was: aluminium sulphate, calcium and barium hydroxides, calcium chloride, magnesium sulphate, magnesium and calcium bicarbonates. The aluminium cation is 5 times as effective as the calcium or barium ion. Sodium hydroxide, carbonate, and sulphate at first retard coagulation when aluminium sulphate is added to a clay suspension. As the quantity of aluminium sulphate is increased, there is at first increased dispersion, then coagulation, followed by renewed dispersion and a final coagulation. The presence of silicic acid has in all cases, whatever salts may be present, a retarding influence on the coagulation of clay suspensions.—E. H. R.

Sewage purified by the activated sludge process; Action of the bacteria of — on proteins, urea, and nitrates. P. Courmont and A. Rochemaix. Comptes rend., 1920, 470, 967–970.

THE proteolytic properties of the seven species of bacteria found in the effluent from sewage purified by the activated sludge process are either absent or very reduced. Only with *B. subtilis* was any positive result obtained. Three out of the seven species ferment urea; *B. subtilis* exerts a slight action, and

the other three are without action on urea. Five out of the seven species are direct denitrifying agents as shown by their action on potassium nitrate.—W. G.

[*Sewage*] *sludges; Action of activated*. — F. Diéner, F. Wandenbulke, and M. Launey. Comptes rend., 1920, 170, 1089–1092.

By varying the amount of activated sludge used it was found that the ratio of ammonia destroyed to the dry weight of sludge introduced diminishes as the amount of sludge used increases. This process of purification rapidly yields an effluent free from ammonia and putrefactive matter, but the reduction of the number of germs is apparently very variable.—W. G.

Sterilising power of acids. E. Aubel. Comptes rend., 1920, 170, 970–972.

A comparison of the action of certain organic and inorganic acids on the pyrocyanic bacillus shows no preponderating influence of the cations. The nature of the acids seems to play an important part. The organic acids used (formic, acetic, oxalic, tartaric, lactic) show increased toxicity with decrease in molecular weight and were more active than the inorganic acids (sulphuric, phosphoric, hydrochloric, nitric). Sulphuric acid apparently has some specific action.—W. G.

Carbon monoxide; Removal of — from air. A. B. Lamb, W. C. Bray, and J. C. W. Frazer. J. Ind. Eng. Chem., 1920, 12, 213–221.

Of many substances (ozone, palladium, mercuric oxide and chromic acid, silver oxide, silver permanganate, copper oxide and cobalt oxide, etc.) examined as absorbents or catalytic oxidisers of carbon monoxide, the best consisted of a mixture of manganese dioxide, 50, copper oxide, 30, cobalt oxide (Co_2O_3), 15, and silver oxide, 5%; this mixture is known as "Hopcalite I." The manganese dioxide was prepared by the action of manganese sulphate on potassium permanganate in sulphuric acid solution; the silver oxide was precipitated in the mixed sludge of the other oxides. After washing, the whole mixture was collected on a filter, kneaded, dried, and ground. The granular mixture thus obtained removes more than 90% of carbon monoxide from a 0.25% carbon monoxide-air mixture at ordinary temperature; it appears to act for an indefinite period when the air is dry. For practical use in gas-mask canisters it is necessary to provide a preliminary calcium chloride drying chamber; the "life" of the absorbent then depends on that of the drier.—W. P. S.

Charcoal; Behaviour of — with respect to chlorine. G. S. Bohart and E. Q. Adams. J. Amer. Chem. Soc., 1920, 42, 523–544.

Two processes take place during the passage of chlorine through charcoal, viz., absorption or adsorption, which cannot be distinguished, and catalytic formation of hydrochloric acid. According to the conditions, chlorine or hydrochloric acid may first appear in the emerging gas when the charcoal has become saturated; the period during which the transmitted gases are free from either is called the service time of the charcoal. The service times of different charcoals could not be correlated with their apparent densities. For any one charcoal the service time increases more rapidly than the depth of the absorbing layer. Charcoals with the greatest chlorine capacity have the greatest catalytic activity for the formation of hydrochloric acid. The proportion of hydrochloric acid formed increases with increasing moisture content of the gases, but the service time of the charcoal passes through a minimum, in the case of a gas stream containing air and chlorine in the ratio 500:1, when the gases are 50% saturated. Below 12.5° C. chlorine appears

first in the transmitted gases; above this temperature hydrochloric acid. The service time is at a minimum between 0° and 12.5° C. Reduction of pressure has little or no effect. Exposure to chlorine followed by heating to redness increases the chlorine capacity of charcoal.—E. H. R.

Chloropicrin and carbon tetrachloride; Velocity of adsorption of — by charcoal. H. S. Harned. J. Amer. Chem. Soc., 1920, 42, 372–391.

The velocity of adsorption was measured by exposing a known weight of charcoal to the vapour, at low pressure, for a definite time, and determining the amount adsorbed by the increase in weight of the charcoal. To obtain reproducible results it was necessary to heat the charcoal in a vacuum to 700° C., cool in a vacuum, expose to the vapour under examination and again heat to 700° C. in a high vacuum. With charcoal thus treated the adsorption velocity is a maximum, and the results are expressed by the equation $m = 1/t \log A/(A - K)$, where K is the amount adsorbed per gram of charcoal in time t , A is the amount adsorbed when t is infinitely long, and m is a constant.—E. H. R.

Mustard gas; Minimum effective concentration of —. C. I. Reed. J. Pharm. Exp. Ther., 1920, 15, 77–80.

MUSTARD gas at a concentration of 0.0005 mg. per litre will produce definite reaction in the human eye in less than an hour's exposure.—J. C. D.

Sugar factory waste water. Kraisy. See XVII.

PATENTS.

Water; Treatment of — after lime purification. A. Schreier. Vienna. G.P. 317,720, 18.7.18. Conv., 22.5.18.

The water is subjected to the action of cold or warm air or other gases containing carbon dioxide to eliminate excess of lime, and is then treated with base-exchanging substances.—W. J. W.

Water; Preparation of a purifying agent for —. Permutit-A.-G., Berlin. G.P. 318,145, 4.7.13.

Porous organic or inorganic substances, such as pumice, lava, broken porcelain, clinker, coke, charcoal, wood wool, sawdust, cork, or corkfibre are saturated with an alkaline silicate, then treated with hydrochloric acid, and finally washed with an alkaline solution. Colloidal silicic acid is by this means precipitated.—W. J. W.

Separating liquid from solid matter [sewage sludge]; Apparatus for —. F. J. Schwable, Assignor to French Oil Mill Machinery Co., Piqua. U.S.P. 1,308,918, 8.7.19. Appl., 16.7.17.

The sludge is pumped into upright separating bags of pervious material suspended side by side in a frame with fixed end walls. Between the separating bags are compression bags which may be expanded by pneumatic or hydraulic pressure, thus compressing the separating bags and forcing out liquid from the sludge. The bottoms of the separating bags are normally closed but may be opened to discharge the solid residue.

Lime and sulphur product; Dry — and method of making the same. O. F. Hedenburg, Pittsburgh, Assignor to F. O. Mohurg, Toledo. U.S.P. 1,336,957, 13.4.20. Appl., 20.11.18.

A SOLUTION of lime and sulphur is evaporated to a point at which the concentrated product will solidify on cooling.—W. J. W.

Preserving matter; Process for —. F. S. Benenati. E.P. 127,559, 5.4.19. (Appl. 8637/19.) Conv., 31.5.18.

SEE U.S.P. 1,292,401 of 1919; J., 1919, 335 A.

Absorbent for carbon dioxide. G.P. 303,261. See VII.

Carbon monoxide. G.P. 307,614. See XXIII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Morphine; Determination of the alkaloids of the pharmacopœia with special reference to the estimation of — in opium and its preparations. Rapp. Apoth.-Zeit., 1920, 35, 17–20. Chem. Zentr., 1920, 91, 11., 392.

The method previously described (J., 1919, 336 A) is applicable to the determination of morphine in opium etc., and especially to the determination of small quantities of morphine alone or in presence of narcotine if the latter be first removed by extracting an acetic acid solution with chloroform.

—G. F. M.

Pituitary gland; Pressor compound in —. A. C. Crawford. J. Pharm. Exp. Ther., 1920, 15, 81–91.

By treating extracts of the posterior lobe of the pituitary with mercuric chloride a substance is precipitated which possesses powerful pressor effect. The product appears to be pure, but no crystalline salts or derivatives have been obtained. The yield was very small.—J. C. D.

p-Ureidophenylacetylurea and related compounds; Preparation of —. M. Rising. J. Amer. Chem. Soc., 1920, 42, 128–136.

The preparation of *p*-ureidophenylacetylurea, the first of a series of ureido-phenylacetyl derivatives of urea which are to be investigated with regard to physiological activity, particularly as hypnotics, is described. *p*-Nitrophenylacetyl chloride is condensed with urea in boiling benzene solution to yield *p*-nitrophenylacetylurea; the latter is quantitatively reduced by a solution of stannous chloride in cold glacial acetic acid to *p*-aminophenylacetylurea, which decomposes without melting at 192°–193° C. When the hydrochloride of the latter is treated with potassium cyanate *p*-ureidophenylacetylurea, $\text{NH}_2\text{CO.NH.C}_6\text{H}_4\text{CH}_2\text{CO.NH.CO.NH}_2$, is produced. (Cf. J.C.S., 1., 387.)—H. W.

Cyanamide and its derivatives; Action of chlorine, of hypochlorous acid, and of cyanogen chloride on —. C. Mauguin and L. J. Simon. Comptes rend., 1920, 170, 998–1001.

CYANAMIDE undergoes marked polymerisation when chlorine is passed over it. When the gas is passed into an aqueous solution of the amide or its calcium salt an extremely unstable, lachrymatory product results. When cyanamide, in aqueous solution is added gradually to an aqueous solution of hypochlorous acid free from chlorine and the mixture cooled to 0° C., a very unstable crystalline product, dangerous to handle, separates out. When perfectly dry silver cyanamide is added to an excess of cyanogen chloride at 0° C., a silver compound, AgN(CN)_2 , is obtained as a white powder, which is decomposed by hydrogen chloride in ethereal solution giving dicyanoimide hydrochloride, and in water by dilute hydrochloric acid to give cyanourea.—W. G.

Benzoic acid; Purification of — by fractional condensation. M. Phillips and H. D. Gibbs. J. Ind. Eng. Chem., 1920, 12, 277–279.

Crude benzoic acid is vaporised in a cylindrical iron vessel, the vapour escaping at the top through a 1 in. iron pipe, which extends through two sheets of asbestos and a thick glass plate placed horizontally just above the cylinder into a circular wire-gauze (½ in. mesh) chamber resting on the glass plate; other wire gauze chambers are fitted concentrically round the first and the series is covered with

a second glass plate. The cylinder is heated by an oil bath, and the chambers by a current of hot air. When the temperature inside the first wire-gauze chamber is about 60° C., and that of the oil-bath 120° C. the air inflow into the chambers is discontinued and air is passed into the top of the cylinder through a copper coil in the oil bath. The benzoic acid sublimes and condenses on the gauze screens. There is a sharp drop in temperature as the gases pass through the screens and very pure benzoic acid may be obtained in one or other of the chambers.

—W. P. S.

Chlorobenzenes; Analysis by distillation of industrial —. F. Bourion. Comptes rend., 1920, 170, 933–935.

From 1500 to 1800 g. of the crude chlorobenzene is divided by distillation into three fractions of b.p. 80°–130° C., 130°–131.5° C., and 131.5°–172° C. respectively. The first fraction (benzene plus monochlorobenzene) is further fractionated, using a Vigreux column, into six fractions, the b.p. and benzene content of which are as follows: b.p. 81°–90° C., 98% C_6H_6 ; 90°–100° C., 90%; 100°–110° C., 50%; 110°–120° C., 20%; 120°–128° C., 10%; 128°–130° C., 2% C_6H_6 . The portion b.p. 131.5°–172° C. (monochlorobenzene and polychlorobenzenes) is also fractionated, giving the following fraction: b.p., 131.5°–134° C., 98% $\text{C}_6\text{H}_5\text{Cl}$; 134°–140° C., 90%; 140°–150° C., 60%; 150°–160° C., 10%; 160°–165° C., 2% $\text{C}_6\text{H}_2\text{Cl}_2$. The fraction b.p., 130°–131.5° C. is not pure monochlorobenzene, and an aliquot portion of it is redistilled to determine its content of polychlorobenzenes.—W. G.

Chlorobenzenes; Physico-chemical method of analysis of industrial —. F. Bourion. Comptes rend., 1920, 170, 990–993.

A SIMPLER, and much more rapid, though somewhat less exact, process than the method previously described (preceding abstract) is to divide the known weight of the chlorobenzene into two fractions, the first distilling below 130° C. consisting of benzene and chlorobenzene, the second not passing over at 130° C. consisting of chlorobenzene and *p*-dichlorobenzene. The densities of these two fractions are determined, and from these results and the known densities of benzene, chlorobenzene, and *p*-dichlorobenzene, the relative amounts of the two ingredients in each fraction can be calculated and hence the purity of the original sample determined. The method gives results for benzene accurate to 5% of the actual amount present, but for the polychlorobenzenes, if present, the result is only accurate to 6–24% of the amount actually present.

—W. G.

Acrolein; New reaction of —. L. Tsalapatanis. Anal. Soc. Quim. Argentina, 1917, 5, 241–245.

THE liquid to be tested is heated with 1–2 c.c. of an aqueous solution of resorcinol and a few drops of 10% caustic soda solution. After 2 mins. a bluish-green coloration appears in dilute, and a red one in concentrated, solutions. The reaction is extremely sensitive. The colour, which is very stable, is destroyed by acids, but restored by caustic soda.

—W. R. S.

Fatty acids; Catalytic decomposition of — by carbon. J. B. Senderens and J. Aboulenc. Comptes rend., 1920, 170, 1061–1067.

ACETIC acid and its homologues are decomposed when passed over animal charcoal at 360°–380° C., giving carbon dioxide, ethylenic hydrocarbons, carbon monoxide, paraffin hydrocarbons, and hydrogen, together with water and small amounts of ketones and aldehydes. With carbon from sugar as the catalyst a considerably higher temperature is necessary, but the products are similar.—W. G.

Distillation apparatus for separation of water [from organic solvents]. G. W. Walker. Chem. News, 1920, 120, 222—223.

WATER may be removed from liquids such as chloroform, benzene, etc., by distillation from a flask provided with two side tubes on its neck. The liquid is boiled until all the water has been expelled through the lower of these side tubes; this tube is then sealed, and the remainder of the dry liquid distilled through the upper side tube, which is connected to a condenser.—W. P. S.

Catalytic oxidation. Weiss and Downs. See III.

Cholesterol. Csonka. See XII.

Adsorption of chloropicrin and carbon tetrachloride. Harned. See XIXb.

Indicators and their application. Lubs. See XXIII.

Amino-acids. Tague. See XXIII.

PATENTS.

Hyoscyamine; Isolation of —. Chem. Fabr., vorm. Sandoz. E.P. 131,283, 28.7.19. (Appl. 18,686/19.) Conv., 15.8.18.

The powdered leaves, seeds, or roots of *Atropa belladonna*, L., *Hyoscyamus niger*, L., *Datura stramonium*, L., or other solanaceous material, are treated with a dilute solution of a mineral or organic acid, or an acid salt such as aluminium, ferrous, or copper sulphate, or ferric chloride, and the acid mixture is extracted with a fat solvent such as ether, benzene, or the like, containing from 1 to 7% of alcohol. The residue is suspended in a further quantity of a fat solvent and made alkaline by the addition of ammonia or other alkali; after separation of the insoluble matter the extract is concentrated under reduced pressure. Hyoscyamine separates from the solution on standing as long white needles of m. pt. 107°—108° C., $[\alpha]_D = -20.5^\circ$ to -21.3° .—L. A. C.

Glyoxal; Preparation of —. E. C. R. Marks. From A. Bosshard, Zurich. E.P. 140,478, 11.10.17. (Appl. 14,731/17.)

GLYOXAL is formed by the action of ozone on acetylene when the reaction occurs in the presence of a large excess of oxygen, air, or other indifferent gas. Suitable proportions are 1—2% of ozone, 3—4% of acetylene, and 94—96% of air. The process is facilitated by the presence of water vapour or a fine spray of water, whereby the glyoxal is washed out of the gaseous mixture and separates as a concentrated solution.—G. F. M.

Denatured alcohol; Production of —. P. V. H. Pascal, Lille. E.P. 140,527, 3.1.19. (Appl. 224/19.) Conv., 16.1.18. Addition to 140,115.

IN the manufacture of alcohol by the electrolytic hydrogenation of aldehyde or its polymers in an acid bath (J., 1920, 385A), the temperature of the bath is raised slowly to 70°—80° C., whereby reduction and polymerisation products of crotonic aldehyde are formed, which act as denaturants.—L. A. C.

Pyrogallol acid; Preparation of —. E. C. R. Marks. From Nitritfabrik A.-G., Koepenick. E.P. 140,694, 29.8.19. (Appl. 21,281/19.)

PYROGALLOL is obtained in nearly theoretical yield by heating solutions or suspensions of tannin or gallic acid, at temperatures preferably above 160° C., in an autoclave, with a proportion of alkali, alkaline-earth, etc., carbonates or hydroxides not materially exceeding twice the amount required

to effect substitution of the hydrogen atoms of the carboxyl groups initially present, and those formed by hydrolysis (if any).—G. F. M.

Chlorhydrins; Manufacture of —. R. Haddan. From The Commercial Research Co., Long Island City. E.P. 140,831, 27.2.17. (Appl. 2916/17.)

STEAM and oil gas are blown into the anode chamber of a cell for the electrolytic decomposition of sodium chloride solution; the electrolyte is maintained at 95°—100° C., escaping gases from the anode chamber are condensed and the dilute hydrochloric acid obtained is returned to the cell, thereby lowering the voltage necessary for operation. Alternatively an olefine may be mixed with the sodium chloride solution in the anode chamber by agitation, portions of the electrolyte then being removed from time to time from the cell, and the chlorhydrin separated by steam distillation.—L. A. C.

Alkamine esters of alkoxy-aminobenzoic acids and other similar compounds of said acids. Alkamine esters of amino-toluic acids and other similar compounds of said acids. E. A. Wildman, Assignor to Parke, Davis, and Co. U.S.P. (A) 1,317,250 and (B) 1,317,251, 30.9.19. Appl., 29.7.18.

ALKAMINE esters of disubstituted benzoic acids in which the substituents (A) NH_2 and OR (R=alkyl) or (B) NH_2 and CH_3 may occupy any positions in the benzene ring are of value as local anaesthetics. Examples. (A) *m*-Amino-anisoyldiethylaminoethanol, $\text{NH}_2(\text{CH}_2\text{O})\cdot\text{C}_6\text{H}_4\cdot\text{CO}\cdot\text{O}(\text{CH}_2)_2\cdot\text{N}(\text{C}_2\text{H}_5)_2$ (m.p. of hydrochloride, 160° C.) is prepared by interaction of *m*-nitro-anisoyl chloride and diethylaminoethanol, and reduction of the resulting nitro-compound. (B) The diethylaminoethanol esters of *o*-amino-*p*-toluic acid and *p*-amino-*o*-toluic acid are prepared in a similar manner from diethylaminoethanol and *o*-nitro-*p*-toluoyl chloride or *p*-nitro-*o*-toluoyl chloride. The hydrochlorides melt at 157°—158° C. and 139°—140° C. respectively.

Methane; Oxidation of — [to formaldehyde]. R. K. Bailey, Lawrence. U.S.P. 1,319,748, 28.10.19. Appl., 13.5.15.

METHANE is heated with a substance containing at least a considerable proportion of combined oxygen and nitrogen (e.g., higher oxides of nitrogen or a mixture of nitric oxide and air or oxygen); the mixture is then cooled and certain of the products (higher oxides of nitrogen) are simultaneously neutralised by means of a suspension or sludge of calcium carbonate or the like. After filtering the sludge, formaldehyde may be recovered from the filtrate by distillation.—L. A. C.

Acetic acid; Preparation of — from acetaldehyde and oxygen. Verein für Chem.-Ind. in Mainz, Frankfurt. G.P. 301,274, 4.6.11.

BEFORE oxidation the volatility of the aldehyde is reduced by admixture with an inert diluent such as tetrachloroethane. The oxidation is carried out at 70°—90° C., using air or oxygen at 1—2 atm. pressure, either with or without a catalyst, the acetic acid being finally separated by fractional distillation.—G. F. M.

Pinacone; Preparation of —. Farbenfabr. vorm. F. Bayer und Co. G.P. 306,304, 6.5.17.

IN the electrolytic reduction of acetone in acid solution, the formation of more highly reduced products, such as isopropyl alcohol, is repressed by the addition of metallic salts such as those of bismuth, mercury, manganese, nickel, antimony, silver, and iron.—G. F. M.

Pinacone chlorhydrin; Preparation of —. Farbenfabr. vorm. F. Bayer und Co. G.P. 317,635, 2.9.16.

PINACONE chlorhydrin is obtained by the action of

hydrogen chloride on pinacone at temperatures above that of the formation of the addition product and below that at which pinacoline is formed. At 35°—15° C. the chlorhydrin is formed in quantitative yield, and it may likewise be obtained by heating the hydrogen chloride addition product to that temperature.—G. F. M.

Diarylurethanes; Preparation of —. Chem. Fabr. vorm. Weiler-ter Meer, Uerdingen. G.P. 308,316, 8.4.17.

DIARYLURETHANES are obtained by the interaction, either with or without a diluent, of 1 mol. of a diarylamine or a diarylamine hydrochloride and 1 or more mols. of chloroformic ester, the reaction being accompanied by the evolution of hydrogen chloride.—G. F. M.

Acetaldehyde from acetylene; Preparation of —. Konsort. für Elektrochem. Ind. G.m.b.H., Nuremberg. G.P. (A) 309,103, 19.5.16, and (U) 309,104, 18.10.16.

(A) IN the preparation of acetaldehyde from acetylene by means of acid mercury salt solutions a ferrous salt is added to the reagent, and in order to remove the aldehyde continuously as soon as it is formed, an excess of acetylene is circulated through the catalytic solution, and the unabsorbed excess of the gas, with the addition of an amount of fresh gas corresponding to that absorbed, is re-circulated through the liquid. The formation of condensation products of acetaldehyde is thereby hindered and the acetylene absorption accelerated. (B) The acid solution of a mercury salt containing an iron salt is used at a temperature below its boiling point, and has a sulphuric acid content of 6—35%. By careful regulation of the stream of acetylene it is possible to arrange matters so that the unabsorbed gas carries away the surplus heat of the reaction.—G. F. M.

Aluminium-casein compounds; Preparation of water-soluble —. J. A. Wülfing, Berlin. G.P. 312,702, 20.1.18. Addition to 312,222 (J., 1919, 925 A).

CASEIN is allowed to react with equimolecular quantities of caustic soda and aluminium hydroxide in concentrated aqueous solution. The solution of the soda-alumina-casein compound so obtained is fluid when hot, and sets to a jelly on cooling. It has a limited miscibility with alcohol. Ether precipitates the product from its aqueous alcoholic solution.—A. J. H.

Desoxycholic acid; Preparation of compounds of —. H. Wieland, Munich. G.P. 317,211, 11.2.16. (Cf. U.S.P. 1,252,212; J., 1918, 108 A.)

ADDITION products of desoxycholic acid with hydrocarbons, or other organic compounds, are produced by dissolving the components in a hot solvent, or by dissolving the desoxycholic acid in the other component in a molten state, allowing to crystallise, and washing away the excess by means of a suitable solvent. These addition compounds, being analogous to the choleic acid of the bile, which is an addition product of desoxycholic acid and a fatty acid in the proportion of 8 mols. to 1 mol., are accordingly designated in general as choleic acids. Specially mentioned are stearincholeic acid, m.p. 186° C.; naphthalenecholeic acid (1 mol. naphthalene+3 mols. desoxycholic acid), m.p. 182° C.; phenolcholeic acid, an odourless substance giving a slightly soluble barium and calcium salt; and benzaldehydecholeic acid, m.p. 186° C., from 3 mols. of desoxycholic acid and 1 mol. of benzaldehyde.—G. F. M.

Silver salts; Manufacture of therapeutic agents containing —. Sächsisches Serumwerk und Institut für Bakteriotherapie, Dresden. G.P. 317,509, 12.9.17.

By dissolving silver salts in a considerable excess

of bile, the caustic effects even of concentrated solutions are very much diminished owing to the colloidal nature of the bile. The penetrating and disinfecting powers of the silver salts are increased.—D. W.

Ether; Preparation of —. Elektrizitätswerk Louza, Basle. G.P. 317,559, 29.10.18.

MIXTURES of alcohol and ether containing up to 15% of the latter are obtained by the catalytic reduction of acetaldehyde with hydrogen with the absolute exclusion of oxygen. If only a few tenths per cent. of oxygen is present the ether formation falls off practically to nil. The reaction temperature must be maintained between 90° and 170° C.; at lower temperatures no ether is formed, and above 170° C. the aldehyde commences to decompose.—G. F. M.

Amino-sulphonic acids; Preparation of —. W. Traube, Berlin. G.P. 317,668, 4.7.16.

AMINOSULPHONIC acids are obtained by the action of fluorosulphonic acid or its salts on the corresponding bases, either direct, or in solution in water, alcohol or chloroform, etc. (J., 1919, 695 A).—G. F. M.

Mellitic acid; Preparation of —. R. Lorenz and J. Hausmann, Frankfurt. G.P. 318,200, 29.11.17.

MIXTURES of finely divided coal or soot with tar, pressed into rods or plates and then heated in the absence of air, are subjected to anodic oxidation in alkaline electrolytes, such as solutions of alkali hydroxides or carbonates. By employing a diaphragm hydrogen is obtained as a by-product. Finely divided wood charcoal, coke, etc. may be substituted entirely or in part for the soot.—G. F. M.

Fatty or wax-like compounds [adipic acid esters]; Preparation of —. Farbenfabrik. vorm. F. Bayer und Co. G.P. 318,222, 10.8.17.

THE esters of adipic acid and its derivatives or homologues with dihydric alcohols form tallowy or butter-like masses. The glycol ester may be prepared by mixing a solution of ethyleneglycol and pyridine in pinacoline with a similar solution of the dichloride of adipic acid or by the action of ethylene dibromide on potassium adipate. The propyleneglycol ester is similarly obtained from propylene di-iodide and potassium adipate, whilst α -methyladipic acid and ethylene oxide react to give a brownish waxy mass consisting of the glycol ester of methyladipic acid.—G. F. M.

Arsenic compounds, Aromatic —. The Rockefeller Institute for Medical Research, Assignees of W. A. Jacobs, W. H. Brown, M. Heidelberger, and L. Pearce. E.P. 120,382, 3.10.18. (Appl. 16,093/18.) Conv., 3.10.17.

SEE U.S.P. 1,280,120, and 1,280,125 of 1918; J., 1919, 268 A.

Oxidation of benzene. E.P. 122,167. See III.

Diacyanodiamide. G.P. 318,136. See VII.

Emulsifiable solid hydrocarbons. G.P. 308,442. See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic colour process; Theoretical basis of a new direct — by means of coloured substances. P. R. Kögel. Phot. Korr., 1919, 56, 332—337. Chem. Zentr., 1920, 91, II., 511—512.

THE process depends on the fact that only absorbed light is chemically active, so that a coloured enolic

compound which is changed by light into a colourless keto compound is affected only by light complementary to its own colour. The sensitive film, dark brown in colour, consists of a mixture of three enolic substances, yellow, red, and blue; it bleaches right out in white light but leaves the corresponding colour when acted on by coloured light. Sensitizers may be added to the mixed colours, and the pictures may be fixed.—B. V. S.

Auramine as a [photographic] sensitiser. O. Bloch and F. F. Renwick. *Phot. J.*, 1920, 60, 145—146. (See also *J.*, 1919, 926 A.)

AURAMINE, when used by itself, has very slight effect on the colour sensitiveness of silver gelatino-bromide-plates; the effect on gelatino-chloride plates is, however, very marked, a new band of sensitiveness to the light blue being added. In conjunction with dyes of the isocyanine class (e.g., Sensitol Green—German "Pinaverdol") or of the quino-cyanine class (e.g., Sensitol Red—German "Pina-cyanol") there is marked enhancement of the sensitising properties of the latter dyes in regions of the spectrum for which auramine itself does not sensitise at all. The amount of auramine required is much less than is required to produce a definite colour-screening effect. The auramine also restrains the fogging tendencies of the iso-cyanine dyes. Spectrograms are given illustrating the effects obtained.—B. V. S.

Theory of organic [photographic] developers. B. Homolka. *Phot. Korrr.*, 1919, 56, 387—391. *Chem. Zentr.*, 1920, 91, II., 660.

THE loss of developing power by the introduction of a carboxyl group into pyrogallol in the formation of gallic acid is explained by the formation of an anhydride by the carboxyl group and the hydroxyl group in para position to it. Pyrogallolcarboxylic acid and catechol-*o*-carboxylic acid in which such anhydride formation is not possible are both developers.—B. V. S.

Colorimeter. Gamble. See XXIII.

PATENTS.

Photographic films; Hardening — during development. J. H. Christensen, Sölleröd. E.P. 135,477, 14.11.19. (Appl. 28,287/19.) Conv., 20.11.18.

THE hardening effect on the gelatin of developers containing, e.g., pyrogallol, catechol, or quinol, is considerably increased by the addition to the developer of substances which counteract the swelling of the gelatin, such as alcohol, carbonates, silicates, etc. The addition of a restrainer such as bromide or a borate is preferable in order to prolong the time of development. A film in relief is obtained from which the unexposed and unhardened parts may be washed with hot water, and which may be used for various printing processes, including colour transfer processes.—B. V. S.

Coloured photographs or kinematograph films; Producing —. W. Finnigan, London, and R. A. Rodgers, Hove. E.P. 140,349, 7.1.19. (Appl. 28,247/19.)

Two negatives are taken on two plates or films placed face to face, the upper one being an ordinary plate or film and the lower one panchromatic. From these negatives two positives are printed which are coloured blue-green and pink-red respectively and combined in register.—B. V. S.

Brown tone on [photographic] development prints; Process for producing —. C. Schleussner A.-G., Frankfurt. G.P. 318,503, 10.8.18.

LIGHT-FAST brown tones are obtained by bleaching the developed print, before or after fixing, in a

solution of mercuric chloride containing bromide and then treating it with a solution of sodium thio-sulphate containing lead.—B. V. S.

Gradation of photographic negatives and diapositives; Improvement of —. J. Rieder, Berlin-Steglitz. G.P. 318,820, 5.4.19.

THE picture is provided on the film side with a coating of asphalt, caoutchouc, or similar light-sensitive resin, exposed through the support, and the coating developed with alcohol or acetone, etc. The picture is then treated with an intensifying or reducing solution or with a dye solution, the action of such reagents being modified by the variable permeability to aqueous solutions produced by the top coating.—B. V. S.

Pigment [carbon] prints to metal; Process for transfer of — as etching resists for process blocks. Rotophot-A.-G. für graphische Industrie, Berlin. G.P. 318,687, 26.9.15.

A MATT surface is produced on the metal by electrolytic action before transfer of the carbon print; this prevents the formation of blisters.—B. V. S.

Photographic films; Production of — in natural colours. R. Wellesley, Goodmayes, and T. M. Sanders, Sevenoaks. E.P. 140,560, 18.8.19. (Appl. 4053/19.)

XXII.—EXPLOSIVES; MATCHES.

Tetryl; Stability of —. C. L. Knowles. *J. Ind. Eng. Chem.*, 1920, 12, 246—248.

STABILITY tests of tetryl and of mixtures containing it were made in a modification of Obermüller's apparatus (*J.*, 1905, 347). The purity of tetryl is directly proportional to its m.p. Quantitative heat tests are of little value as an indication of the stability. The instability of ordinary tetryl is due to a large extent to the presence of tetranitrophenylmethylnitramine (*m*-nitrotetryl) formed by the nitration of the monomethylaniline in the dimethylaniline used. *m*-Nitrotetryl may be removed from tetryl by boiling with water which converts it into soluble trinitromethylaminophenol, or with sodium carbonate solution, which yields a soluble salt of trinitroresorcinol. Tetryl is slightly hydrolysed by a dilute solution of sodium carbonate forming sodium picrate, which, however, is stable and does not lower the stability of tetryl to any appreciable extent. Hence sodium carbonate is a satisfactory stabilising agent for tetryl.—C. A. M.

Trinitroxylenes; Manufacture of — for use as a substitute for TNT in bursting charges for high explosive shell. J. Marshall. *J. Ind. Eng. Chem.*, 1920, 12, 248—250.

A MIXTURE of 30 to 50 pts. of trinitroxylenes suspended in 70 to 50 pts. of TNT at 100° C. yields a fluid mass which can be cast directly. The castings resemble paraffin wax, have high and uniform density, are not hygroscopic, and do not become oily even when stored at high temperatures. They can be completely detonated with smaller primers of tetryl than can castings of TNT, and better results can be obtained with crude mixtures of the two compounds than with refined products. Trinitroxylenes is best obtained by the nitration of *m*-xylene, the yields being low from *p*-xylene, whilst xylenes rich in *o*-xylene give low yields of a product with low freezing point. A xylene with the following h.pt. specifications gives the best yields and most satisfactory freezing point:—Range, first drop to flask dry, 3° C.; first drop between 137° 2° and 139° 2° C.; flask dry between 138° 5° and 140° 5° C. Trinitroxylenes with a freezing point of 161° 5° C. or higher shows no tendency to agglomerate into

crystals on treatment with hot water, and can be readily neutralised. Coke-oven xylene gives a better trinitroxylene than xylenes from other sources such as water-gas.—C. A. M.

Hexanitrodiphenylamine; Preparation of — and its use as a primer for shell charges. J. Marshall. J. Ind. Eng. Chem., 1920, 12, 336–340.

HEXANITRODIPHENYLAMINE (hexyl) is exceedingly stable and much safer to handle than tetranitromethylaniline (tetryl) or tetranitraniline (TNA). It is somewhat inferior in detonating power to these, but is superior to TNT. It also has the advantage over the latter of simplicity of manufacture, and involves less costs for plant and labour. It is made by the successive nitration of dinitrodiphenylamine and tetranitrodiphenylamine. In the preparation of the former from aniline yields of 90–95% are obtained by the process claimed in U.S. Pat. 1,309,580/19 (J., 1919, 676A). The dinitro compound can be satisfactorily nitrated by adding it at 70° C. to 3.5–1 parts of a mixed acid containing 30 to 45% of nitric acid and 50 to 40% of sulphuric acid and then raising the temperature to 80°–90° C. until the evolution of NO₂ has practically ceased. The charge is then cooled and filtered and is ready for further nitration. It is added to 3.75 parts of an acid mixture containing 60% HNO₃ and 40% H₂SO₄ at 70° C. and the mixture heated for an hour at 90° C., cooled and filtered from the crystalline hexanitrodiphenylamine. Nitration of the dinitro to the hexanitro compound in one stage (Hoffmann and Dame, J., 1919, 555A) yields an amorphous product. Acid is removed from the "hexyl" by flooding the crystalline material with a large volume of water, and then washing it 2 or 3 times for an hour with boiling water; it is finally centrifuged and dried in trays at temperatures up to 80° C.—C. A. M.

Nitrocellulose; Examination of —. A. Schrimpf. Z. ges. Schiess- u. Sprengstoffw., 1920, 15, 91–93.

THE results obtained with Bergmann and Junk's stability test (J., 1920, 314A) are tabulated. To determine the explosion temperature, 0.05 g. of dried nitrocellulose is placed in a test-tube, inserted in an oil bath at 100° C., and the temperature of the bath is raised 5° per min. The results obtained with various explosives are given. With stable nitrocellulose a sharper detonation is obtained than with impure samples; in the latter case nitrous fumes are evolved above 140° C., and the explosion is more of the nature of an ignition than a detonation.—W. J. W.

Smokeless powders; Acidity in —. A. Angeli and G. F. Errani. Gazz. Chim. Ital., 1920, 50, i., 139–148.

DIMETHYLAMINOAZOBENZENE has been applied to the investigation of the presence of acid in various smokeless powders (J., 1918, 608A). Microscopical examination of very thin sections of the powders treated with this indicator shows that in some cases the acidity is distributed almost uniformly throughout the mass of the explosive, whereas in others it is localised in isolated regions often with sharply marked outlines. With certain altered nitrocellulose powders, the intact strips were not coloured on immersion in the reagent, but the latter soon assumed a more or less intense pink colour; if, however, the strips were broken or scraped, the fresh surfaces thus made were immediately coloured scarlet by the indicator. The acids occurring at these surfaces were not completely removed by prolonged washing with boiling water, and were almost insoluble in ether and only partially soluble in alcohol. Towards litmus these strips showed an acid reaction, even at their surface. Coloured plates are given showing the different effects obtained.—T. H. P.

Mercury fulminate; Oxalates in —. A. Langhans. Z. ges. Schiess- u. Sprengstoffw., 1920, 15, 89–90.

MERCURIC oxalate cannot be determined in mercury fulminate by a mercury estimation on account of the slight difference in the mercury content of the two salts. Mercurous oxalate (81.96% Hg) can be more readily detected. The oxalates may be separated by means of sodium thiosulphate, in which they are insoluble. The brown coloration which occasionally is found in mixtures of mercury fulminate and potassium chlorate after drying may be due to overheating. Traces of oxalates in the fulminate may produce this coloration after exposure to light owing to their decomposition into mercuric oxide. The explosive effect of mercury fulminate is diminished by presence of oxalates. With 1% the lead plate shows a fairly good impression and radial markings, while with 10% no indentation at all is produced.—W. J. W.

PATENTS.

Explosives; Manufacture of —. G. A. Hedberg, Alingsas. U.S.P. 1,335,785, 6.4.20. Appl., 9.9.19.

MEAL of pine-bark or of pine cones is used as an ingredient in explosives.—W. J. W.

Explosive. J. R. Mardick, Assignor to Electro Metallurgical Co., Niagara Falls. U.S.P. 1,335,788, 6.4.20. Appl., 15.12.17.

AN explosive contains a solid nitro-aromatic compound with at least three nitro-groups, an oxidising agent, a plasticising ingredient, and a silicon-containing substance, the proportions of these ingredients being adjusted so as to render the composition plastic.—W. J. W.

Explosive. J. R. Mardick, and F. M. Becket, Assignors to Electro Metallurgical Co. U.S.P. 1,335,789, 6.4.20. Appl., 19.6.18.

AN explosive comprises a nitro-aromatic compound, an oxidising agent, and a magnesium-silicon alloy.—W. J. W.

Explosive, and method of preparing the same. J. R. Mardick, Assignor to Electro Metallurgical Co. U.S.P. 1,335,790, 6.4.20. Appl., 22.8.18.

AN explosive composition consists of an oxidising salt, a metallic reducing agent, a solid nitro-aromatic compound containing at least three nitro-groups, and a gelatinised plasticising ingredient, the amount of nitro-compound being at least equal to that of the reducing agent, and the proportion of plasticising agent being the minimum required to render the composition plastic.—W. J. W.

Nitroglycerin explosive; Desensitised —. A. A. Starring, Kansas City, Assignor to B. Mossman. U.S.P. 1,336,661, 13.4.20. Appl., 30.11.18.

A DESENSITISING agent comprising carbon tetrachloride and an oil miscible with nitroglycerin is added to nitroglycerin explosives.—W. J. W.

Material capable of nitration; Preparation of — from wood cellulose. C. Claessen, Berlin. G.P. 300,069, 29.7.15.

THE dried cellulose pulp is rolled or pressed and the compressed material reduced to a fine state of division by a suitable wood-working machine. A uniform porous substance is obtained which can be nitrated in the usual way.—B. V. S.

Cellulose; Process and apparatus for nitration of —. A. Vajdady, Budapest. G.P. 300,740, 21.10.15. Conv., 24.7.15.

DURING the nitration process the material is kept in motion so as to absorb the acid in a gradual, continuous and uniform manner, and is then pressed

to yield a product containing only 70% of acid. The rotary nitration apparatus, which is mounted on an inclined shaft and gradually diminishes in size towards the top, is provided with spirally-arranged, internal ribs.—W. J. W.

Nitrocellulose powders; Manufacture of —. Chem. Fabr. von Heyden, A.-G., Radebeul. G.P. 302,460, 17.7.17.

As a partial or entire substitute for acetone fural is employed, either alone or mixed with methyl alcohol, ether, benzene, ethyl formate, ethyl acetate, or chloroform.—W. J. W.

Cellulose derivatives [nitrocellulose]; Process for gelatinising —. Elektro-Osmose A.-G., Berlin. G.P. 309,260, 6.6.17.

THE water in moist cellulose derivatives, e.g., nitrocellulose, is replaced by a gelatinising liquid by means of electro-osmosis.—L. A. C.

Granulated explosives; Process for manufacture of compressed charges from —. Fürstlich Plessische Miedziaknitfabrik, Mittel-Laszk. G.P. 304,000, 9.6.17.

By addition of albumin and formaldehyde to explosives they may be granulated and then compressed, the formaldehyde-albumin mixture also serving as a protection against absorption of moisture.—W. J. W.

Ammonium nitrate; Process for the liquefaction of — for explosive charges. Dynamit Akt.-Ges. vorm. A. Nobel und Co., Hamburg. G.P. (A) 305,567, 31.1.17, and (B) 305,568, 8.1.18.

(A) AMMONIUM nitrate (m.p. 152° C.) is mixed with dicyanodiamide (m.p. 205°), a mixture containing 85% of the former becoming fluid at 115° C. The use of the mixture in the preparation of explosives is easier and less dangerous than that of ammonium nitrate alone. (B) Water is used as the liquefying medium. The pouring temperature of a mixture of 66% ammonium nitrate, 10% sodium nitrate, 20% nitrotoluol, and 4% water is about 80° C.—B. V. S.

Ammonium perchlorate explosives; Manufacture of — capable of being cast. Chem. Fabr. von Heyden A.-G. G.P. 317,030, 2.9.17.

AMMONIUM perchlorate is mixed with nitrates of the alkalis, or of nitrogen bases, and alkali salts of fatty acids or amides. An explosive consisting of ammonium perchlorate, 2.5 parts; ammonium nitrate, 1.5 parts; sodium nitrate, 1.8 parts; and acetamide, 1.5 parts; can be cast at 50° C.—W. J. W.

Incendiary composition for flame-projectors. R. Fieldler, Berlin-Halensee. G.P. 310,148, 21.2.18.

AN incendiary composition consists of nitrocellulose dissolved in ether, and converted into a gelatinous mass by addition of carbon bisulphide and a suitable hydrocarbon. Phosphorus or sulphur, alone or in combination with an oxygen carrier, may be added.—W. J. W.

Signal lights and fire balls; Manufacture of — free from metals and shellac. Geka-Werke Offenbach, G. Krebs. G.P. 314,868, 10.11.17.

PITCH is intimately mixed with alkaline-earth nitrates and barium peroxide, after which chloroform, ethyl acetate, acetone, or carbon bisulphide is added, and the mixture compressed to the desired shape. The resulting product after heating, drying, and cooling possesses the requisite hardness and durability, and burns with intense brilliancy and little smoke.—W. J. W.

Sulphur dye. U.S.P. 1,318,022. See IV.

Washing cellulose preparations. G.P. 307,078. See V.

XXIII.—ANALYSIS.

Viscosimeter; The MacMichael torsional —. W. H. Herschel. J. Ind. Eng. Chem., 1920, 12, 282—286.

THE error due to the elongation of the wire in the MacMichael torsional viscosimeter (J., 1915, 1226) is about 1.6% at 80° C. The ratio between the results obtained with this instrument and with Saybolt's viscosimeter decreases with rise in temperature, and the conversion of the degrees into each other is impracticable except for some assumed sp. gr. A formula is given for converting the readings into poises, but it is not possible to calculate the deflection from the dimensions of the viscosimeter. Each instrument should be separately calibrated by means of liquids of known viscosity and of approximately the same density as the liquid to be examined.—C. A. M.

Colorimeter; The Bawtree —. W. Gamble. Phot. J., 1920, 60, 149—151.

A LONG rectangular box is divided longitudinally into equal parts, each part being lined with mirrors. A lamp illuminating both compartments is placed at one end. The box is interrupted in the middle to allow the insertion of shuttered diaphragms in each compartment, that on one side being plain glass and gelatin film, and that on the other side being in three equal parts, red, green, and blue-violet colour-filters. Each diaphragm is provided with a graduated shutter, and the colours are so adjusted that their combination in equal parts is a colour match for the white light. At the other end of the box are two opal reflectors arranged at 45° over the two compartments respectively, and one of them is provided with a centre aperture to enable the other one to be seen through it when viewed from the side of the box. A coloured filter, placed at the diaphragm, or a sample of pigment placed on the opal can be matched and expressed in terms of the three standard colour screens.—B. V. S.

Flask for the fractional distillation of frothing liquids. E. Lenk. Chem.-Zeit., 1920, 44, 330.

THE lower end of a reflux condenser, the inner tube of which is formed into a series of pear-shaped bulbs, is fused on to the neck of a distillation flask at a point opposite to but below the side tubulure of the flask. By means of a three-way cock the air may be exhausted from the condenser or from the receiver attached to the outlet tube of the flask. In the distillation of viscous liquids a vacuum is first created in the condenser, into which the froth rises and is broken against the walls of the bulbs, and when the liquid is boiling quietly the air is exhausted in the other direction and the distillation continued.—C. A. M.

Electrodes; Platinised glass — for electrolytic determinations. G. Meillère. J. Pharm. Chim., 1920, 21, 311—313.

A SOLUTION of platinum chloride in methyl alcohol, to which a small quantity of an essential oil is added, is applied to the surface of the glass, into which has previously been fused a short piece of platinum wire; the glass is then heated until all the organic matter has been burnt. Several successive treatments may be applied in order to obtain a good coating of platinum. If the glass is etched previously with hydrofluoric acid the platinum adheres better and has a more or less matt surface.—W. P. S.

Indicators and their industrial application. H. A. Luhs. J. Ind. Eng. Chem., 1920, 12, 273—274.

IN the precipitation of anthranilic acid from its alkaline solution by the addition of mineral acid, an

acid reaction to methyl-red indicates that the end-point is approached; on further addition of acid the point of maximum precipitation is shown by an acid reaction to thymol-blue. Thymolsulphophthalein is a useful indicator in the "liming" of sulphonation mixtures; a red coloration indicates that free acid is still present. When the indicator shows a yellow colour the end-point is near, whilst a blue colour shows that the mixture is alkaline. Dibromoresulphophthalein or dibromothymolsulphophthalein may be used as substitutes for litmus; the former changes from yellow to purple and the latter from yellow to blue.—W. P. S.

Arc images in chemical analysis. W. R. Mott. Trans. Amer. Electrochem. Soc., 1920, 121—160. [Advance copy.]

An electric arc is arranged to project through a lens an image of the arc on to a screen, a magnification of 20 times being generally employed, though a smaller magnification is necessary for faint arcs; the lower carbon has a small depression drilled in it for reception of about 0.5 gm. of the material under examination; generally the lower carbon is positive. Observations are made as to the behaviour of the substance on melting, formation of a metallic bead or wetting of the carbon, the colour of the flame, smoke formation, odour, nature and sequence of distillation, and especially the nature and colour of the deposits on the electrodes, both hot and cold. Reagents may be added to modify the course of the reaction, such as silica for the formation of characteristic coloured beads with certain metallic oxides, lead as a solvent for gold in the presence of iron, etc. The metals are divided into two main classes, (A) those that do not wet carbon, form molten beads, and give light in the core of the arc stream only, and (B) those that wet and dissolve carbon when melted and give light in both the core and the shell of the arc stream. Generally class A metals are easy to reduce from their oxides and class B difficult. The classes are subdivided into two groups in class A (group I including the metalloids) and seven groups in class B. A long list of distinctive tests is given for 65 elements arranged in groups as above. The delicacy of the tests is greatest where ordinary methods, including spectrum analysis, are least effective, especially as regards the very refractory elements; the amount which can be detected varies from 0.02 mg. for silver, gold, and tungsten to 0.1 mg. for sulphur, phosphorus, cadmium, platinum, chromium, vanadium, titanium, uranium, yttrium, zirconium, thorium, columbium, molybdenum, and tantalum, and about 1 mg. for most of the other elements. In mixtures the delicacy generally decreases about 20%. Separations can be often effected, in some cases an addition with intermediate boiling point assisting the separation. The analysis is mainly qualitative, but semi-quantitative results can often be obtained; e.g., cadmium in zinc can be estimated to within a few per cent., and chromium can be estimated by the amount of colour produced in the barium aluminate test for that metal.—B. V. S.

Chlorine; Determination of — with the nephelometer. A. B. Lamb, P. W. Carleton, and W. B. Meldrum. J. Amer. Chem. Soc., 1920, 42, 251—259.

SILVER chloride was precipitated in 50% alcohol-water solutions of sodium chloride of widely differing concentrations, and the mixtures were heated for 30 mins. at 40° C. after precipitation. In this way a more intense and constant opalescence was produced than could be attained at room temperature. By this method chlorides can be estimated over the concentration range $4-300 \times 10^{-4}N$. The

opalescence of silver chloride in water is 15% greater than in 50% alcohol or 50% acetic acid.

—J. F. S.

Iron [in grape must etc.]; Determination of minute quantities of —. L. Mathieu. Bull. Assoc. Chim. Sucr., 1919, 37, 205—208.

THE ash from 10 c.c. of grape must or a corresponding amount of other material is treated with 5 c.c. of hydrochloric acid (1 in 4), and, without filtration, 10 c.c. of 1% potassium thiocyanate is added. The liquid is made up to 50 c.c., and its colour is matched by adding standard ferric chloride solution to 50 c.c. of a solution containing the same quantities of acid and thiocyanate as the test liquid.

—J. H. L.

Proteins; Use of ethyl acetate as a precipitant for —. A. Marie. Ann. Inst. Pasteur, 1920, 34, 159—161.

ETHYL acetate gives a marked precipitate with even dilute solutions of proteins, and a turbidity with peptones, proteoses, or albumoses. On the other hand, solutions of amino-acids or of vegetable or animal alkaloids show no change on the addition of ethyl acetate.—W. G.

Amino-acids; Determination of — by means of the hydrogen electrode. E. L. Tague. J. Amer. Chem. Soc., 1920, 42, 173—184.

THE neutralisation curves of amino-acids are obtained as follows: Sufficient standard alkali is added to a definite volume of the aqueous solution of the amino-acid to give it a pH value of about 12.5. Then to an equal volume of water the same standard alkali is added to give the same pH value, care being taken to add sufficient water to give the blank the same volume as the original at the pH value compared. By subtracting the number of c.c. used in the blank from that required in the original the number of c.c. of alkali necessary to neutralise the amino-acid alone is obtained.—J. F. S.

Crude fibre [in feeding stuffs etc.]; Condenser for use in the determination of —. C. A. Clemens. J. Ind. Eng. Chem., 1920, 12, 288—289.

A CONDENSER possessing several advantages over the apparatus devised by Spears (J., 1919, 230a) is described.—C. A. M.

See also pages (A) 395, *Combustion products etc.* (Cistek). 397, *Naphtha* (Hill and Chillas). 400, *Cellulose in woods* (Dore). 401, *Calcium nitrate* (Platon). 402-3, *Spent oxide. Hydrosulphite* (Bruhns). *Carbonyl chloride* (Delépine and others). 409-10, *Iron ore* (Little and Hult, also Schwarz). *Phosphorus in iron etc.* (Kinder, Ridsdale). 417, *Linseed oil* (Wolff). *Nephelometric values* (Csonka). 418, *Sugar in tanning extracts* (Grasser). 419, *Soil acidity and alkalinity* (Wherry). *Chlorides in soil* (Hirst and Greaves). 420, *Molasses* (Lippmann). 422, *Melanin-value of potatoes* (Haehn). *Water in foods* (Scheringa). 423, *Caffeine in coffee* (Vantier). 425, *Morphine* (Rapp). *Chlorobenzenes* (Bourion). *Aerolein* (Tsalapatanis). 426, *Distilling water from solvents* (Walker). 429, *Nitrocellulose* (Schrumpf).

PATENTS.

Carbon monoxide; Oxidation of —. K. Hofmann, Charlottenburg. G.P. 307,614, 19.7.16.

THE oxidation of carbon monoxide by chromic acid solutions is much accelerated by the addition of mercuric oxide, and absorption of this gas from air or gaseous mixtures by the reagent is sufficiently rapid for analytical or hygienic purposes.

—G. F. M.

Soda-lime. U.S.P. 1,335,949. See VII.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Beekton. Settling tanks, separators, etc. 13,423. May 15.

Bilsland, Macbeth, and Dunlop Rubber Co. Mixing or grinding mills etc. 14,115 and 14,118. May 21.

Blom. Effecting condensation reactions. 13,055. May 11.

De Wendel. Transport and storage vessels for liquid air or gas. 12,942. May 10. (Ger., 7,11.16.)

Doverdale. Filtering, clarifying, etc. liquids. 14,030. May 21.

Duckham, and Thermal Industrial and Chemical Research Co. Fractional distillation. 14,104. May 21.

Emerson. Apparatus for distillation. 12,922. May 10. (U.S., 8.5.19.)

Krüger and Krüger. Mixing air and liquids. 13,210. May 13.

Morgan, and Thermal Industrial and Chemical Research Co. Recovering gases absorbed by solids. 14,102. May 21.

COMPLETE SPECIFICATIONS ACCEPTED.

8954 (1918). Barbet et Fils et Cie. Heat exchangers for continuous rectification of liquefied gases. (142,519.) May 19.

3851 and 18,742 (1919). Bulgin, Hall, and Searle. Apparatus for treating air or gases with liquids or *vice versa*. (142,941.) May 27.

3985 (1919). Schuck. Production of a catalyst. (142,576.) May 19.

17,817 (1919). Tiesen. Furnaces. (142,710.) May 19.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Duckham, and Woodall, Duckham, and Jones. Gasifying fuel. 12,937 and 13,373. May 10 and 14. Durrant and Wilford. Motor fuel. 13,173. May 12.

Edser. Production of fuel. 14,126. May 21.

Félizat and Guignard. Destructive distillation of shales, schists, etc. 12,951. May 10. (Fr., 12.4.19.)

Hughes. Artificial fuel. 13,882. May 20.

Hutton and Neilson. Acid refining of mineral oils. 13,887-8. May 20.

Lewis and Quain. Purification of gases. 13,554. May 17.

Morgan, and Thermal Industrial and Chemical Research Co. Treatment of acid used for purifying oils etc. 14,103. May 21.

Perry. Apparatus for distilling carbonaceous material. 12,894. May 10.

Rambush. Removal of sulphur from gases. 14,106. May 21.

Sawtelle. Destructive distillation of wood. 13,718. May 18. (U.S., 10.9.16.)

COMPLETE SPECIFICATIONS ACCEPTED.

2661 (1919). La Porta and Bartolomeis. Furnaces for distilling asphalt rock, bituminous shale, etc. (142,541.) May 19.

4154 (1919). Freeman. Gasifying solid and liquid fuels. (142,945.) May 27.

11,178(1919). Marks (U.S. Industrial Alcohol Co.). Liquid fuel. (143,017.) May 27.

14,475 (1919). Groundstroem. Gas retorts. (112,689.) May 19.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

Brunner. Manufacture of hydrazobenzene etc. in solid form. 13,592. May 17. (Switz., 12.7.19.)

Morgan. Manufacture of colour-producing intermediates. 13,954. May 20.

Poncelot. Colouring matter. 13,157. May 12. (Fr., 12.5.19.)

COMPLETE SPECIFICATION ACCEPTED.

11,101 (1919). Brassard and Crawford, and Buckle. Manufacture of indophenolic compounds. (143,014.) May 27.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

British Cellulose and Chemical Manuf. Co., and Roy. Apparatus for making threads. 13,796 and 13,926. May 19 and 20.

Bronnert. Manufacture of artificial silk. 12,972, 13,387, 13,586, 14,068. May 10, 14, 17, and 21.

Commin. Manufacture of fibre compositions. 13,869. May 20.

Dreyfus. Manufacture of cellulose derivatives. 13,289 and 13,290. May 13.

Heilbronner. Impregnation of paper etc. 13,084. May 11. (Ger., 12.1.17.)

Lilienfeld. Producing viscous liquids. 13,186. May 12. (Austria, 1.8.19.)

Lilienfeld. Production of cellulose ethers. 13,187. May 12. (Austria, 5.5.20.)

Lilienfeld. Manufacture of alkali cellulose. 13,286. May 13. (Austria, 1.8.19.)

Lilienfeld. Manufacture of plastic masses. 13,350. May 14. (Austria, 1.8.19.)

Lilienfeld. 13,188, 13,287, and 13,568. See XVII.

Pollak (A.-G. Seeriet Bleicherei). Refining and perfecting cotton fabrics. 13,840. May 19.

Thunert. Production of paper. 13,842. May 19. (Ger., 12.7.19.)

COMPLETE SPECIFICATION ACCEPTED.

24,123 (1919). Kron. Obtaining fibre for the manufacture of pasteboard. (135,466.) May 27.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Bayer und Co. Dyeing with acid sulpho amino dyestuffs. 13,161. May 12. (Ger., 31.10.14.)

Kershaw. Bleaching fabrics. 13,731. May 19.

Lord. Apparatus for treating hanks of yarn with liquid. 13,875. May 20.

Sharp. 14,024. See IX.

Soc. Chim. des Usines du Rhône. Dyeing cellulose acetate artificial silk, films, etc. 13,541. May 17. (Fr., 5.9.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

798 (1919). Heymann. Dyeing. (142,899.) May 27.

6362 and 22,202 (1919). Whitaker and Whitaker. Machines for dyeing, scouring, and washing wool, etc. (142,602.) May 19.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

- Asheott. Production of anhydrous magnesium chloride or anhydrous double chlorides of magnesium. 13,061. May 11.
- Craig, Spence, and Spence and Sons. Purification of sulphur. 13,293. May 13.
- Hickman. Fixation of nitrogen. 13,348. May 14.
- Mackay. 12,957. *See X.*
- McKee. Process of making zinc oxide and hydrogen. 13,579. May 17.
- Morgan and others. 14,103. *See II.*
- Nitrogen Corporation. Production of ammonia. 13,810. May 19. (U.S., 23.3.16.)
- Norsk Hydro-Elektrisk Kvaestofaktieselskab. Production of ammonia and formic acid or ammonium formate from barium cyanide. 13,702. May 18. (Norway, 26.5.19.)
- Norsk Hydro-Elektrisk Kvaestofaktieselskab. Producing mixtures of nitrogen and hydrogen. 14,069. May 21. (Norway, 23.5.19.)
- Schweiz. Sodafabrik. Production of caustic soda or soda lye. 13,518. May 17. (Switz., 1.6.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 3311 (1918). Pope and Heycock. Production and utilisation of sulphur dichloride. (142,879.) May 27.
- 20,339 (1918). Audianne. Heat interchangers for gases for use in the contact process of making sulphuric acid. (142,522.) May 19.
- 3508 (1919). Darrasso Frères, and Dupont. Utilising marine algae for making formic, acetic, and butyric acids and for extracting salts of iodine and potash. (123,325.) May 27.
- 14,713 (1919). Blair, Campbell, and McLean, Blair, and Ferguson. Preparing powerful oxidising gases for removing carbon and sulphur deposits in hydrogen manufacture. (143,064.) May 27.
- 32,568 (1919). Fabr. de Prod. Chim. de Thann et de Mulhouse. Manufacture of potassium sulphate. (137,296.) May 27.
- 32,886 (1919). Fredriksstad Elekt. Fabr. *See XI.*

VIII.—GLASS; CERAMICS.

APPLICATIONS.

- Atkinson, and Stein and Atkinson. Glass furnaces. 13,924. May 20.
- Beardmore, and Boulton, Ltd. Potters' stove for making earthenware etc. 13,502. May 17.
- Hailwood. Glass manufacture. 14,028. May 21.
- Jones and Sparkes. Fire-brick. 13,747. May 19.

COMPLETE SPECIFICATIONS ACCEPTED.

- 14,455 (1919). Musiol. Enamels and enamelling. (142,688.) May 19.
- 17,458 (1919). Möhn. Oil-fired furnaces for melting glass etc. (143,091.) May 27.
- 20,386 (1919). British Thomson-Houston Co. (General Electric Co.). Refractory material. (142,721.) May 19.
- 22,261 (1919). White (Fours et Procédés Mathy). Crucible furnaces for glass etc. (143,117.) May 27.
- 24,437 (1919). Wilkinson. Glass-furnaces. (143,125.) May 27.

IX.—BUILDING MATERIALS.

APPLICATIONS.

- Crozier. Manufacture of cementitious articles. 13,808. May 19.
- Sharp. Solution for fireproofing wood, fabrics, etc. 14,024. May 21.
- Wennerström. Manufacture of cement. 13,164. May 12. (Sweden, 10.2.19.)

COMPLETE SPECIFICATION ACCEPTED.

- 5153 (1919). Bravin. Concrete etc. (142,958.) May 27.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

- Budd and Ledwinka. Annealing metals. 13,899. May 20.
- Coles. Apparatus for electro-deposition of metals. 13,272. May 13.
- Hess and Kirschner. Electroplating. 13,210. May 13. (Austria, 10.10.17.)
- Jones. Fusing and purifying non-ferrous metals etc. 13,870. May 20.
- Kaiser and Obenauer. Hardening-agent for wrought iron, steel, etc. 13,513. May 17. (Ger., 18.1.18.)
- Kroll. Producing alloys containing alkaline-earth metals. 13,849. May 19.
- Langer. Electrolytic separation of metals. 13,775. May 19.
- Mackay. Extraction of tungstic acid from tungsten ores. 12,957. May 10.
- Mining and Metallurgical Processes, Ltd. Blast roasting apparatus etc. 13,958. May 20. (Australia, 9.7.19.)
- Moffat. Treating metallic ores. 13,570. May 17. (U.S., 30.4.18.)
- Walter. Producing alloys containing silicon. 13,830. May 19. (Austria, 28.10.18.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 11,628-9 (1916). Stahlwerke Lindenberg A.-G. Steel alloys. (103,809 and 103,810.) May 27.
- 18,685 (1918). Hadfield. Refining of steel. (142,885.) May 27.
- 3773 (1919). Turnbull. Cleaning blast-furnace gas. (142,938.) May 27.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

- Chile Exploration Co. Electrodes. 13,572. May 17. (U.S., 21.1.20.)
- Coles. 13,172. *See X.*
- Fuller, and Fuller's United Electric Works. Galvanic batteries. 13,262. May 13.
- Greenwood and Batley, Leitner, and Wood. Electric accumulators etc. 13,955. May 20.
- Herrmann. Manufacture of micanite. 13,670. May 18. (Fr., 29.9.19.)
- Herrmann. Manufacture of electric insulating materials. 13,671-2. May 18. (Fr., 7 and 8.11.19.)
- Hess and Kirschner. 13,210. *See X.*
- Houston. Storage batteries. 12,868. May 10.
- Langer. 13,775. *See X.*
- Pouchain. Positive accumulator electrodes. 12,939. May 10.
- Pouchain. Primary battery. 12,940. May 10.
- Siemens-Schuckertwerke. High-tension electric depositing plants. 13,694. May 18. (Ger., 31.3.16.)
- Webb. Electrolysers. 13,070. May 11.
- Zubiria y Smith. Electric furnaces. 12,953-4. May 10. (Spain, 21 and 22.5.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 10,179 and 16,154 (1919). Withers (National Electro-Products, Ltd.). Apparatus for electrolysing liquids. (143,002 and 143,082.) May 27.
- 19,378 (1919). Collins. Electric furnaces. (133,026.) May 19.
- 32,886 (1919). Fredriksstad Elektrokemiske Fabriker Akt. Apparatus for electrolysing metal salt solution. (138,862.) May 27.
- 9997 (1920). Moore. Electric furnaces. (143,176.) May 27.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

- Lilienfeld. Production of oily bodies of high boiling point. 14,091. May 21. (Austria, 10.5.20.)

Salomonson. Neutralising oils or fats. 13,970. May 20.

Soc. Anon. l'Oxydrique Franç. Catalytic hydrogenation of oils, fats, etc. 13,085. May 11. (Fr., 28,519.)

Wade (Wilson and Co.). Compounding fats and oils. 13,582. May 17.

White. Mechanical digester for obtaining oils and fats by cooling. 13,041. May 11.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

McKee. 13,579. See VII.

Nelson. Manufacture of carbon for pigmental etc. purposes. 14,065. May 21.

Scholz and Tiedemann. Working up linoleum scraps into new linoleum. 13,930. May 20. (Ger., 16,518.)

COMPLETE SPECIFICATION ACCEPTED.

20,577 (1919). Arie. Antifouling paint for ships' bottoms. (131,601.) May 27.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

COMPLETE SPECIFICATIONS ACCEPTED.

4242 (1919). Smith (Claessen). Utilising bark shavings from rubber trees and extraction of rubber therefrom. (142,946.) May 27.

13,980 (1919). Neilson. Rubber compounds. (127,821.) May 19.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Broens. Product for leather dressing or tanning, dyeing, and tannery. 13,705. May 18.

Dunham. Making casein solution. 13,561. May 17.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

Lilienfeld. Production of ethers of carbohydrates, their conversion products and derivatives. 13,188, 13,287, and 13,658. May 12, 13, and 17. (Austria, 6,819 and 5,520.)

COMPLETE SPECIFICATIONS ACCEPTED.

3508 (1919). Darrasse Frères, and Dupont. See VII.

3509 (1919). Darrasse Frères, and Dupont. Utilising carbohydrate in plants, vegetable offal, and waste products. (123,326.) May 27.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATION.

Fazi. Brewing or preserving beer. 13,177. May 12. (Ital., 22,519.)

COMPLETE SPECIFICATIONS ACCEPTED.

14,046 (1919). Baycr and Jensen. Production of alcohol and yeast from seaweed. (119,030.) May 27.

22,905 (1919). Fazi and Peroni. Malting process. (143,121.) May 27.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Activated Sludge, Ltd., and Coombs. Separating matters suspended in sewage etc. 13,202. May 13.

Activated Sludge, Ltd., and Coombs. Sewage etc. purification plants. 13,203. May 13.

Bateman. Treatment of tea. 13,375. May 14.

Clark. Compounds for baking processes etc. 13,815. May 19.

Longley. Drying sewage etc. 13,256. May 13.

Pannevis. Roasting cocoa beans etc. 12,956.

May 10. (Holland, 1,219.)

Westaway. Production of cream. 13,472. May 17.

COMPLETE SPECIFICATIONS ACCEPTED.

7119 (1919). Crosfield and Sons, and Wheaton. Manufacture of a base-exchanging substance and its use in purifying and/or softening water. (142,974.) May 27.

32,839 (1919). Bollmann. Manufacture of food-stuffs. (142,764.) May 19.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Lilienfeld. Manufacture of dialkyl sulphates. 13,349. May 14. (Austria, 15,1213.)

Soc. Chim. des Usines du Rhône. Manufacture of normal butyl *p*-aminobenzoate. 13,158. May 12. (Fr., 30,719.)

Wesson. Isobornyl-ester-camphene mixture. 13,562. May 17.

COMPLETE SPECIFICATIONS ACCEPTED.

5686 (1918). Pope, Gibson, and Thuillier. Production of β -dichlorethyl sulphide. (142,875.) May 27.

7802 (1918). Orton and Pope. Production of chlorpicrin. (142,878.) May 27.

9593 (1918). Pope. Production of aromatic arsenic compounds. (142,880.) May 27.

2657 (1919). Selden Co., and Selden. Purification of organic substances by sublimation. (142,902.) May 27.

3508 (1919). Darasse Frères, and Dupont. See VII.

4245 (1919). Mouneyrat. Preparing aromatic arsenic acids. (142,947.) May 27.

24,331 (1919). British and Foreign Chemical Producers (Rheinische Kampfer-Fabr.). Manufacture of para-cymene. (142,738.) May 19.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Forbes. Natural colour photography. 14,125. May 21.

Siemens u. Halske A.-G. Photographic plates, papers, and films. 13,351. May 14. (Ger., 3,717.)

XXII.—EXPLOSIVES; MATCHES.

APPLICATIONS.

De Wendel. Blasting-cartridges of liquid air. 12,941. May 10. (Ger., 30,516.)

Du Pont de Nemours and Co. Propellant powder, 13,811. May 19. (U.S., 23,819.)

COMPLETE SPECIFICATIONS ACCEPTED.

746 (1919). Rintoul, Weir, Lowndes, and Nobel's Explosives Co. Explosives. (142,898.) May 27.

32,690 (1919). Hedberg. Manufacture of explosives. (142,763.) May 19.

XXIII.—ANALYSIS.

APPLICATIONS.

Bawtree, and Hadfields, Ltd. Viscometer. 13,850. May 19.

Krogh and Pedersen. Recording analysing apparatus for automatic analysis of gases. 12,961. May 10.

I.—GENERAL; PLANT; MACHINERY.

"Settling" problems [; Continuous —]. W. A. Deane. Trans. Amer. Electrochem. Soc., 1920, 659—688. [Advance copy.]

THE introduction of the Dorr continuous thickener has resulted in greatly increased application of continuous settling methods. Settling may be divided into clarification, which is directly dependent on the area of the settling vessel and is independent of its height, and thickening, which depends on the volume of the thickening zone. The clarification capacity required may be calculated from the formula, $A = 4(F - D) \cdot (3R - Sp)$, where A represents unit thickener area in sq. ft. per ton of solids per 24 hrs., R the settling rate in ft. per hr., Sp the sp. gr. of the liquid, F the ratio of liquid to solids in the feed, and D the ratio of liquid to solids in the discharge. The thickening capacity is calculated from $V = 4T(G - Sp) \div 3G(S - Sp)$, where V represents the volume in cub. ft. required for thickening per ton of solids per 24 hrs., S the sp. gr. of thickened pulp, Sp the average sp. gr. of solution, G the average sp. gr. of solids in the pulp, and T the period of detention in hrs. The governing factor in clarification is the rate of settling of the finest particles. The content of particles in the colloidal condition may be reduced by modifying the previous mechanical treatment so as to reduce the amount of slimes produced, by modification of any previous chemical treatment in the direction of obtaining a coarser precipitate, and by a preliminary heat treatment of the material. Flocculation of colloidal particles may be brought about by the addition of electrolytes and/or colloids, by the electric current, and by biological action. The addition of a colloid together with an electrolyte that will flocculate it is sometimes useful, e.g., the addition of a little clay together with enough sulphuric acid to flocculate it to waste liquors containing silk gum. The passage of an electric current between electrodes immersed in the suspension caused no flocculation when carbon electrodes were used, but was effective when metal was substituted for the carbon. The growth of bacteria under certain conditions tends to cause clarification, as in the activated sludge sewage process. Raising the temperature in almost all cases improves the settling of the solids. Thickening starts from the bottom of the vessel and extends upwards to the zone where the particles just begin to touch one another. The slow action of the rake in the Dorr thickener tends to increase the density of the sludge by enabling the particles to pack more closely. As thickening proceeds the upward escape of the liquid is retarded but may be facilitated by mechanical means. Endosmose has been applied to thickening pulps, and although still in the experimental stage, offers considerable promise as a practical means of reducing the moisture content of substances, like clays, that are particularly difficult to filter. Flocculation and heating both help settling, but the former, unlike the latter, hinders thickening. A comprehensive bibliography of the subject is given.—W. H. C.

Extraction processes; Numerical relationship between cells and treatments in —. L. F. Hawley. J. Ind. Eng. Chem., 1920, 12, 493—496.

THE relationship between the number of treatments and the number of cells in terms of the time required may be represented by the following formula when the pumping of all the cells in action is done simultaneously $n = b(2c - t) + p(2c - t - 1)$, where n represents the time during which each cell stands idle, c the number of cells, and t the number of treatments in terms of the boiling period, b , and

the pumping period, p . In the case of continuous extraction the formula becomes $n = p(2c - t - 1)$, p here representing the time required for pumping the contents of one cell into the next. When each cell is pumped separately modified formulae are required. The formulae show that simultaneous pumping has little if any advantage over separate pumping. (See J., 1917, 1121.) C. A. M.

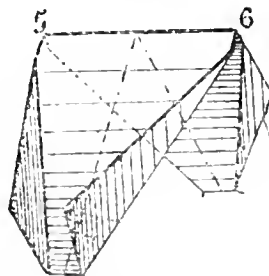
Liquefied gases; Construction of containers for the transport of —. F. Bannett, G. Rhein, and B. Kurze. Ann. Physik, 1920, 64, 113—114. Chem. Zentr., 1920, 91, 11, 662—663.

VESSELS of celluloid gave very favourable results with liquefied gases, but cannot be employed on account of the brittleness of the material at low temperatures. Transport containers have been made consisting of two concentric spherical brass vessels, the inner vessel being suspended from the neck of the outer. The neck of the inner vessel is made of manganese steel to lower the conductivity. The inter-wall space is reduced to a few mm. pressure by an oil pump, and the remainder of the gas is absorbed by cooled charcoal. In a 25-l. container of this type, the loss was 1.258 kg. per day. In the case of a nickel container with walls 1.5 mm. thick, soldered with a silver alloy, and evacuated with a rotary Gaede pump, the vessel being heated to remove occluded gases, the heat lost through the metal wall of the neck was specially important; the cool gases streaming from the neck did not influence this loss. Heat insulation can be effected by providing a glass tube, sealed to the metal on both sides by a platinum ring. In the case of large containers a long tube or coil of some badly conducting material may be provided. Good results cannot be obtained, however, with uncoated metallic vessels of iron or nickel on account of radiation losses. The best coating material is silver, which must be applied electrolytically. By silvering on both sides, the heat losses by radiation can be reduced by one-half. The most satisfactory vessels, when used for liquid air, showed a daily loss by evaporation of 4%.—E. H. R.

PATENTS.

Pyramidal surfaces for the purification of liquids, vapours, and gases; Combinations of —. P. H. A. Gaillet. E.P. 133,971, 17.10.19. Conv., 18.10.18.

RECTANGULAR plates bent as shown in the figure are arranged in layers in a tank or containing vessel, being mainly supported by the ridge 5, 6, the ele-



ments of each layer being at right angles to the elements in the layer above or below.—B. M. V.

Filling material for absorption and reaction towers. Pryn and Co. G.P. 317,167, 28.3.18. Addition to 317,166.

THE surface of the filling material described in the chief patent (J., 1920, 321 A) is increased without altering the length or width by bending in the end of the inner tube along a diameter of the tube.

—L. A. C.

Feeding material through a rotating cylinder whilst subjected to roasting, mixing, or similar operations; Means for —. A. Sonsthagen. E.P. 140,880, 17.2.19.

THE material is fed through a rotating drum by two sets of helical blades, one of which is fixed to the interior of the drum and the other to a central shaft. One set of blades causes the material to move forward and the other checks the movement so that the material is well mixed. The two sets of blades may be of the same pitch and rotated at different speeds, or the pitch may be different and the speed the same.—W. H. C.

Chemical treatment and grinding or/and mixing; Method of subjecting material to — and apparatus therefor, also ball-mill and combined grinding or/and mixing and vacuum drying apparatus. F. R. Ablett. E.P. 142,176, 27.1.19.

THE material is allowed to fall (with or without balls or pebbles to assist grinding) down a down-take tube where it is crushed by gravity and returned to the top of the same or another down-take by means of a screw conveyor. The undersize material may be removed by a screen or exhaust fan in the return tube, or the whole material may pass through a series of down- and up-takes, being subjected to vacuum drying or to chemical treatment, washing, and de-watering; in the last case the final screw conveyor is preferably made tapering to squeeze the water out. The whole apparatus may be made acid-proof and when a series of up- and down-takes are used the grinding balls may be returned through a separate race.—B. M. V.

Drying excreta, mucilaginous and other solid matters containing a large proportion of water; Apparatus for —. M. M. Lavarack (Rep. of E. C. Lavarack, dec.). E.P. 141,861, 11.3.19.

THE material is fed into a disintegrator from which it falls on to the surface of the upper of two flues, spaced apart within a casing, and is moved along the surface of the flue by scrapers attached to an endless chain. From the upper flue it drops on to the lower flue and is again moved in the opposite direction, and the dried material falls from the lower flue into a worm conveyor by which it is discharged. The drying is effected by the heat from a number of furnaces, built beneath the upper flue, into which the products of combustion pass first, travelling thence through the lower flue to the stack. Air for the furnaces enters the chamber and, together with the water and other vapours given off from the material, passes through the fire-bars so that any noxious fumes are destroyed.—W. H. C.

Drying or cooling coal, limestone and other substances or materials in granular form; Apparatus for —. A. E. Davis. E.P. 141,873, 11.9.19.

THE material is passed downwards through a chamber having double front and back walls, the hollow spaces being connected to each other by inverted V-shaped bars which extend through the material. The drying medium is introduced into the upper part of the space in the double front or back, so that the hot gas comes into contact with the wettest material first. If it is desired to pass the gas backwards and forwards in a zigzag manner through the material, the bars in the upper part are replaced by tubes.—W. H. C.

Desiccation; Method for —. A. W. Lissauer, W. L. Fleisher, and R. E. Keyes, Assrs. to American Drying Processes, Inc. U.S.P. 1,339,115, 4.5.20. Appl., 27.3.19.

AN air sheet is formed and caused to flow in a curved path having the shape of a parabola, and a current of an atomised substance intersects this

curved path in a direction parallel with the axial plane of the curved path.—A. G.

Dryer; Rotary —. A. O. Hurxthal, Assr. to The Philadelphia Textile Machinery Co. U.S.P. 1,339,260, 4.5.20. Appl., 1.12.19.

AIR is circulated by a fan through a chamber containing heating tubes, to a drying chamber which contains a rotating horizontal perforated cylinder. The cylinder contains curved blades which project inwards and which raise the material to be dried and then allow it to fall. A fixed curved plate surrounds the lower part of the cylinder to retain the material, and a partition is provided in the upper part of the drying chamber to cause the hot air to circulate through the cylinder back to the heating chamber.—W. F. F.

Drying machines; Method of preventing eaking and burning-on of material in —. L. Buszinsky and A. Liebbeit. G.P. 315,213, 28.5.18.

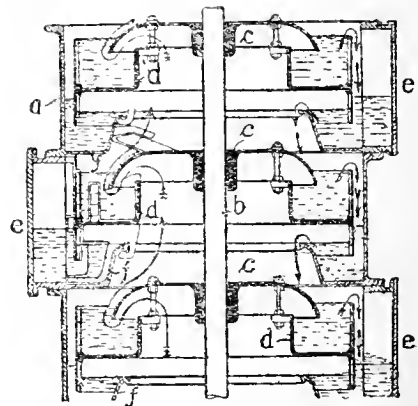
IN drying machines provided with revolving blades a water pipe is fixed along the blade shaft. By means of the heat of the dryer steam is generated in the pipe and directed on to the blades through nozzles, thus preventing eaking, and possible subsequent "burning" of the material.—W. J. W.

Still-heads. T. H. Durrans, and A. Boake, Roberts and Co., Ltd. E.P. 142,330, 30.5.19.

APPARATUS similar to that described in E.P. 19,999 of 1908 is constructed with a continuous, uninterrupted spiral having the convolutions of varying pitch, or it is formed of a number of sections bolted together, the spiral being of decreasing pitch from section to section upwards, but of the same pitch throughout each section.—W. H. C.

Distillation column. Zimmermann und Jansen, G.m.b.H. G.P. 316,491, 17.8.17.

A COLUMN particularly adapted for the distillation of liquids containing suspended matter, as any section can be readily cleaned, consists of a number



of annular receptacles, *f*, between which are annular vessels, *d*, attached to a central shaft, *b*, by discs, *c*. Each section is provided with a removable door, *e*, in the outer wall, *a*, and thus, by rotating the central shaft, any part of the interior is readily accessible.—L. A. C.

Filter-press frame. G. F. Miller. U.S.P. 1,337,251, 24.4.20. Appl., 17.6.19.

INSIDE the frame proper is placed a removable cloth-holding frame, and a channel for the material to be filtered is made all round the inside circumference of the frame proper, with radial passages leading from the channel through the cloth-holding frame.—B. M. V.

Filtering apparatus. J. M. Izquierdo. U.S.P. 1,337,517, 20.4.20. Appl., 10.12.18.

A NUMBER of pairs of sloping filtering surfaces are arranged in a casing, the surfaces of each pair sloping in the opposite direction relatively to each other and to the surfaces of the pair next below, so that the material to be filtered takes a zigzag course from top to bottom of the apparatus.

—B. M. V.

Filtering-belt. J. A. McCaskell. U.S.P. 1,338,232, 27.4.20. Appl., 21.6.18.

A FILTERING belt with an interior fluid space is provided with a filtrate collector associated with the belt and having independent fluid passages. Flexible conductors connect the fluid space of the belt to the fluid passages, and are wound and unwound during the travel of the belt. Discharge of the filtrate is controlled.—J. H. J.

Filter; Rotating-screen —. C. L. Peck, Assr. to The Dorr Co. U.S.P. 1,338,999, 4.5.20. Appl., 5.10.18.

A PERFORATED hollow cylinder is rotated in a tank containing the material. The material flows from the tank through the cylindrical surface of the screen, and from the interior of the screen through an axial opening at one end arranged directly opposite to a corresponding opening in the wall of the tank.—B. M. V.

Furnace gas-producer. L. M. Underwood. U.S.P. 1,337,298, 20.4.20. Appl., 10.3.19.

IN a kiln provided with a stationary furnace and gas-producer chamber, the latter communicates with the combustion chamber to which an independent air supply is maintained. The portable furnace grate consists of a series of hollow, tilting bars each provided with an end nozzle, into which steam and air are injected, and with a row of tuyères, along the top, through which these gases are passed into the fuel bed.—W. E. F. P.

Muffle furnaces; Method of heating —. W. Zieren. G.P. 317,888, 17.10.18.

TO prevent local overheating of a muffle, the furnace is so built that the hot gases enter through cavities between the outer wall and the top and bottom of the muffle; after leaving the muffle, the hot gases are conveyed away through flues, heated again by the furnace, and returned to the muffle.

—L. A. C.

Catalysers; Apparatus for making —. W. D. Richardson, Assr. to Swift and Co. U.S.P. 1,338,698, 4.5.20. Appl., 26.11.15.

A VERTICAL, cylindrical reduction chamber, provided with a heating coil, downwardly acting propeller agitators carried on a vertical shaft, and a perforated coil for introducing reducing gases, communicates with a baffle chamber where the gases and liquids are separated and which is connected to a vacuum pump.—W. H. C.

Catalyst and process for producing the same. N. Sulzberger. U.S.P. 1,338,709, 4.5.20. Appl., 28.5.15.

A BORATE of a metal possessing catalytic activity is reduced, sufficient of the borate radical being present to serve as an effective carrier of the catalytic metal after reduction.—W. H. C.

Emulsification; Method of and apparatus for —. C. E. Norcross. U.S.P. 1,338,996, 4.5.20. Appl., 12.11.19.

THE materials to be emulsified are delivered to the rotor of a centrifugal machine which is provided with an extremely narrow outlet passage, through

which the materials impinge against a wall which also is part of the rotor.—B. M. V.

Emulsions and suspensions; Thickening, drying, and effecting chemical reaction in —. G. A. Krause. G.P. 303,942, 7.6.16. Addn. to 297,388.

THE emulsion or suspension enters a vaporising chamber through atomisers and is sprayed in all directions in a horizontal plane against a stream of gas for drying or reacting with the particles.

—L. A. C.

Separating condensable vapours from air; Apparatus for —. J. S. Fries Sohn. G.P. 316,652, 21.1.11.

Air containing vapours of volatile liquids is led through a cooling tube containing an inner tube provided at intervals with projections which extend nearly to the outer tube. The latter is thus divided into compartments separated by annular nozzles formed by the projections and the wall of the outer tube. The cooling action is thereby improved.

—J. S. G. T.

Electrical precipitation; Device for — employing high-tension current. Siemens-Schuckertwerke, G.m.b.H. G.P. 316,790, 1.4.16.

HIGH-TENSION rectifying valves are installed in such manner that they can convey at least double the normal working current of the installation, and provide at least one and a half times the working tension, the excess in normal working being taken up by resistances.—J. S. G. T.

Electro-osmotic dehydration of vegetable, animal, and mineral material; Process and apparatus for —. Elektro-Osmose A.-G. (Graf Schwerin Ges.) G.P. 317,403, 13.12.17.

DURING the first stage of the process the volume of the dehydrating chamber is kept constant, and fresh material is introduced continuously, the charge being subjected to the pressure of the material being fed to the chamber or to external pressure. When the supply of material is interrupted, the volume of the dehydration chamber is diminished by causing the electrodes to approach one another, the movement of the electrodes being effected automatically and controlled by the pressure in the chamber.

—J. S. G. T.

Crushers. R. E. Trottier. E.P. 132,503, 25.8.19. Conv., 12.9.18.

Hot air; Means for producing —. H. A. M. Pederson. E.P. 136,160, 2.12.19. Conv., 18.1.19.

Raising water or other fluids by compressed air, through special reversing valve, automatically operated. E. J. H. Norman. E.P. 141,773, 17.12.18 and 13.5.19.

Centrifugal apparatus with planetary movement. W. Mauss. U.S.P. 1,339,272, 4.5.20. Appl., 4.9.19.

Crystallisation from solution; Effecting —. Norsk Hydro-Elektrisk Kvaestofaktieselskab. E.P. 131,207, 13.10.19. Conv., 23.10.18.

SEE U.S.P. 1,329,158 of 1920; J., 1920, 392 A.

Heat treatment of chemical compounds; Apparatus for — and method for operation thereof. F. X. Govers. U.S.P. 1,297,833, 18.3.19. Appl., 18.7.18.

SEE E.P. 129,962/18; J., 1920, 391 A.

See also pages (A) 440, *Liquid-heating furnace* (U.S.P. 1,335,774). 445, *Boiling indirectly* (U.S.P. 1,337,701). 448, *Flake material* (E.P. 141,942). 449, *Crystallisation* (G.P. 318,350). 456, *Nickel catalyst* (G.P. 318,177). 461, *Separating solids from liquids* (E.P. 128,944).

II.A.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal dust and fusain. F. S. Sinnatt, H. Stern, and F. Bayley. Bull. 5, Lancs. and Cheshire Coal Res. Assoc., 1920, 19 pp.

COAL dust produced during normal working in the pits by natural fracture of the coal may have properties and composition quite distinct from those of coal dust obtained by pulverising lump coal. Fusain (J., 1919, 452 A) is found in naturally-produced dusts in much greater quantity than in the mass of coal. It possesses little cohesive power, the coal therefore fracturing most readily along planes in which it occurs, exposing surfaces of coal covered with a layer of fusain, which quickly powders on exposure to the air. Dust from a dust collector attached to a belt picking room, and of which 95 by weight would pass through a sieve of 1-90th mesh, was found to contain about 49% of fusain. Fusain ignites with considerable ease at a low temperature, and continues to smoulder at a dull red heat without flame or odour, and may have a marked influence upon the tendency of a seam to produce gob fires.—W. P.

Sub-bituminous coal: Low-temperature distillation of —. H. K. Benson and R. E. Canfield. J. Ind. Eng. Chem., 1920, 12, 443-446.

In the distillation of sub-bituminous coal from Newcastle, Wash., which was unsuitable for commercial fuel, the maximum yield of tar oils was obtained at 350° C. About 3.5% of the coal was converted into crude oils composed of a mixture of tar oils and petroleum-like oils, whilst the yield of paraffin wax was about 5.3 lb. per ton of coal. The gas liberated up to 600° C. was small in quantity; it was relatively rich in illuminating hydrocarbons, but low in calorific value. The tar water yielded an appreciable amount of ammonia and a small amount of cyanides. A temperature of pronounced decomposition (between 350° and 450° C.) was observed in the destructive distillation of this coal corresponding to a sudden increase in the yields of hydrogen and methane and of the quantity of oil distilled. The residue left at 350° showed an increase of 22.8% in calorific value as compared with the coal as mined, and of 14.7% over the dry coal.—C. A. M.

Naphthenes: Occurrence of — in Galician crude oil. F. J. Skowronski. Metan, 1918, 61-67, 73-79, 86-91, 97-105, 109-118. Chem. Zentr., 1919, 90, IV., 585; 1920, 91, II., 655.

PETROLEUM from Tustanowic (sp. gr. 0.8766 at 25°/1° C., 1.34, distilling below 150° C., 29-12 below 300°, and 67.95% residue, hard asphalt content 1.59%, soft asphalt 4.17%, wax 8.37%) was fractionated, the fractions were purified, freed from unsaturated hydrocarbons by treatment with cold fuming sulphuric acid, and tested for naphthenes by heating with iodine to oxidise the naphthenes, and then brominating in the presence of ferric chloride. Galician oil is midway between American and Russian oils in its content of naphthenes. There have been identified definitely hexanaphthene (cyclohexane), heptanaphthene (methylcyclohexane), octonaphthenes (1,2-, 1,3- and 1,4-dimethylcyclohexanes), nononaphthene (1,2,4-trimethylcyclohexane), α - and β -decanaphthenes, and also hexahydro-*p*-xylene (not hitherto observed in petroleum). The decanaphthenes appear to be dimethylethylcyclohexanes).—A. E. D.

Water in petroleum and other organic emulsions: Determination of —. E. W. Dean and D. D. Stark. J. Ind. Eng. Chem., 1920, 12, 486-490.

DISTILLATION in the presence of an immiscible solvent is the most trustworthy method of determin-

ing water in petroleum or other organic emulsions. A 500 c.c. round-bottomed distillation flask is connected to a "distilling tube receiver," made by fusing a side tube to a tapered graduated test tube. This side tube passes through the stopper of the flask, whilst the lower end of a rellux condenser is fitted into the cork of the test tube. A commercial naphtha or a mixture of commercial xylene with 20% of commercial benzene gives good results as immiscible solvent. The emulsion (100 c.c.) and solvent (100 c.c.) are distilled at the rate of 2 to 4 drops per sec., until all water in the flask, distillation tube, and condenser has disappeared (about 1 hour) and the volume of water collected in the graduated tube is read. For the determination of less than 0.5% of water in an emulsion 200 c.c. of both emulsion and solvent are used. The method gives good results with coal-tar emulsions, using the xylene-benzene solvent.—C. A. M.

Sulphur in [petroleum] oils; New method of determining —. C. E. Waters. J. Ind. Eng. Chem., 1920, 12, 482-485.

FROM 0.5 to 1.5 g. of the oil is treated with 5 c.c. of concentrated nitric acid, saturated with bromine, and the crucible covered with a watch glass and heated for about 30 mins. on a steam bath, and finally for 2 to 3 hours in direct contact with the steam. From 10 to 12 g. of anhydrous sodium carbonate is then introduced beneath the crucible cover in successive small portions, and the resulting pasty mass dried on the steam bath at 100° C., and ignited over a low flame. It is then digested with about 150 c.c. of water, the aqueous solution filtered, the filtrate acidified with hydrochloric acid (about 2 c.c. in excess), and the sulphate precipitated as barium sulphate. A blank test should be made with the reagents. For heavy and vulcanised oils the method gives results as accurate as those obtained with the bomb calorimeter.—C. A. M.

Mineral oil refining; Recovery of waste acids from —. E. A. Kolbe. Petroleum, 1919, 14, 837-847. Chem. Zentr., 1919, 90, IV., 197.

ABOUT 90% of the sulphuric acid in the waste acids from lubricating oil refining may be recovered in the form of sulphur dioxide by heating the waste acid; a lower temperature is required for the decomposition, less pitch is formed, and the sulphur dioxide contains less sulphur trioxide when the waste acid from lubricating oil refining is mixed, before heating, with waste acid from petroleum (burning) oil refining.—W. P. S.

Paraffin wax; New sweating process for —. A. Székely. Petroleum, 1919, 15, 109-111. Chem. Zentr., 1920, 91, II., 23-24.

CRUDE melted paraffin wax is introduced into a vessel, partly immersed in water, constructed with perforated walls and containing perforated tubes. Those parts above the water-level are protected by solid walls and tubes until the wax has solidified sufficiently not to pass through the perforations. After removal of the protective coverings the cake may be subjected to moist or dry sweating, or a combination of both. Sharp fractionation and the production of a valuable product from inferior material are effected.—C. A. M.

Montan wax; Resinous constituents of —. J. Marcusson. Braunkohle, 1919, 17, 245-247. Chem. Zentr., 1920, 91, II., 417-418.

THE products hitherto described as montan resin contain large quantities of wax; e.g., 100 g. of the resin obtained by the technical method of extraction with toluene, when dissolved in 300 c.c. of ether and treated with an equal volume of 96% alcohol, gave 56 g. of precipitated wax and a further quantity on cooling to -20° C. From the mother liquor two resin fractions of 12.5 and 19.8 g.

respectively were obtained by partial and complete removal of the solvent. The second of these fractions was completely soluble in 96% alcohol, whilst the first was only slightly soluble even on boiling. The behaviour of these resins towards sulphuric acid and nitric acid was compared with that of lignite extracted with benzene. It is concluded that the former are probably the remains of plant resins originally present in the wood. Apart from quantitative differences lignite resembles pit coal in its behaviour to sulphuric and nitric acids. It contains besides mineral constituents, waxes, plant resins, nitrogenous bases, and humic acids, two main constituents, *viz.*, saturated polycyclic compounds containing oxygen in "bridge formation" and resembling in character the chief constituent of pit coal, and also more readily decomposable substances which can be converted into soluble compounds by sulphuric and nitric acids. The transformation of lignite into pit coal may consist in the decomposition of the last-mentioned compounds, and the conversion of the waxes and plant resins into hydrocarbons, which by polymerisation and oxidation yield asphalt-like products.

—J. H. L.

Fatty acids from petroleum. Ubbelohde and Eisenstein. See XII.

PATENTS.

Powdered fuel; Fluid-pressure apparatus for burning — P. A. Leonard, M. F. Maloney, and E. Friedrich. U.S.P. 1,338,451, 27.9.20. Appl. 16.8.19.

A jet of mixed fuel and air is blown into a furnace, and a jet of air is delivered under pressure in line with, and unobstructed from direct impingement against, the first jet. A volume of air under pressure is also introduced at an angle to the common axial line of the first two jets.—A. G.

Coke ovens. E. Hurez. E.P. 123,099, 6.2.19. Conv., 26.1.18.

In regenerative ovens the circulation of the gases is arranged so that the heating surfaces of the walls of the furnace are all continuously heated by gases in active combustion. The circuits of air and gas are arranged so as to obtain automatically as uniform a distribution as possible in each of the compartments of the furnace, positive control of the gas and air supplies being provided for. The recuperators are formed of specially constructed earthenware blocks, arranged in such a manner that the air or gases to be heated as well as the burned gases traverse them always in the same direction and without the necessity of reversal.

—A. G.

Coke-ovens. G. Hinselmann. G.P. 319,216, 19.10.17.

In a coke-oven plant steam is led through the chambers in which coke formation is almost complete, and the mixture of steam and water-gas obtained is led without fall of temperature into the remaining chambers in which coking is not so far advanced. Residual steam is converted therein to water-gas, and the passage of the heated gases over the coal during the early stages of coking shortens the time of the process and increases the yield of by-products and combustible gas.—L. A. C.

Coke for use in blast-furnaces; Saturating — with chloride solutions. A. Riedel. G.P. 319,550, 4.1.16.

INCANDESCENT coke from coke-ovens is quenched with chloride solutions such as the waste liquors of the potash and soda industries, which are employed in such proportions that the quantity of chloride absorbed by the coke is equivalent to its nitrogen content. The nitrogen is converted to ammonium chloride during combustion of the coke, and is recovered as such.—L. A. C.

Gasification of carboniferous substances; Process and apparatus for — H. Strache. E.P. 117,083, 22.6.18. Conv., 28.6.16.

GASIFICATION is effected in a producer surmounted by a distillation chamber into which the fuel is fed intermittently from a hopper. The producer-gas is partly burnt in flues surrounding the distillation chamber, and the hot gases pass on to a steam superheater, where combustion is completed. The superheated steam is supplied in part to the top of the producer near the junction with the distillation chamber and the remainder through an inlet at the bottom of the producer. Alternatively the whole of the steam may be supplied to the top of the producer, in which case water-gas is withdrawn through an outlet at the bottom. The distillation gases are withdrawn from the top of the distillation chamber. The steam is superheated to a temperature below 800° C. if a high yield of ammonia is desired, or above 800° C. to obtain a high yield of gas.

Gas producing plant. E. H. Crush. E.P. 111,771, 17.12.18.

In gas producers where part of the sensible heat of the gas is used for the generation of steam, to prevent accumulation of dust on the water tubes the gas off-take is situated in the upper part of the chamber, and the water tubes of the steam generator are inclined or curved inwards and upwards. Baffles are arranged so that the producer gas enters radially at several points and changes its path of travel to the off-take.—A. G.

Gas; Method of manufacturing — from coal or like carbonaceous material. C. E. Holt and T. Walker. E.P. 141,828, 4.2.19.

The finely-divided coal is dried by passage through a jacketed hopper, and steam is supplied at comparatively low pressure, so as to flow gently into the heat zone of the retort to meet the falling coal dust.—A. G.

Gas; Method of producing — O. F. Bean. U.S.P. 1,337,637, 20.4.20. Appl., 5.8.19.

SUPERHEATED steam is passed over hot iron to liberate hydrogen. Oil is vaporised, the vapour is raised to the same temperature as the hydrogen, and then added to it. The mixture is passed through a conduit, where its temperature is raised to the gasification point.—J. H. J.

Gaseous fuel; Process of manufacturing — J. R. Rose. U.S.P. 1,339,225, 4.5.20. Appl., 25.1.18.

HYDROCARBON vapours are injected into a space heated by an electric arc, and are thereby dissociated into solid carbon and a gas containing a preponderating amount of hydrogen.—A. G.

Gas; Apparatus for washing — T. H. Hack and H. F. Boughton. E.P. 142,203, 5.2.19.

FLOW tubes for the washing liquid are disposed in circular formation in plan and vertical in elevation, and pressure tubes are so arranged that the gas under pressure issuing therefrom blows freely over and across the mouths of the flow tubes. Similar circles of tubes are fixed at a higher elevation and arranged to intervene in plan. An annular spray of washing liquid is thus produced. In another form of apparatus an inner and outer annular orifice arranged concentrically are formed by means of an adjustable or fixed valve which does not cover the outer orifice. Gas under pressure issuing from one orifice blows freely across the other which induces or is supplied with liquor, and an annular spray is thereby produced. A. G.

Tar extractor apparatus and method. J. Becker, Assr. to The Koppers Co. U.S.P. 1,337,984, 20.4.20. Appl., 2.12.18.

PERFORATED baffle-plates, partially immersed in the

accumulating tar, are used to effect deposition of the tar from the gas. The level of the tar in the chamber is controlled automatically in response to changes in the pressure differential of the gas flow through the baffles.—A. G.

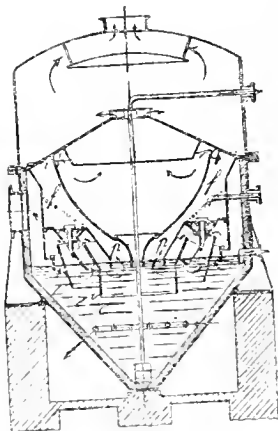
Gas washing apparatus. J. S. Kennedy. U.S.P. 1,339,112, 4.5.20. Appl., 15.1.19.

A CASING is divided into a number of separate unobstructed treating chambers, including a washing chamber wherein the gases are cleansed and a chamber wherein the moisture is precipitated. The flowing gas is subdivided by a horizontal grating in the path of the gases between the washing and precipitation chambers.—A. G.

Washing and absorbing gases; Apparatus for —. W. Steinmann. G.P. 316,594, 20.8.18.

GAS enters the cylindrical washing chamber (see fig.), and is forced to pass through the liquid in *c* in the direction indicated by the arrows, by means of a number of annular rings so arranged that the spaces through which the gas leaves are narrower than those through which it enters the liquid. Any liquid suspended in the gas is removed as it passes upwards to the exit through *p, f, g*. The liquid container, *c*, may be fitted with a heating or cooling coil.

—L. A. C.



Spent oxide; Revivifying —. W. J. Dibdin. E.P. 141,172, 15.3.19.

SPENT oxide on trays with fine wire gauze bottoms arranged in a chamber is heated by a current of steam superheated to about 500° C. to remove the sulphur; the trays are then transferred to a second chamber in which the iron is re-oxidised, and organic matter and residual sulphur are removed by a current of air regulated to avoid too high a temperature. The steam passes into a condensing chamber in which the greater part of the sulphur separates.—L. A. C.

Hydrocarbons and other substances; Decomposition of — in the liquid and/or vapour phase. W. Mann. E.P. 142,206, 6.2.19.

Porous septa are introduced between the decomposition chamber and the condenser, whereby products of lower molecular weight are separated by virtue of their superior velocity of diffusion. —A. E. D.

Distillation of heavy hydrocarbons of petroleum, lignite tar, and coal tar; Distilling boiler for —. L. Steinschneider. E.P. 142,376, 14.8.19.

To avoid the rapid destruction of coking stills an ordinary still is equipped with a series of retorts connected with the lowest part of the still. The coking process takes place in the retorts which are provided with cleaning openings and can be disconnected independently from the still.—A. E. D.

Cracked oils; Refining —. E. H. Leslie and C. Barbre, Assrs. to General Petroleum Corp. U.S.P. 1,337,523, 20.4.20. Appl., 13.9.17.

The oil is heated in the presence of a porous catalyst, then filtered, and distilled to separate any

resinous matter produced in the preliminary heating.—A. E. D.

[Mineral] oil; Apparatus for refining —. Process of and apparatus for refining liquid hydrocarbons. System for cracking mineral oil. Apparatus for cracking hydrocarbons. Apparatus for refining oil. Apparatus for refining mineral oils. Liquid-heating furnace. F. E. Wellman, Assr. to Kansas City Gasoline Co. U.S.P. (A) 1,335,767, (B) 1,335,768, (C) 1,335,769, (D) 1,335,771, (E) 1,335,772, (F) 1,335,773, (G) 1,335,774, 6.4.20. Appl., (A) 15.9.15, (B) 30.12.15, (C) 7.10.16, (D) 15.1.17, (E) 12.5.17, (F) 21.1.18, (G) 9.4.18.

(A) OIL, heated under relatively low pressure in a preheater, is pumped into a superheater and maintained under pressure and at a relatively high temperature (B) Oil vapour is compressed and introduced into a retort heated to a temperature sufficient to crack the oil. The vapour produced is withdrawn near the bottom of the retort and passed upwards through a separate coil wherein different products are recovered by fractional condensation. (C) Oil is heated in a cracking coil jacketed with a lagged steam pipe. Superheated steam is simultaneously fed into the coil in a whirling spray. (D) A cracking still is provided with valved charging and discharging ports. An automatic pressure-operated relay valve operates the discharge valve at any desired pressure. (E) A tubular retort has inlet and discharge pipes at opposite ends, the discharge pipe being at the bottom of the retort. Charges of oil are fed intermittently into the retort, and provision is made for scouring the retort in the intervals between discharging and charging. (F) A cracking coil of large diameter is provided with feed and discharge pipes of smaller diameter. A pressure valve at the discharge end of the cracking coil is connected to a cooling coil of the same diameter as the cracking coil, and a safety discharge valve at the feed end of the cracking coil is connected to a cooling coil of the same diameter as the feed pipe. (G) A liquid-heating furnace is composed of horizontal layers of parallel pipes. A number of combustion chambers and flues are provided, and means for controlling them independently. An even longitudinal distribution of heat is thereby achieved.—A. E. D.

Hydrocarbon oils; System for treating —. F. E. Wellman and F. H. Sibley, Assrs. to The Kansas City Gasoline Co. U.S.P. 1,335,770, 6.4.20. Appl., 19.10.16.

OIL is cracked in a still under a suitable pressure. The still is heated with steam superheated to the desired temperature.—A. E. D.

Motor fuel; Composite —. Method of distilling. A. Hayes, Assr. to A. F. Ray. U.S.P. 1,338,983 and 1,338,984, 4.5.20. Appl., 19.9.19.

ALCOHOL-CONTAINING material is distilled and ether is added to the vapour during condensation to obtain a motor fuel containing alcohol, ether, and water, the amount of water present being greater than that of ether.—A. E. D.

Mineral oils; Substitute for — [from tar oils] for use in switches and transformers. Tetralin G.m.b.H. G.P. 299,691, 7.2.17.

SULPHUR compounds, fatty acids, and unsaturated compounds are removed from tar oils, yielding a product suitable for use in electric switches and transformers, by heating the crude or distilled oils with a finely divided or readily fusible metal, metal hydride, or other metallic compound in which the metal is only loosely combined with a non-acidic residue; the process may be supplemented by catalytic hydrogenation of the oil, e.g., by introducing hydrogen under pressure during the above treat-

ment. Suitable metals or metal compounds are sodium, potassium, copper, iron, finely divided nickel, calcium hydride, sodamide, calcium carbide, or the like, and other finely divided or porous materials, such as fuller's earth, and a solvent or diluent may also be added. Lignite tar fuel oil can be purified sufficiently for lubricating machinery by agitation above 100° C. with metallic sodium or potassium.—L. A. C.

Lubricating oils; Production of — from coal tar oils. H. Klever. G.P. (A) 301,775, 19.2.15, and (B) 301,777, 8.4.15. Additions to 301,774 (J., 1920, 327 A).

(A) THE tar oils may be heated under pressure at temperatures up to 100° C., or at atmospheric pressure under a reflux condenser, and preferably in absence of air. The highly viscous oils obtained may be used for thickening less viscous tar oils. Catalysts such as iron, copper, or other metallic powders, may be used. (B) Bases or phenols or both together may be added, or they may first be heated by themselves and the viscous products thus obtained may be used for thickening other tar or pitch oils. Alkalis and metal hydroxides have been successfully used as catalysts.

—J. H. L.

Washing coke and like materials; Apparatus for —. A. L. Jennings. E.P. 142,319, 23.5.19.

Coke-oven ironwork. L. Wilputte. E.P. 127,551, 7.2.19. Conv., 27.5.18.

SEE U.S.P. 1,320,132 of 1919; J., 1920, 5 A.

Drying coal. E.P. 141,873. See I.

Furnace gas-producer. U.S.P. 1,337,298. See I.

Nitric oxide in gas engines. G.P. 316,253. See VII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Wood charcoal; Activation of — by heat treatment. J. C. Philip, S. Dunnill, and O. Workman. Chem. Soc. Trans., 1920, 117, 362–369. (Cf. J., 1920, 363 A.)

THE main factor in determining the degree of activation, or increase of absorptive power, of wood charcoal resulting from prolonged heating, is not the duration of such heating, but rather the alteration of bulk density of the charcoal as a result of oxidation during the heat treatment by adsorbed oxygen, which causes an opening up of the capillaries, and a large increase of effective surface. The activated charcoal shows an increased absorptive power for colouring matters as well as for gases. For example, a sample of Japanese ash charcoal which only removed 0.6% of methylene blue from aqueous solution removed 60.3% after 19 hours' heat treatment at 830° C., considerably surpassing the activity of ordinary animal charcoal. It seems improbable, therefore, that the activity of animal charcoal can be correctly correlated with its nitrogen content, nor does the absorptive power depend on any specific inorganic constituents in the ash. The charcoal must be heated at a temperature not lower than 800° C. for any rapid improvement in absorptive power to take place. Further, whilst charcoals from light woods, such as willow and poplar, are activated more rapidly, they are ultimately surpassed in activity by charcoals derived from denser woods such as box.—G. F. M.

Rice chaff; Distillation of — to obtain acetic acid and furfural. F. Scurti and C. E. Zay. Staz. Sper. Agrar. Ital., 1919, 52, 278–290. Chem. Zentr., 1919, 90, IV., 1105.

Rice chaff contained: Water, 9.0; fat, 0.5; crude

proteins, 3.5; cellulose, 42.0; ash, 18.0; and N-free extractives, 27.0% (including 17% pentosans). To obtain acetic acid and furfural simultaneously it is best to distil 1 pt. of the chaff with 10 pts. of 30% H₂SO₄, and to keep the acid concentration of the liquid constant by adding water until 3 pts. of distillate have been obtained. The concentration of the acid in the distillation vessel is then raised to 50% by distilling a further 4 pts. The distillate will contain the whole of the furfural formed from the pentosans. An additional 3 pts. is then distilled to obtain the acetic acid. The first fraction is neutralised with sodium carbonate, the furfural distilled and purified by repeated distillation with sodium chloride. The neutral solution left after the distillation and the neutralised second fraction contain sodium acetate. 1 kg. of chaff yields 40 g. of furfural and 110 g. of sodium acetate.—C. A. M.

PATENTS.

Coal, carbonaceous material, oil shales, and the like; Distillation, carbonisation, or gasification of —. J. E. Christopher. E.P. 142,163, 30.12.18.

A RETORT or chamber has a main external heating flue and two auxiliary chambers, flues, or regenerative sections, or two series thereof connected thereto and controlled by dampers and the like, the arrangement being such that heating gas, with air if necessary, is admitted to the main external flue, the products of combustion passing alternately into one or other of the connected auxiliary chambers, flues, or regenerative sections or series thereof according to the setting of the dampers or the like, and thus further heating the retort or chamber.—A. G.

Coal; Process for the distillation of —. T. Limberg. G.P. 303,951, 15.10.16.

THE process is specially adapted for the treatment of coals rich in water and bituminous matter, such as bituminous shale and peat, for the purpose of obtaining a light tar with a high content of aliphatic hydrocarbons. The material is heated in a specially constructed oven at reduced pressure and at a temperature somewhat below dull redness, and the products of distillation are drawn off along the whole length of the oven into a series of separate vessels. The material may be arranged in layers in the oven and draw-off and internal heating devices provided between the layers.—B. V. S.

Wood distillation; Process for —. C. T. Clark, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,337,342, 20.4.20. Appl., 11.8.17.

WOOD, which has been cut transversely to the grain into plaques the dimension of which parallel to the grain is less than a dimension perpendicular to the grain, is heated, introduced into a retort, and then subjected to destructive distillation.—A. de W.

Wood being distilled; Holder for —. A. C. Badger, Assr. to E. B. Badger and Sons Co. U.S.P. 1,338,667, 1.5.20. Appl., 26.7.16.

A HOLDER capable of introduction and withdrawal from the retort, has separate compartments for the wood to be distilled and spaces between the compartments open above and below to permit of the escape of the products of distillation.—A. G.

Wood-tar and pyroligneous acid; Method of separating —. W. E. Clifford. U.S.P. 1,339,022, 4.5.20. Appl., 25.6.18.

THE liquor is maintained in a heated state and agitated by aeration, afterwards being allowed to settle into separate layers.—A. G.

Gas mantles; Manufacture of — from cellulose. Deutsche Gasglühlicht A.-G. (Auerger.). G.P. 312,577, 27.2.18.

CELLULOSE threads are converted partially into

hydrocellulose; the mantles made from these threads have properties similar to those of ramie and artificial silk mantles.—W. P. S.

Cellulose waste lyes. G.P. 301,624. See V.

Photometers. E.P. 141,825. See XXIII.

III.—TAR AND TAR PRODUCTS.

Water in petroleum etc. Dean and Stark. See IIA.

Chlorobenzenes. Bourion and Courtois. See XX.

Halogen in organic substances. Van Winkle and Smith. See XXIII.

PATENTS.

Lower boiling hydrocarbon [toluene]; Production of — from higher boiling hydrocarbon [xylene]. A. E. Houlehan, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,337,317, 20.4.20. Appl., 25.5.17.

XYLENE is subjected to the action of a catalyst whereby toluene is produced. The toluene is distilled off under vacuum as it is formed.—A. E. D.

Colourless organic compounds; Manufacture of —. South Metropolitan Gas Co., and H. Stanier. E.P. 141,440, 18.1.19.

DISCOLORATION in aromatic amino- or hydroxy-compounds, such as β -naphthol, benzidine, sulphanilic acid, or naphthionic acid is removed by treatment of a solution or suspension with a small proportion of a hydrosulphite or of a sulphonylate; similarly discoloration may be prevented, e.g. in the product obtained on heating a sulphonic acid with caustic soda solution by the addition of a small proportion of hydrosulphite or sulphonylate to the reagents.—D. F. T.

Decahydronaphthalene; Purification of —. Tetralin Ges. G.P. 310,781, 29.9.17.

THE crude product obtained by the incomplete hydrogenation of naphthalene is treated with liquid sulphur dioxide, which readily dissolves naphthalene and its lower hydro-derivatives, whereas decahydronaphthalene is almost insoluble in this solvent.

Chlorinated nitronaphthalenes; Manufacture of —. O. Matter. G.P. 317,755, 12.2.18.

CHLORINE addition products of naphthalene are treated with nitric acid at low temperatures, and the products are heated with water or a compound capable of combining with an acid. Nitronaphthalene dichloride, prepared by the action of nitric acid (sp. gr. 1.5) on naphthalene dichloride, on heating with water or milk of lime yields 1,8-chloronitronaphthalene, m.p. 93°–94° C.; this on reduction with stannous chloride and hydrochloric acid forms 1,8-chloronaphthylamine, m.p. 88°–89° C. Naphthalene tetrachloride on treatment with nitric acid (sp. gr. 1.5) yields a resinous nitro-compound, which by heating with methyl alcohol and sodium hydroxide, or with benzene, methyl alcohol, and sodium carbonate to 140°–150° C., is converted into 5,8-dichloro-1-nitronaphthalene, m.p. 94° C.; this on reduction with tin and hydrochloric acid forms 5,8-dichloro-1-naphthylamine, m.p. 104°–105° C.—L. A. C.

Chlorinated derivatives of naphthalene of waxy consistency; Production of —. Chem. Fabr. Griesheim-Elektron. G.P. 319,253, 21.10.16.

NAPHTHALENE is treated with chlorine at 100°–170° C. in the absence of a halogen carrier until the reaction product contains 50–57% Cl. This pro-

duct, containing approximately a tetra-atomic proportion of chlorine, possesses an advantage over the more highly chlorinated derivatives in that it is non-crystalline and free from brittleness. A softer wax may be obtained by checking the chlorination when the reaction product has attained a solidifying point of 118°–120° C.; for a harder product the solidifying point is allowed to rise to 126°–128° C. The products have sp. gr. 1.55–1.65.—D. F. T.

2-Aminoanthraquinone; Process of making —. M. Fischer, Assr. to J. R. Geigy S.A. U.S.P. 1,338,363–4, 27.4.20. Appl., 16.10.19.

SEE E.P. 127,223 of 1919; J., 1919, 529 A.

Distilling tar etc. E.P. 142,376. See IIA.

Substitute for mineral oils. G.P. 299,691. See IIA.

Lubricating oils. G.P. 301,775 and 301,777. See IIA.

IV.—COLOURING MATTERS AND DYES.

Indigotin; Continuous scheme for preparation of — from phthalimide. P. Holland. Chem. News, 1920, 120, 208–209.

ANTHRANILIC acid was prepared by slowly adding 10 c.c. of sodium hypochlorite solution (5.1% available chlorine) to a solution of 1 g. of recrystallised phthalimide and 2 g. of pure sodium hydroxide in 10 c.c. of water; after standing for 15 mins. 0.1 g. of sodium bisulphite was added, the solution was partially neutralised with sulphuric acid, and then faintly acidified with acetic acid. Anthranilic acid was converted to *o*-carboxyphenylaminoacetonitrile by adding an acidified solution of 1.2 g. of sodium cyanide and 2 c.c. of 37–38% formaldehyde to 1 g. of anthranilic acid dissolved in sufficient sodium hydroxide to give a faintly alkaline solution. After standing for 2 hrs. the solution was diluted with 200 c.c. of a cold saturated solution of the nitrile, filtered, washed with more saturated solution, and dried. A yield of 117.1% of nitrile from phthalimide was obtained. Indigotin was prepared from phthalimide without isolation of intermediates by drying the solution obtained in the preparation of the nitrile together with 10 g. of sodium hydroxide, heating the product covered with paraffin wax in a nickel crucible to 270°–275° C., and subsequently converting the sodium indoxylate in the melt to indigotin in the usual manner. Sodium phenylglycine-*o*-carboxylate was prepared from phthalimide by drying the solution of sodium anthranilate and sodium sulphate, adding the requisite weight of sodium monochloroacetate, and warming to 30° C., keeping the mass slightly alkaline; the sodium hydroxide fusion was then carried out as before.—L. A. C.

Anthocyanin colouring matters of the beet-red group; Occurrence and reactions of —. F. Kryz. Oesterr. Chem.-Zeit., 1920, 23, 55–56.

MEMBERS of the beet-red group of anthocyanins occur in the skins of fuchsia and cactus berries and in the petals of scarlet cactus blooms. Fuchsia anthocyanin gives a carmine solution in acidified acetone or amyl alcohol, but is insoluble in ether, chloroform, and carbon bisulphide. Cactus bloom anthocyanin is also insoluble in these media, but soluble in water, methyl, ethyl, or acidified amyl alcohol, and in glycerin. The colouring matter from red beet is soluble in cold acetone and similar to the others in its behaviour towards reagents. Wing cactus blooms apparently contain two anthocyanins, since the reddish-yellow extract obtained with hot acetone shows slight differences from the foregoing compounds, whilst the aqueous solution of the residue exhibits close agreement. (Cf. J.C.S., July, 1920.)—J. K.

Activation of hydrogen peroxide. Curtis. See VII.

Dyes in butter. Gilmour. See XIXa.

Isocyanine dyes. Mills and Pope. See XXI.

PATENTS.

Disazo dyestuffs; Manufacture of mordant-dyeing — and their application in dyeing and printing. Manufacture of mono-azo dyestuffs. O. Imray. From Society of Chemical Industry in Basle. E.P. (A) 111,101 and (B) 111,613, 10.12.18.

(A) MORDANT-DYEING disazo dyestuffs are prepared by diazotising the mono-azo dyestuffs described under (B) and coupling with an aminonaphtholsulphonic acid, an acetylammonaphtholsulphonic acid, or a pyrazolone containing a carboxyl group or a carboxyl group and a hydroxyl group in the aryl residue. The products give, after chroming, fast yellow to red or greenish-blue tints on wool and similar tints when chrome-printed on cotton. (B) Yellow to orange monoazo dyes applicable in wool dyeing and in chrome printing on cotton are made by combining diazo compounds with methyl- α -sulphonic acids of the type $R.NH.CH_2.SO_3H$, where R is an aromatic radical, and subsequently eliminating the N-methyl- α -sulphonic acid group.

—A. J. H.

o-Hydroxyazo-dyestuffs; Substantive —. E. Anderwert and H. Fritzsche, Assrs. to Soc. Chem. Ind. in Basle. U.S.P. 1,335,111, 27.4.20. Appl. 11.6.19.

AZO-DYESTUFFS dyeing unmordanted cotton direct tints sensitive to alkalis and acids and giving by treatment with coppering agents on the fibre cleared to blue-violet tints fast to alkalis, acids, and light, are prepared by coupling the diazo derivative of an *o*-amino-hydroxyarylsulphonamide of the formula (1)OH.(2)NH₂.R.SO₂NHX (where R is an aryl radical which may be further substituted, and X is hydrogen or an aryl radical) with a 2,5,7-aminonaphtholsulphonic acid compound in which a hydrogen atom of the amino group is substituted by one of the groups C₆H₅—, C₆H₄CO—, C₆H₄.(3)NH₂.(4)CO—, C₆H₄.(7)SO₃H.(5)OH.(2)NH₂CO—, —C₆H₄.(3)SO₃H.(5)OH, or C₆H₄.(7)SO₃H.(5)OH.(2)NH₂CO(1)—C₆H₄.(3)NH₂CO—(3)NH₂.C₆H₄—(1)CO—.

—L. A. C.

Black [sulphur] colouring material; Preparation of a —. H. R. Vidal. E.P. 141,759, 10.1.17.

A MIXTURE of 40 kg. of 1,2,4-dinitrophenol, 100 kg. of crystalline sodium sulphide, 20 kg. of commercial cresol, 25 kg. of sulphur, and 100 l. of water is boiled under a reflux condenser for 4–5 hrs., and the colouring matter is precipitated from the product by air or acid. The dye is soluble in sodium sulphide, and dyes cotton a direct black which does not bronze, has no grayish reflection, and is fast to chlorine.—L. A. C.

Triarylmethane dyestuffs suitable for after-chroming; Production of —. Farb-fabr. vorm. F. Bayer und Co. G.P. 318,956, 14.10.11.

THE products obtained by the condensation of sulphonated dyes, derived from aromatic aldehydes and hydroxycarboxylic acids, with aromatic amines (G.P. 287,003; J., 1916, 171) give feeble, dull and unequal shades in cotton printing, but on further sulphonation superior dyestuffs are produced of much more than double the intensity.

—D. F. T.

Dye recovery. E. J. Wall, Assr. to Kalms, Comstock and Wescott, Inc. U.S.P. 1,337,673, 20.4.20. Appl. 18.5.17.

FIBROUS cellulose material is immersed in a dye

solution to absorb the dye, which is subsequently extracted from the material by a suitable solvent.

—L. A. C.

Ind. E.P. 111,631. See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Retting of textile plants in Italy. D. Carbone. Bull. Agric. Intell., 1919, 10, 177–179. (See J., 1918, 25 A.)

THE retting of hemp submerged in water is effected by a slender bacillus, *B. felsineus*. This could never be obtained quite free from *Amphibacter*, and it is possible that the latter may be a special form of the micro-organism. This bacillus sometimes occurs in chains, forms elongated spores like those of *Clostridium*, and produces orange-coloured colonies on agar must. When grown alone it is strictly anaerobic, but it can also live aerobically in symbiosis with other organisms, notably *Saccharomyces ellipsoideus*. Its retting action on textile plants differs from that of aerobic bacteria of the *B. asteropus* group, for it separates the bark in wide strips, whereas after the action of the latter the bark is still attached to the fibres. *B. felsineus* is found on unretted hemp, on the surface covering of retting pits, and in the mud of water in communication with the pits. Its optimum temperature is 37° C. It has given good results in the retting of ramie, *Furcraea*, and *Sansevieria*, and also of broom, nettle, mallow, etc.—C. A. M.

Best fibres; Retting of —. W. P. Kraus, Z. angew. Chem., 1920, 33, 102–103. (Cf. J., 1919, 169 A, 530 A; 1920, 11 A.)

FURTHER work is described dealing with the retting of fibrous materials by steeping in a 0.5–1.0% solution of sodium bicarbonate for 3–5 days. The addition of a small quantity of sodium sulphite to the retting liquor greatly improves the colour of the resulting fibres without much retarding the action, but, apparently owing to the formation of sulphur compounds, an extremely disagreeable odour is produced. This can be reduced by the simultaneous addition of dextrose to the retting liquor. Bacteriological tests showed the retting to be largely dependent on the activity of *Plectridia*. The fibres obtained on a large scale trial of this method were much superior to those ordinarily obtained.

—A. J. H.

Cellulose; Titration of —. W. Qvist. Pulp and Paper Mag., 1920, 18, 261–262, 285–287.

THE alkalinity of various samples of cellulose was determined using an ether solution of iodeosine as indicator. Samples of sulphite-cellulose had an alkaline reaction which diminished on repeated extraction with water to a neutral or faintly acid reaction. Bleached samples were less alkaline than unbleached. Sulphite-cellulose heated at 100° C. for a long time became acid, but became alkaline after prolonged extraction. Sulphate-cellulose and wood pulp reacted alkaline. Hydrocellulose and oxycellulose had an acidic reaction which became alkaline on washing. The alkalinity of the samples of cellulose is probably connected with the alkalinity of the river water with which the samples were prepared. Experiments showed that cellulose, especially oxycellulose, could absorb both acids and alkalis from solutions.—A. J. H.

Paper pulp from flax straw. Pulp and Paper Mag., 1920, 18, 315–350.

IN view of the demand for paper pulp, its production from flax straw would be profitable. The process of manufacture has been shown possible on

a semi-commercial scale. In 1919 Canada grew sufficient flax to produce 100,000 tons of pulp. Pulp from flax straw is comparable to that obtained from a fair grade of cotton rags.—A. J. H.

Paper pulp manufacture; Recovery of alkali from "black liquor" obtained in —. H. K. Moore. Paper, 1920, 25, 1157—1161, 1197—1201, 1241—1246.

THE "black liquor" is treated with salteake and passed through a coil heated well above 100° C. and sprayed into a combustion chamber where it is immediately vaporised. The "black liquor" particles are at once evaporated to dryness, ignited, and the alkaline products are condensed on the surface of a boiler, whereby the heat developed is utilised. The fused alkaline product percolates through the char on the bottom of the chamber, whereby the formation of sodium sulphide is completed, and thence passes to a dissolving tank. The combustion chamber is supplied with a blast of low-pressure air, but no extra fuel is needed.—A. J. H.

Colloidal rosin in beater sizing [of paper]. J. Traquair. Paper, 1920, 26, 185—186.

IF rosin size and an alum solution be mixed in the presence of a colloid, the precipitate of aluminium resinate is colloidal in character and is much more efficient for paper sizing. In practice, 50—66% of the usual amount of rosin sizing required can be saved with the production of an equally hard sized paper. The most suitable colloid is feculose (starch acetate). It is four times more efficient than maize starch, and the amount required is 18% on the weight of dry rosin.—A. J. H.

PATENTS.

Hemp; Manufacture of imitation —. H. Nishimura. Assee. of T. Honda. E.P. 130,590, 11.7.19. Conv., 27.7.18.

COTTON thread is drawn through a solution of Konnyaku-nori (a paste or size made by grinding the root of *Hydrosme rivieri* with water, which is converted into a white insoluble compound by tannic acid), and then through a $\frac{1}{2}$ —1% solution of tannic acid. The nap on the fibre is laid down and a hemp-like appearance is obtained. By boiling the treated thread for about 5 mins. in a 0.3—0.7% solution of sodium stannate or a 1—3% sodium silicate solution, a translucent lustre is produced. The treated threads can be dyed directly with basic dyestuffs.—A. J. H.

Flax; Production of fibres from —. W. J. Robinson. E.P. 141,982, 21.7.19. Conv., 23.4.19.

IN fibrous plants the fibres are bound to the woody portion by means of a cutose gum, while they are held end to end by a pectous gum. By the following procedure the cutose gum may be disintegrated without destroying the pectous gum. Flax straw is soaked in a saturated salt solution at 43° C., drained, and then exposed to the action of a hot or boiling liquid containing 10—20 lb. of an almost completely saponified oil (preferably the product obtained from flax seed) per 100 galls. of water. The addition of one pint of turpentine per 2 galls. of liquid is desirable. The fibres are secured to the surface of a drum which revolves partly immersed in the liquid, so that the fibres are alternately immersed in the liquid and exposed to the air. The cutose gum is disintegrated by this treatment, and after 45 mins. the drum is removed and revolved in a washing tank, then in a slightly acid bath, and finally rotated in a drying chamber at 110°—130° F. (43°—55° C.). Fibres thus prepared are three-quarters bleached and very lustrous.—A. J. H.

Artificial silk; Method and means for manufacture of —. C. L. Stulemeyer. E.P. 142,038, 5.11.19.

IN forming artificial fibres the jets through which the cellulose solutions are squirted are made of molybdenum, which is not affected by acids and alkalis, and is cheaper and stronger than platinum.—A. J. H.

Textile fibres; Production of — from wood. L. Enge. G.P. (A) 302,424, 13.3.17, (B) 303,293 24.6.17, (C) 304,313, 26.6.17, and (D) 304,312, 2.9.17.

(A) By various methods, such as treatment with alkalis and then acids, or *vice versa*, followed by digestion in a closed vessel with a solution of a neutral salt, a kind of half cellulose can be obtained from wood. Wooden logs and planks thus treated may then be mechanically disintegrated in a moist state, and further treated to obtain textile fibres. If heated under pressure these are especially strong and light coloured. (B) Pieces of wood not thicker than 1 mm. can be used instead of logs, and the chemical treatment is omitted. (C) The treatment of the wood before digestion can be varied. The acid and alkali treatment may be replaced wholly or partly by treatment with hydrocarbons, such as benzol, petroleum, naphtha, and gasoline, or by solutions of sodium sulphite, sodium chloride, magnesium sulphate, etc., which do not readily dissolve lignin. The wood may be first heated with alkalis and then treated with argillaceous earth in acetic or sulphuric acid. After one or other of the various treatments above mentioned the wood may be impregnated with oils and fats or solutions of metallic salts etc. (D) The bark bast of coniferous woods can be treated by the methods described under (B) or (C) or by gentle heating with dilute acid or alkaline solutions, and is dried, moistened or oiled, and then carded. Bast fibres may also be obtained from branches. The carding operation demands that the fibres be evenly moistened or oiled to prevent breakage. Bast fibres are finer than those from the wood itself.—A. J. H.

Textile fibres; Obtaining — from woody vegetable substances, such as nettle stalks, barks, etc. A. Hammer. G.P. 312,730, 19.11.15.

TO remove gummy substances and separate the fibres from the wood the nettle stalks or the like are treated with a malt solution, then fermented, boiled with alkali solution, and, finally, subjected to mechanical treatment.—W. P. S.

Wool and artificial wool fabrics; Production of durable —. M. Becke. G.P. 317,725, 21.12.16. Conv., 14.6.16.

IF in the treatment of wool the use of mineral acids and of salts capable of liberating such acids is avoided, the resulting fibres are much more durable, compact, and elastic, and more resistant to alkalis. The removal of burs and coarse plant particles from wool should be done by mechanical means. In the subsequent dyeing only weak organic acids and their salts are used.—A. J. H.

Balloon-fabrics and the like. C. Cleghorn, and The Gayner Pneumatic Co., Ltd. E.P. 142,160, 27.11.18.

A FABRIC impervious to gases such as hydrogen is composed of two sheets of fabric. The face of one sheet may be bare (if it constitutes the interior face of the balloon), and the other is coated with rubber. The second sheet has one face treated with an alcoholic solution of Turkish bird-lime (E.P. 130,379; J., 1919, 730 A), and the other is rubber-coated. The rubber-coated face of the first sheet is placed in contact with the face of the second sheet treated with bird-lime.—A. J. H.

Boiling indirectly; Method of —. [Sulphite-cellulose manufacture.] R. H. Hult. U.S.P. 1,337,701, 20.4.20. Appl., 24.10.17.

THE liquid is first heated by means of heating elements, such as steam coils, which are connected in parallel, and the heating then continued by means of the same or other elements connected in series. In the case of boiling the lye in the sulphite process of manufacturing cellulose the heating elements are connected in parallel until the temperature of the liquid has reached 100°–105° C., and then in series till the temperature reaches about 138° C.—B. M. V.

Acetylcellulose and process of making same. W. G. Lindsay, Assr. to The Celluloid Company. U.S.P. 1,338,661, 27.4.20. Appl., 9.4.18.

CELLULOSE is impregnated with benzene and alcohol and then acetylated with a mixture of acetic anhydride and benzene.—A. J. H.

Gutta-percha substitute from acetylcellulose; Preparation of a — resistant to alcohol. O. Skaller. G.P. 317,145, 13.11.18.

IF a film obtained from acetylcellulose solutions be immersed for a short time in concentrated alcohol, and then dried in air, it becomes quite insensitive to alcohol.—A. J. H.

Fireproofing composition and process of making the same. T. B. Munroe, Assr. to B. Q. Dahlberg. U.S.P. 1,338,322, 27.4.20. Appl., 28.8.19.

ZINC chloride is dissolved in excess of ammonia, and the free ammonia is neutralised by the addition of a substance capable of forming therewith a fire-resisting compound.—A. J. H.

Yarns or fabrics; Method of impregnating textile or paper —. Deutsche Pyroxitges. m.b.H. G.P. 312,686, 4.7.18.

THE yarn etc. is impregnated with wood tar and then dusted over with zinc oxide; the latter combines rapidly with the tar and forms a dry, wax-like mass.—W. P. S.

Cellulose waste lyes; Dry distillation of concentrated —. W. Schacht. G.P. 301,684, 27.8.16.

CONCENTRATED cellulose waste lyes are distilled in a current of an inert gas or steam or a mixture of both. The residue in the retort is then made alkaline and further distilled. From 100 pts. of cellulose lye pitch (sp. gr. 1.5), 50 l. of an aqueous distillate containing ammonia, alcohols, and oils was obtained. On the addition of alkali and further distillation there was obtained 2–4 kg. of oil of sp. gr. 0.930, which yielded about 25% of a good burning oil, sp. gr. 0.800–0.815, and various pyridine bases.—A. J. H.

Drying varnished or gummed sheets of paper and the like; Machines for —. E. and H. Bush. E.P. 141,997, 13.8.19.

Paper-making [machine]. C. E. Pope, Assr. to Great Northern Paper Co. U.S.P. 1,338,091, 27.4.20. Appl., 25.10.18.

Cellulose acetate; Manufacture of solutions of — and of celluloid-like masses, films, and other products therewith. H. Dreyfus. E.P. 128,215, 10.6.19. Conv., 4.3.14.

SEE Third Addn. to F.P. 432,261 of 1911; J., 1916, 40.

Gas mantles. G.P. 312,577. See III.

Coating substances. E.P. 111,111. See XIII.

Saccharifying cellulosic materials. G.P. 316,696. See XVIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Steaming fabrics; Apparatus for —. J. Grasser. E.P. 138,637, 4.2.20. Conv., 4.2.19.

A CHAMBER of masonry is divided by a partition having an opening through which the fabric can be raised from the lower chamber to the upper one, which constitutes the real steaming chamber. Steam is introduced at the top of the steaming chamber and is evenly distributed so that all the air is expelled through the partition as the steam fills the chamber. The fabric, wound on a framework, is introduced into the lower chamber, and when the air in the top chamber is expelled is raised through the partition. After steaming, the material is lowered and removed from the lower chamber. The apparatus can be used with advantage for discharging with hydrosulphites.—A. J. H.

Coating process. W. Alexander. U.S.P. 1,337,380, 20.4.20. Appl., 20.10.15.

A FIBROUS surface is coated with an adhesive solution of a water-soluble salt of a base metal capable of readily forming sulphides, such salt being then caused to react with a sulphur-containing substance whereby a decorative coating is produced.

—A. de W.

Dyeing, washing, and similarly treating yarn in the form of hanks, skeins, or the like. J. Annick. U.S.P. 1,337,877, 20.4.20. Appl., 2.9.19.

COMPRESSED yarn is allowed to expand in and completely fill a dyeing chamber. A dye solution is forced through the material, which is then washed and freed from liquid by applying vacuum to one end of the chamber.—A. J. H.

Textiles; Dressing, printing, and dyeing —. F. Günther. G.P. 318,509, 20.6.14.

WATER-SOLUBLE phenol-formaldehyde condensation products, either alone or mixed with cellulose derivatives, such as cellulose esters, nitrocellulose, acetylcellulose, or viscose, and/or thickeners, are employed as textile dressings for fixing metal powders, pigments, or dyes. The condensation products are subsequently hardened by heating. The addition of a cellulose derivative increases the elasticity of the condensation product.—L. A. C.

Dyeing or otherwise treating textile fabrics; Apparatus for use in processes for —. Färberei Weidmann A.-G. E.P. 129,975, 3.7.19. Conv., 18.7.18.

SEE U.S.P. 1,329,166 of 1920; J., 1920, 400 A.

Disazo-dyestuffs. E. P. 141,401. See IV.

Dye recovery. U.S.P. 1,337,673. See IV.

Durable wool etc. fabrics. G.P. 317,725. See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Nitric acid; War experiences in the manufacture of — and the recovery of nitrous fumes. J. Walker. Chem. Soc. Trans., 1920, 117, 382–389.

AT H.M. Factory, Craigleith, the catalytic oxidation of ammonia to nitric oxide was effected by leading an ammonia-air mixture containing 10–12% NH₃ through a glass or slag wool filter to a platinum gauze catalyst placed horizontally across an aluminium box. The gases emerging from the converter were cooled and led through oxidation and absorption towers, the last traces of nitric acid

being removed by wash towers fed with milk of lime. The recovery of nitric acid and nitrous gases from the waste acid of T.N.T. manufacture was effected in denitrators of acid-resisting brick, square in section, and divided into six compartments by finely perforated earthenware plates (malting plates). Steam was blown in at the base and waste acid fed in at the top. The denitrated acid removed at the base was passed to sulphuric acid concentrators, and the gases from the denitrators, after passing through cooling coils which condensed out a dilute nitric acid, were led through a series of six recovery towers 90 cm. in diameter and 5 m. high, worked on the counter-current principle, the nearly exhausted gas coming into contact in the last tower with the dilute nitric acid from the denitrator condensers. The recovery by this system was more than 93% of the inorganic nitrogen delivered to the denitrators in the waste acid.

—G. F. M.

Sodium nitrate; Determination of nitrogen in — by the modified Devarda method and the use of the Darvison scrubber bulb. C. A. Butt. *J. Ind. Eng. Chem.*, 1920, 12, 352—354.

THE modified Devarda method (Allen, J., 1915, 726) yields accurate and concordant results, particularly when the Darvison scrubber bulb (J., 1919, 443 A) is used to prevent alkali spray passing over into the absorption vessel.—W. P. S.

Nitrite+nitrate+nitric oxide; Electrolytic potential of the change —. Energy relationships of the most important compounds of nitrogen with oxygen and hydrogen. H. Pick. *Z. Elektrochem.*, 1920, 26, 182—196.

THE normal potential of the change $2\text{NO}_2' + \text{NO}_3' + \text{NO}$ is +0.49 volt at 25° C. A series of normal potentials are given for N_2 , N_2O , NH_3 , NH_4 , NO_2 , and N_2O_4 . (Cf. J.C.S., July.)—J. F. S.

Iodide and bromide in mineral waters and brines; Determination of —. W. F. Baughman and W. W. Skinner. *J. Ind. Eng. Chem.*, 1920, 12, 358.

A COMBINATION of two methods described previously by the authors (J., 1919, 498 A; 1920, 19 A) is recommended. After the iodine has been removed by distillation with ferric sulphate, the residual solution is treated with a slight excess of ammonia, boiled, the ferric hydroxide separated by filtration, the filtrate evaporated to dryness, and the residue thus obtained used for the determination of the bromide.—W. P. S.

Potassium bromide; Solubility of — in bromine water. A. F. Joseph. *Chem. Soc. Trans.*, 1920, 117, 377—381.

THE solubility of potassium bromide in water is increased by the addition of bromine; for moderate concentrations about 0.5 grm.-mol. additional bromide is dissolved for each grm.-mol. of bromine added to the water. The limit of the solvent capacity of the water was not reached at bromine concentrations of more than 2060 g. per litre of solution, for example with a solution of the composition by weight—water 5.4%, bromide 15.1%, bromine 79.5.—G. F. M.

Potassium ferriocyanide; Electrolytic preparation of —. O. W. Brown, C. O. Henke, and I. L. Miller. *J. Phys. Chem.*, 1920, 24, 230—237.

THE maximum yield obtainable in the electrolytic oxidation of potassium ferrocyanide in an undivided cell was 50%. When a diaphragm was used with a current of 0.90 amp. caustic potash was found in both compartments of the cell and a current efficiency of about 98% was obtained. The presence of potassium hydroxide increased the current effi-

ciency and decreased the voltage slightly. All the ferrocyanide decomposed was not converted into ferriocyanide. Electrodes of different materials gave different current efficiencies, and in all cases the maximum efficiency was not reached until the electrolysis had been in progress for 4 hrs. The following maximum current efficiencies were obtained working with an anode $7\frac{1}{2} \times 11\frac{1}{2}$ cm. and a current of 0.95 amp.: copper 96.3%, nickel 97.7%, platinum 96.6%, Acheson graphite 99.7%, and lead covered with lead dioxide 93%.—J. F. S.

Slaked lime and milk of lime. B. Kosmann. *Z. Elektrochem.*, 1920, 26, 173—181.

THE views of Kohlschütter and Walther (J., 1920, 25 A) are criticised. The author holds the view that the slaking of lime consists in a gradual step-wise combination with water in which the following substances are produced in order: $\text{Ca}(\text{OH})_2$, $\text{H}_2\text{Ca}(\text{OH})_4$, $\text{H}_2\text{Ca}(\text{OH})_6$, $\text{H}_2\text{Ca}(\text{OH})_8$, $\text{H}_2\text{Ca}(\text{OH})_{10}$, $\text{H}_2\text{Ca}(\text{OH})_{12}$, and $\text{H}_2\text{Ca}(\text{OH})_{14}$.—J. F. S.

Alum; Rapid estimation of aluminium sulphate in —. P. W. Codwise. Paper, 1920, 26, 187—189.

THE method is based on the reaction: $\text{Al}_2(\text{SO}_4)_3 + 6\text{NaOH} = 2\text{Al}(\text{OH})_3 + 3\text{Na}_2\text{SO}_4$. A quantity of alum or aluminium sulphate equivalent to about 40 c.c. of N/10 caustic soda solution is dissolved in exactly 25 c.c. of distilled water, the solution heated to the boil and one drop of a 1% alcoholic solution of phenolphthalein added. Three successive quantities of 10 c.c. of N/10 caustic soda are added, and the liquid heated to boiling for exactly 1 min. after each addition, and then the caustic soda is added gradually until a slight pink tinge is produced and persists after boiling for 1 min. If the sample contains free acid, this must be determined and allowed for. If the above procedure be followed exactly, the results agree within 0.1%. The method only measures combined alumina, so that with basic alums the results are low. The presence of iron impurities does not affect the results.

—A. J. H.

Radium and mesothorium; Concentration of — by fractional crystallisation. J. L. Nierman. *J. Phys. Chem.*, 1920, 24, 192—200.

THE crystallisation factor (concentration of mesothorium in crystals separated: concentration in original crystals) of mixtures of barium, radium, and mesothorium bromides is independent of the concentration of the hydrobromic acid from which crystallisation takes place. The value of the crystallisation factor decreases with increasing percentage of separation: thus when 24.3% of the salt crystallises the factor is 2.43, and when 69% crystallises 1.44. For concentrations of mesothorium up to 2 mg. per 1 g. of salt the crystallisation factor remains constant. The separation of radium and mesothorium from barium can be advantageously carried out in either neutral or slightly acid solutions.—J. F. S.

Hydrogen peroxide; Activation of — by light. H. A. Curtis. *J. Amer. Chem. Soc.*, 1920, 42, 720—724.

SOLUTIONS of Aniline Green and fuchsine are not decolorised by 3% hydrogen peroxide, nor are they bleached by light of short wave lengths, but if a mixture of the dye solutions and 3% hydrogen peroxide is exposed to the light from a quartz mercury lamp the solution is bleached rapidly. Bubbling oxygen through the dye solution during illumination does not effect bleaching.—J. F. S.

Catalytic actions at solid surfaces. II'. Interaction of carbon monoxide and steam as conditioned by iron oxide and by copper. E. F. Armstrong and T. P. Hilditch. *Proc. Roy. Soc.*, 1920, A, 97, 265—273.

At high temperatures copper influences the reaction

$\text{CO} + \text{H}_2\text{O} \rightleftharpoons \text{CO}_2 + \text{H}_2$ much less than iron oxide, but over the range $200^\circ\text{--}300^\circ\text{C}$. copper is definitely more active than iron oxide. Copper commences to react with a fair velocity at 220°C ., whereas iron oxide is only slightly active at 250°C . and does not reach full activity until 400°C . Small quantities of ammonium formate are produced when water-gas (5.1 CO_2 , 39.9 CO , and 47.3 H_2) with steam in the presence of ammonium is passed through a tube at 350°C . (Cf. J.C.S., July.)

—J. F. S.

Sulphur; Determination of minute quantities of "oil" in —. L. S. Bushnell and H. S. Clark. J. Ind. Eng. Chem., 1920, 12, 485.

ABOUT 50 g. of the pulverised sulphur is shaken at intervals with 50 c.c. of petroleum spirit, and after some hours the extract is decanted through a filter into the tube of a Wiley continuous extractor, into which is introduced a clean dry coil of copper foil, 4 in. by 15 in., rolled up so that no points of its surface are in contact. The residue in the flask and the filter are washed with petroleum spirit (about 175 c.c. in all) until the washings are sufficient to fill the Wiley tube. The extract is then boiled until the whole of the dissolved sulphur has been deposited as copper sulphide upon the coil, after which it is filtered and evaporated to dryness at a low temperature, and the residue of "oil" weighed.—C. A. M.

Sorption of vapours by charcoal. B. Gustafson. Ark. Kemi, 1917, 7, No. 22, 1–17.

CHARCOAL takes up water vapour and acetic acid vapour at low pressures (1.6–17.4 mm.) proportionally with the pressure. Up to a pressure of 7.4 mm. the sorption process consists in the formation of a solid solution, and above this pressure simple absorption occurs. The velocity of sorption is greater the lower the pressure. (Cf. J.C.S., July.)—J. F. S.

Alkali from "black liquor." Moore. See V.

Alkali amalgams. Frank and Withrow. See X.

Catalase. Yamasaki. See XVIII.

Acetates. Curtman and others. See XX.

Calcium, strontium, and barium salts. Denigès. See XXIII.

PATENTS.

Nitric and sulphuric acid; Process of concentrating —. A. C. Bohre. U.S.P. 1,338,418, 27.4.20. Appl., 28.7.19.

HOT nitrous gases are brought in contact with nitric or sulphuric acid to be concentrated, the resulting vapours are condensed by contact with a cooling liquid, and the cooling liquid is then cooled for condensing further quantities of the vapours by passing a gas through it.—L. A. C.

Nitric acid; Manufacture of — by catalytic oxidation. A. Maschke. G.P. 307,052, 10.11.17.

THE gases obtained by heating ammonium bisulphate or ammonium sulphate are mixed with air and substituted for ammonia in the catalytic process. The resulting vapours may be cooled, yielding a mixture of nitric and sulphuric acids suitable for nitration purposes, or dilute sulphuric acid and highly concentrated nitric acid may be obtained by cooling in stages.—W. J. W.

Nitrous gases; Process for rapid cooling of — obtained by combustion of nitrogen. R. Mewes. G.P. 316,214, 25.4.15. Addition to 298,846.

THE electrically-heated gas mixture under high pressure, containing the nitrous gases which have been formed, is mixed with air at an equal or higher pressure and previously cooled or liquefied, after

which the mixture is allowed to pass into an expansion chamber. After absorption of the nitrous gases the residual mixture is returned to the system.

—W. J. W.

Nitric oxide; Method of working gas engines for producing —. J. Goring, G.P. 316,253, 6.4.19.

A MIXTURE of coal gas and producer gas is used as fuel. The coal gas should preferably contain 40–50 H_2 and have a calorific value of 4000–5000 cals. per cb. ft., but good results are obtained so long as the calorific value does not fall below 2000 cals. per cb. ft., provided the ratio of hydrogen content to calorific value is about 10 H_2 per 1000 cals.

Sulphate of ammonia; Manufacture of —. S. E. Linder and R. Lessing. E.P. 141,787, 18.1.19.

COMMERCIAL ammonium sulphate is treated with lime, magnesia, or other alkaline earths or their carbonates, or with suitable ammonium salts such as the sulphide, sulphate, or carbonate, applied in the solid state or in solution, to neutralise the free acid. The sulphate is treated with one of the above agents or with gaseous ammonia in some form of heated disintegrating chamber, the treated material forming a dry granular or pulverulent product. If the treatment is effected in a partial vacuum or in a current of air or other gas, the pyridine normally present in the crude sulphate is wholly removed and recoverable.—D. F. T.

Ammonium sulphate; Manufacture of —. South Metropolitan Gas Co., E. V. Evans, and H. Hollings. E.P. 141,798, 20.1.19.

CAKING of ammonium sulphate may be avoided by neutralisation of the free acid present in the crystals, followed by drying at an elevated temperature such as $80^\circ\text{--}100^\circ\text{C}$.—D. F. T.

Sulphate of ammonia; Manufacture of —. South Metropolitan Gas Co., and O. W. Wright. E.P. 141,799, 20.1.19.

THE free acid in crude ammonium sulphate is neutralised by mixing with solid or dissolved ammonium sulphite or bisulphite, or by the addition of either of these substances to the liquor used for washing the ammonium sulphate crystals. Any small excess of sulphite finally undergoes natural oxidation to sulphate.—D. F. T.

Sulphate of ammonia; Manufacture of neutral —. South Metropolitan Gas Co., P. Parrish, and W. A. M. Valon. E.P. 141,819, 23.1.19.

FOR neutralising ammonium sulphate crystals aqueous ammonia is prepared by condensing the vapours of the "fixed ammonia" still and diluting the resulting solution so that its vapour pressure at the temperature of application to the crystals will be too low to entail substantial loss of ammonia. The temperature of washing should be as low as possible, because both the reduced temperature and the use of more concentrated solutions of ammonia tend to decrease the loss of ammonium sulphate during the operation. A particularly suitable diluent for the condensed vapours is produced by subjecting the hot waste liquor of the fixed ammonia still to a slightly reduced pressure, the condensed liquid thus obtained containing a considerable portion of the residual ammonia which would otherwise be lost.—D. F. T.

Ammonium sulphate; Production of — from distillation gases by means of sodium bisulphate. P. von der Forst. G.P. 298,012, 8.3.16.

A SATURATED or nearly saturated hot solution of sodium bisulphate is neutralised with the ammoniacal gases so that Glauber's salt separates. By evaporation and crystallisation of the remaining

solution ammonium sulphate is obtained, or the solution may be added to the sulphuric acid fed to a saturator of the usual type.—E. H. R.

Ammonium sulphate; Reaction tower for conversion of ammonium carbonate into — by means of calcium sulphate. C. Otto und Co. G.P. 299,621, 18.2.16.

In the manufacture of ammonium sulphate by treatment of ammonium carbonate with plaster of Paris paste, sodium chloride may be added to increase the solubility of the calcium sulphate. The paste is of such a concentration as to retain a large amount of the ammonium sulphate produced. By mixing steam with the ammonia and carbon dioxide vapours a suitable temperature is maintained, and the liquors will be free from ammonia. The conversion may be carried out in a reaction-tower comprising a top section with inlet for the plaster of Paris paste and exit for gases, and a bottom section with steam inlet and run-off for the paste. The tower is packed with a number of rings, each of which forms a receptacle in which the reacting liquids can remain sufficiently long to ensure complete conversion, an additional, smaller cup in the ring serving for the introduction of steam into the liquid. Scrapers, connected to a central shaft, operate in the cups and receptacles.—W. J. W.

Ammonia; Process of manufacturing — from crude calcium cyanamide. C. Otto und Co. G.P. 303,842, 28.3.16.

By the use of the reaction-tower described in G.P. 299,621 (preceding abstract), the formation of lime crusts is prevented. The ring receptacles are of such dimensions as to admit of the calcium cyanamide mud remaining in the tower about four times as long as in other processes.—W. J. W.

Ammonium chloride; Manufacture of — from ammonia and alkali or alkaline-earth chlorides. Salzwerke Heilbronn, A.-G. G.P. 306,351, 5.10.16.

CHLORIDES are melted, or heated above 150° C., and treated with ammonia or ammoniacal gases in presence of steam. Treatment with steam may precede the interaction with ammonia. Carbon dioxide may be introduced into the process, and substances such as iron oxide, clay, etc. may be added to accelerate the reaction. By conducting the conversion in a reducing atmosphere side-reactions, such as combustion of the ammonia, are avoided.—W. J. W.

Halogen compounds of silicon, boron, or titanium; Production of fluid —. British Thomson-Houston Co. From General Electric Co. E.P. 141,908, 23.4.19.

THE formation of undesirable solid by-products on account of undue liberation of heat during the interaction of chlorine or bromine and silicon, boron, or titanium or suitable derivatives of these, such as their carbides, is obviated by circulating water around the reaction chamber so as to maintain the temperature below the point at which such by-products are liable to be produced.—D. F. T.

Barium carbonate; Manufacture of finely sub-divided —. H. Langwell. E.P. 141,925, 10.5.19.

FINELY sub-divided barium carbonate is obtained by heating the natural or artificial material with sodium chloride; the fused salt dissolves approximately twice its weight of the carbonate, and impurities separate as a sediment or scum. The molten mass is then treated with water and the barium carbonate collected.—D. F. T.

Caustic soda; Apparatus for the manufacture of "detached" or flake — or other matter required to be obtained in a state of division from a fused or molten mass. T. Minton, and The United Alkali Co., Ltd. E.P. 141,942, 23.5.19.

MOLTEN material such as sodium hydroxide or the

like is fed into a rotating, circular, open pan, water-cooled from underneath. A stationary scraper is fixed along a radius of the pan, and a ribbed or toothed roller rotates above the scraper so that the material separated from the pan by the scraper is broken up into flakes. A rotating brush sweeps the flaked material up an inclined plane, also radially disposed, on to a trough, whence it is removed by an endless chain conveyor.—L. A. C.

Tin; Methods and apparatus for the oxidation of — and the production of tin oxide, and for the treatment of zinc and zinc-containing materials for the production of zinc oxide. H. Maconochie and D. de Ros. E.P. 142,157, 30.10.18.

As an improvement in the processes described in E.P. 118,664 and 118,665 (J., 1918, 622 A), tin (or zinc) is preheated to about 900° C. by an oil burner, and the temperature is then allowed to rise to 1500°–1700° C. by the application of a high-pressure air-blast (i.e., about 4–5 lb. per sq. in.) with the production of tin (or zinc) oxide, which is removed. Completion of the oxidation is effected by means of a low-pressure blast (i.e., about 0.15–0.5 lb. per sq. in.), and the temperature may be maintained by the addition of the necessary amount of oil to the blast. Different types of furnace suitable for the process are described.—L. A. C.

Alkali chromates; Process for isolating or purifying —. Soc. Ind. des Prod. Chim. E.P. 138,111, 25.1.18. Conv., 2.8.17.

A CONCENTRATED solution of crude alkali chromates containing only a small percentage of alkali hydroxides is treated with carbon dioxide whereby silicates and aluminates are converted to silica and alumina, and alkali hydroxides to bicarbonates; the solution is then filtered and evaporated to crystallise pure alkali chromates. The dry crude chromates may be treated with carbon dioxide, and the product extracted with a little water.—L. A. C.

[Alkali] manganates; Process of making —. H. McCormack, Assr. to Research Laboratory of Chicago. U.S.P. 1,337,239, 20.4.20. Appl., 6.1.19.

A MIXTURE of manganese dioxide and an aqueous solution of an alkali hydroxide is evaporated to dryness and heated to complete the reaction; the product is extracted with water, and, after separation of insoluble matter, the clear solution is cooled to crystallise the manganate.—L. A. C.

[Alkali] manganates; Process of making —. C. E. Vanderkleed, Assr. to Hercules Powder Co. U.S.P. 1,339,242, 4.5.20. Appl., 21.11.18.

MANGANESE dioxide and sodium peroxide are fused together to form sodium manganate.—L. A. C.

Potassium salts; Recovery of —. P. C. McIlhiney, Assr. to California Alkali Co. U.S.P. 1,338,234 and 1,338,235, 27.4.20. Appl., 11.2.18.

COMMERCIAL pure potassium salts, e.g., the chlorate, may be obtained from natural brines containing potassium salts associated with borax by the addition of a soluble salt of which the acid radical forms an insoluble potassium salt, e.g., a chlorate, and removing the insoluble potassium salt from the solution.—J. H. J.

Potash salts; Process for increasing the potash content of crude —. J. Kiermayer, and Hannoversche Kaliwerke A.-G. G.P. 317,771, 21.11.15.

AFTER melting and removing the impurities, the potash salts are treated with reducing agents, by which means the magnesium sulphate is converted into magnesite and sulphur compounds. Coke and charcoal are suitable agents; coal gives unstable by-products. By this treatment the potash content of a mineral of the composition K₂O, 8.5%; kieserite, 25%; NaCl, 50%; and clay etc., 5.5%, was increased to 12% K₂O.—W. J. W.

Crystallisation of solutions, especially of potassium salts; Process for —. G. Sauerbrey. G.P. 318,350, 30.10.18.

The solution to be crystallised is conveyed by a pipe to the centre of a flat plate, which may have a rim, and which revolves round a vertical axis. The plate may be revolved continuously or intermittently to enable the crystals to be removed, and its speed may be controlled.—W. J. W.

Radium; Producing compounds of —. H. O. Hedström. U.S.P. 1,338,831, 4.5.20. Appl., 31.10.19.

RADIUM is extracted from minerals etc. by treatment with liquid sulphur dioxide containing sulphur trioxide. (Cf. E.P. 136,768 of 1919; J., 1920, 156 A.)—W. E. F. P.

Alkali nitrates; Manufacture of — from mixtures of nitric acid and water, and alkali sulphates. E. Reinau. G.P. (A) 299,001, 1.2.16; (b) 299,002, 25.3.16; (c) 299,003, 23.6.16; (d) 299,006, 25.3.16; and (e) 299,007, 17.2.16.

(A) THE materials are caused to react, under influence of heat, in such proportions as to convert the sulphate into bisulphate. The mixture is then cooled if necessary, and the solid portion consisting chiefly of alkali nitrate is separated from the liquid mixture of water, alkali nitrate, sulphate, bisulphate, and free acid. The solid portion may be mixed with a fresh quantity of nitric acid and water. Instead of nitric acid and water a mixture of water and two or more of the following components may be employed:—alkali nitrate, sulphate, bisulphate, nitric acid, and sulphuric acid. The cooling of the reaction mixture, during separation of the solid portions, may be intermittent. 50% of the combined nitric acid may be recovered as 92–95% alkali nitrate under suitable conditions of temperature and concentration. (u) Mixtures of various alkali sulphates, with or without hydroxides, may be used. (c) The process, or certain phases of it, may be carried out under other than atmospheric pressure, loss of nitric acid on evaporation being thus avoided. (d) Alkali magnesium sulphates, or mixtures of alkali and magnesium sulphates, or hydroxides, may be employed. (e) The liquid portions consisting of mother liquor and wash-waters may be freed from uncombined nitric acid by distillation, and the condensed acid mixed with fresh nitric acid and used again. The residue after distillation consisting of alkali sulphate and bisulphate is also available for the process.—W. J. W.

Alkali nitrates and hydrochloric acid; Manufacture of — from mixtures of nitric acid and water, and alkali chlorides. E. Reinau. G.P. (A) 299,004, 17.2.16, and (n) 299,005, 25.3.16. Addns. to 299,001 (preceding).

(A) By the interaction of nitric acid, water, and alkali sulphate, alkali bisulphate is obtained, which is then caused to react with alkali chloride, with or without addition of alkali sulphate, to form hydrochloric acid. The alkali sulphate required for the process is prepared by treatment of an alkali compound with alkali bisulphate, polysulphate, or free sulphuric acid, or another sulphate. (n) Mixtures of alkali sulphates with or without alkali chlorides, and with or without hydroxides, may be employed.—W. J. W.

Alkali nitrates and ammonium sulphate; Manufacture of — from mixtures of nitric acid and water, alkali sulphate, and ammonia. E. Reinau. G.P. 305,062. 1.4.17. Addn. to 299,001 (preceding).

IN the first stage, by the action of water and nitric acid on alkali sulphate, part of the alkali is separated as alkali nitrate, while the combined sulphuric

acid and acid alkali sulphate remain. In the second stage the acid alkali sulphate is treated with water and gaseous ammonia, causing separation of alkali nitrate and solution of alkali-ammonium sulphate, from which a high percentage of ammonium sulphate may be recovered. The alkali sulphate recovered in this second stage is available for use in the first stage.—W. J. W.

Liquors containing calcium sulphate; Treatment of —. G. Sauerbrey. G.P. 318,310, 15.4.19.

THE liquors are treated with magnesium chloride, the precipitated chlorides of potassium and sodium being removed, and the liquors then evaporated by the action of furnace gases. A further quantity of salts may be separated by cooling the solution before evaporation.—W. J. W.

Argon; Production of —. G. A. Percival, and The Edison Swan Electric Co., Ltd. E.P. 141,783, 9.1.19.

ARGON is separated from air by circulating the air through a heated tube fitted with a worm conveyor, by means of which an absorbent for oxygen and nitrogen, e.g., calcium, is passed through the tube in a direction opposite to that of the air. Air-tight charging and discharging devices, consisting of chambers with double doors, are attached to the ends of the tubes, and an "ometer" for collecting the argon is provided in the air-circulating system.—L. A. C.

Air; Method of and apparatus for fractionating —. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 141,864, 14.3.19.

OXYGEN, nitrogen, and an argon concentrate are obtained from liquid air by fractionation in a rectifying column having an inlet for liquid air and an outlet for distillate near the top, and means for collecting liquid gas (largely oxygen) at the bottom. The distillate withdrawn from the upper part of this column is liquefied and subsequently treated in a second column to produce gaseous nitrogen and a liquid consisting largely of oxygen and argon.—W. E. F. P.

Oxygen concentrator. G. McKerahan. U.S.P. 1,339,211, 4.5.20. Appl., 27.1.20.

A CIRCULAR (disc-shaped) vessel, mounted to rotate about a horizontal axis, has on one side an air inlet around the axis and, on the opposite side, an outlet situated between the axis and the periphery and concentric with the latter. A second outlet is disposed around the extreme periphery of the vessel.—W. E. F. P.

Chlorine liquefaction; Process of —. C. T. Henderson, Assr. to Niagara Smelting Corp. U.S.P. 1,338,654, 27.4.20. Appl., 10.2.17.

CHLORINE is dissolved in carbon tetrachloride and then expelled from the solution into a condenser, the gas being liquefied by the pressure thus produced.—W. E. F. P.

Hydrogen or nitrogen; [Catalytic] production of —. F. Lang. G.P. 307,651, 24.9.16.

A CONTACT chamber for catalytic production of hydrogen or nitrogen by the alternate passage of steam or air and reducing gases over a contact substance comprises a cylindrical outer shell with permeable walls and gas and steam distributing compartments, and an inner permeable wall forming a tubular distributing chamber for the steam and gases. The contact chamber may have internal projecting pieces and partitions. It may be divided into upper and lower compartments, communicating by means of tubes, and connected to common inlets and exits. Low yields in the process are ascribed to the influence of reverse reactions, which increase in proportion to the height of the contact chamber.—W. J. W.

Nitric acid and tetroxide of nitrogen; Production of pure concentrated —. Norsk Hydro-Elektrisk Kvaestofaktieselskab. E.P. 124,191, 4.2.19. Conv., 13.3.18.

SEE U.S.P. 1,324,255 of 1919; J., 1920, 108 A.

Nitric acid; Manufacture of concentrated —. E. Bergey and O. Jensen, Assrs. to Norsk Hydro-Elektrisk Kvaestofaktieselskab. U.S.P. 1,338,417, 27.4.20. Appl., 30.9.18.

SEE E.P. 120,378 of 1918; J., 1919, 817 A.

Alkali chromates; Process for isolating or purifying —. Soc. Ind. de Prod. Chim. E.P. 119,647, 25.1.18. Conv., 2.8.17.

SEE U.S.P. 1,324,328 of 1919; J., 1920, 190 A.

Thorium; Separating — from other rare earths and manufacture of thorium nitrate and other thorium salts. J. V. Clarke. U.S.P. 1,335,482 30.3.20. Appl., 28.9.18.

SEE E.P. 120,748 of 1917; J., 1919, 74 A.

Nitrogen compounds; Method of manufacturing — from carbides. O. F. S. Carlson, Assr. to Aktiebolaget Nitrogenium. U.S.P. 1,337,750, 20.4.20. Appl., 29.7.16.

SEE E.P. 123,796 of 1918; J., 1919, 253 A.

Ammonium perchlorate; Method of manufacturing —. O. B. Carlson, Assr. to Aktiebolaget Carlit. U.S.P. 1,338,357, 27.4.20. Appl., 25.9.17.

SEE E.P. 110,514 of 1917; J., 1918, 301 A.

Titanium compounds; Preparation of —. H. Wrigley and H. Spence, Assrs. to P. Spence and Sons. U.S.P. 1,338,473, 27.4.20. Appl., 4.2.19.

SEE E.P. 133,336 of 1917; J., 1919, 901 A.

Drying limestone. E.P. 141,873. See I.

Spent oxide. E.P. 141,172. See II.

China clay. E.P. 142,161. See VIII.

Detergent. G.P. 318,151. See XII.

Ferric oxide. U.S.P. 1,337,402. See XIII.

VIII.—GLASS; CERAMICS.

PATENTS.

Glass; Manufacture of —. E. H. Langwell. E.P. 142,365, 9.7.19.

AN outlet for molten glass in a glass furnace is provided with several nozzles made of steel and lined with refractory material, and is rotated so that each nozzle is used in turn. After a nozzle has been used it is heated and all adherent glass is removed, so as to avoid the production of defects or marks in the glass.—A. B. S.

Plastic masses and new industrial products obtained therefrom; Manufacture of —. L. Gauthier. E.P. 128,905, 15.5.19. Conv., 22.6.18.

A PLASTIC mass is made by incorporating 70–90% of tale, with or without the addition of 2% of zinc white, in 30–10% of gelatin which has previously been allowed to swell in water and then melted, the mixture being stirred until it thickens. The paste is made into cakes and stored in a damp cloth. It may be moulded under pressure, and after drying in air for 8–10 days may be treated with formaldehyde and varnished or soaked in water-glass. The product may be used as a substitute for porcelain in the manufacture of electrical insulators etc.—A. B. S.

Plastic compositions. L. Deleglise. E.P. 141,987, 29.7.19.

A MIXTURE of 25 parts of fibres of wood, paper, rags, or the like, and 175 parts of plaster is kneaded, heated to 50°–80° C., incorporated with a mixture of a solution of 45 parts of gelatin (glue) containing 10 parts of tin dioxide and 110 parts of water at the same temperature, and the mixture kneaded and heated with 35 parts of a 14% aqueous solution of formalin or sodium bichromate.

—A. B. S.

Brick kilns. A. Sharratt. E.P. 142,237, 4.3.19.

IN a continuous kiln or series of intermittent kilns, instead of the usual metal or paper dampers closing the trace holes or passages between each chamber, a wall of coke is used which filters the gases and so prevents them damaging the goods. When the firing has proceeded sufficiently, the coke is ignited and eventually burns away.—A. B. S.

China clay; Drying —. J. Adair. E.P. 142,161, 16.12.18 and 8.1.19.

CHINA clay slurry is discharged through the sloping bottom of the settling tanks into a belt elevator and thence on to fixed aluminium trays, the bottoms of which may be either flat or corrugated. The trays are in two series, the upper ones being fixed and the lower ones mounted on wheels so that they may be run into a rectangular drying chamber heated by gas burners or electric radiators arranged in parallel lines and mounted to move reciprocally and so ensure uniform heating. The dried clay is discharged by releasing the hinged sides of the trays and tilting the latter.—A. B. S.

Clay ware; Method and apparatus [tunnel kiln] for burning —. C. B. Harrop. U.S.P. 1,338,771, 4.5.20. Appl., 13.6.19.

THE ware is burned in a tunnel kiln having "staggered" clearance spaces in its walls, these spaces increasing towards the bottom of the setting of ware. The upper portion of the setting of ware is placed in such a manner that the gases will pass downwards through the setting to the lower parts of the clearance spaces.—A. B. S.

Abrasive structures; Method of making —. C. W. Thomas. U.S.P. 1,338,598, 27.4.20. Appl., 24.7.16.

ABRASIVE grains are mixed with a temporary binder and a permanent binder composed of pre-shrunk vitreous material. The mixture is moulded and then fired at a temperature sufficiently high to fuse the vitreous binder.—A. B. S.

Aluminous [abrasive] composition and method of making the same. O. Hutchins, Assr. to The Carborundum Co. U.S.P. 1,339,344, 4.5.20. Appl., 18.9.19.

AN aluminous abrasive is made by fusing in an electric furnace a mixture of an aluminous material and a strontium compound which yields strontium oxide.—A. B. S.

Graphite articles and method of making the same. S. C. Linbarger, Assr. to The Carborundum Co. U.S.P. 1,339,266, 4.4.20. Appl., 13.12.18.

THE articles are made of particles of graphite coated with a thin film which prevents oxidation.

—A. B. S.

Glass tubes; Manufacture of reinforced —. K. Küppers. E.P. 134,841, 4.11.19. Conv., 13.11.16.

Solid [refractory] bodies from nitrides; Process of making —. E. Podszus, Assr. to The Chemical Foundation, Inc. U.S.P. 1,337,264, 20.4.20. Appl., 21.10.14. Renewed 26.6.19.

SEE G.P. 282,748 of 1913; J., 1915, 717.

Moulding semi-solid substances [clay, graphite, etc.]. B. J. Allen. U.S.P. 1,337,811, 20.4.20. Appl., 14.8.17.

SEE E.P. 111,762 and 111,775 of 1917; J., 1918, 58 A.

IX.—BUILDING MATERIALS.

Cement raw materials; Reactions of — during heating. R. Nacken. Zement, 1920, 9, 61—63, 74—75, 85—89. Chem. Zentr., 1920, 94, II., 591.

On heating Rüdgersdorf raw materials, a strongly exothermic reaction occurs between 1200° and 1300° C., followed by an endothermic reaction accompanied by sintering. With chemically pure materials (free from MgO, SO₃, etc.) the exothermic effect is most strongly marked at 1350° C.

—W. J. W.

PATENTS.

Wood; Process and apparatus for rendering — impermeable and recovering liquids contained therein. C. Roy. E. P. 130,981, 29.7.19. Conv., 3.8.18.

Logs of wood are made impermeable by attaching to them a head and injecting water at a suitable temperature followed by an aqueous solution of casein and ammonia containing lime so as to replace the sap. The logs are then cut into planks and dried in a hot chamber so as to precipitate the casein. The treated timber is resistant to the action of heat, moisture, and light; it does not warp and may be easily glued.—A. B. S.

[Waterproof] construction materials; Process of making —. R. P. Perry, Assr. to The Barrett Co. U.S.P. 1,338,037, 27.4.20. Appl., 8.12.17. Renewed 27.8.19.

A watery pulp of fibrous material is mixed with a large quantity of comminuted fusible waterproofing material, and the mixture is formed into a layer and heated sufficiently to soften the waterproofing material without evaporating the water.—A. B. S.

Bituminous concrete pavements and processes for making same. G. A. Henderson. E.P. 141,769, 21.11.18.

Cement; Apparatus for manufacturing —. M. L. Boillot and J. Daudignac. E.P. 141,142, 15.1.19.

SEE U.S.P. 1,332,422 of 1920; J., 1920, 337 A.

Cement; Method of manufacturing simultaneously hydraulic — and alkali from alkaliferous mineral substances and lime. E. W. Jungner, U.S.P. 1,338,021, 27.4.20. Appl., 31.7.18.

SEE E.P. 117,460 of 1918; J., 1919, 255 A.

Cement; Non-porous —. F. D. Mulligan. U.S.P. 1,338,033, 27.4.20. Appl., 2.6.19.

SEE E.P. 128,452 of 1918; J., 1919, 637 A.

[Heat-] insulating bodies of "moler"; Process of manufacturing —. A. Fenger-Grön. U.S.P. 1,338,566, 27.4.20. Appl., 29.3.19.

SEE E.P. 139,318 of 1919; J., 1920, 298 A.

Fireproofing. U.S.P. 1,338,322. See V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Ores and iron-making material; Valuation of —. C. H. Ridsdale. Iron and Steel Inst., May, 1920. [Advance proof.] 30 pages.

THE author has collected chemical and thermal

data from the use of which accurate valuations of ores etc. can be made. The type of pig the ore is best adapted to make must be determined and this grade adopted as the basis of valuation. Four examples are given of standard types and grades of pig, with the corresponding basicity of the slag. Ores should be compared on the same basicity of slag. Methods are described for calculating the quantities of ore, limestone, coke, etc., required and slag produced per ton of pig. The requirements of each constituent are calculated by preparing a balance sheet of it and the respective coke multiples obtained. The calculations are made first under ideal conditions and then corrections determined from practical considerations. Startling differences are noticeable in the value of various ores generally regarded as substantially equal, and examples are given showing that iron content is often little guide to value.—T. H. Bu.

Iron ore; Utilisation of titaniferous — in New Zealand. J. A. Heskett. Iron and Steel Inst., May, 1920. [Advance proof.] 14 pages.

THE titaniferous iron sands of New Zealand are low in ferric oxide, and require a greater percentage of direct reduction by carbon than more easily reduced ores. In experimental trials, using a hot blast, a briquetted 8:1 iron-sand-coal mixture was employed. The slags were fluid, but obstructions consisting of a solid mixture of coke and ferro-titanium with entrapped cinder prevented tapping. By tapping at short intervals the front of the hearth was kept clear, but the back side of the hearth became built up. Probably if the high-titanium ores contained sufficient gangue to make a self-fluxing burden, hearth troubles would be eliminated. A satisfactory method would be to mix titaniferous and non-titaniferous ores in proportions adapted to absorb the precipitated titanium-bearing alloy. Pig iron produced from titaniferous ores has a tendency to chill in ordinary sand castings, but is exceptionally strong and tough. The castings have a wonderful finish, but the iron is better adapted to steel production than to ordinary foundry work.

—T. H. Bu.

Grey cast iron of different grades; Strains in —. O. Banse. Stahl u. Eisen, 1919, 39, 313—316, 436—441, 596—600.

THE effect of the grade of the iron and the moisture content of the mould on the appearance of strains in square or round frames of grey cast iron with thin crossbars was determined. The strains produced diminish as the silicon content increases, and are smaller in castings produced in dry moulds than in those made in moulds of "green" sand.

Iron; Defects in electro-deposited —. W. E. Hughes. Iron and Steel Inst., May, 1920. [Advance proof.] 18 pages.

THE defects which may occur in electro-deposited iron include pin-holes, surface roughness, inclusions of foreign matter, and cracks or incipient cracks, especially where different structures meet. Pin-holes are due to the evolution of hydrogen, and are minimised by using low current densities and slowly-formed deposits. Surface roughness is due to suspended matter in the solution or possibly to the solution becoming alkaline around the cathode. The inclusions which may occur in electro-deposited iron are iron oxide, hydrogen, and carbon from the anodes or electrolyte, especially perhaps those containing oxalates. Electrolytic iron could be used to a much greater extent than at present for building up worn parts of machines provided the process were under scientific control.—F. C. Th.

Iron; Effect of various elements on the electrical resistivity of —. A. L. Norbury. Iron and Steel Inst., May, 1920. [Advance proof.] 19 pages.

BENEDICKS' method of plotting the values of the resistivity of quenched carbon steels on a straight line and extrapolating to 7.6 microhms as the value for pure iron cannot be correct. Recent investigations give 9.9 microhms as the resistance of pure iron, and show that equi-atomic solutions of different elements do not give exactly the same resistivity in iron. The electronic properties of the element exert an influence.—F. C. Th.

Iron and mild steel; Microstructure of — at high temperature. H. S. Rawdon and H. Scott. Amer. Inst. Min. Eng., Feb., 1920. Chem. and Met. Eng., 1920, 22, 787—791.

THE microstructure of pure, electrolytic iron (C 0.03%, S 0.002%, Si 0.009%; Ac2 768°, Ac3 910°, Ar2 768°, Ar3 900° C.) was developed by the heat-relief method (heating in vacuo at high temperatures). Specimens of about 3 g. were mounted on the end of a platinum platinum-rhodium thermocouple and heated in a differentially heated, evacuated furnace to 950°, 880°, and 700° C. respectively, the samples being kept at the maximum temperature for 30 mins. and cooled at the rate of 0.15° per sec. The structure is indicated by photographs. A matt surface is sometimes produced by volatilisation, and a roughened surface may be caused by buckling, due to volume change. A low-carbon steel (C 0.18%, Si 0.007%; Ac1 738°, Ac2 769°, Ac3 840°, Ar3 792°, Ar2 769°, Ar1 700° C.) was treated similarly to the iron, the temperatures being 950°, 760°, and 700° C. respectively. A strongly-marked change occurred just above the A1 transformation, diminishing on heating to higher temperatures owing to increased rate of diffusion of carbon. At high temperatures the results obtained by etching are more indicative of the structure of the interior than at temperatures just above A1. Volatilisation of iron from the surface is very slight.—W. J. W.

Steel; Elastic development of —. S. Cornell. Chem. and Met. Eng., 1920, 22, 677—680.

RESULTS are given of the changes in hardness and tensile strength produced by varying the heat treatment of various grades of steel used in the manufacture of rifle components and bayonets. Thus a large number of bayonets which, after being hardened at 1500° F. (820° C.) and drawn at 575° F. (300° C.), had failed to pass the specified mechanical tests, were "stiffened" by re-drawing at 750° F. (400° C.), and then gave satisfactory results. The steel contained about 0.86% C, with Ar 1260° F. (680° C.), and Ac 1335° F. (725° C.).

—T. St.

Steel; Distribution of phosphorus in — between the points Ac1 and Ac3. J. H. Whiteley. Iron and Steel Inst., May, 1920. [Advance proof.] 24 pages.

IN a series of hypoeutectoid steels with the phosphorus varying between 0.006 and 0.12%, heated to and maintained between the Ac1 and Ac3 points, it was found that the phosphorus contained in the γ -iron partly diffuses into the adjacent ferrite owing to its lower solubility in the former, due probably to the presence of dissolved carbides. Below 650° C. the phosphorus diffuses very slowly in ferrite, but above this temperature the velocity increases, being quite rapid above 850° C. The phosphorus diffuses in γ -iron in a similar manner, but not so fast as the carbon. Heating steels slowly through the critical range produces a cellular structure also due to the movement of the phosphorus, the temperature at which this structure

is formed depending on the carbon content. By heating sections of steel to temperatures just below Ac3 the excess of phosphorus contained in micro ghost lines is completely removed, but as the lines still persist, though in a less pronounced manner, their presence appears to be due to the uneven distribution of some other substance besides phosphorus.—T. H. Bu.

Steel ingots; Micro-pipes of —. G. Charpy. Comptes rend., 1920, 170, 306—311.

MICRO-PIPES occurring in steel ingots are most frequently observed in the centre of the ingot, and it is suggested that this is due in part to the meeting of the dendritic crystals coming from opposite walls of the ingots. These micro-pipes may be distinguished from the *retassures en chapelets* (prolongations of the ordinary "pipe") by their sinuous form and their general orientation. They appear to be the origin of local faults which may, in some cases, become serious. Working the metal hot may, under certain conditions, diminish these faults, or, on the contrary, may materially accentuate them. Thus forging or rolling into plates may have a considerable effect on the quality of the products.

—W. G.

Steel; Effect of initial temperature upon physical properties of —. J. H. Andrew, J. E. Rippon, C. P. Miller, and A. Wrang. Iron and Steel Inst., May, 1920. [Advance proof.] 82 pages.

THE volume change which takes place in steel on passing through the transformation points is composed of two changes, working in opposition, the allotropic change and that due to the carbide. The latter is not complete at the ferrite line in steels of high carbon content. Quenching merely stereotypes the condition of the carbide. At and above the normal transformation temperature the carbide dissociates. Nickel-chrome steels behave in a similar manner except that the time factor is of a lower order. Martensite is probably a solution of the dissociated carbide in α -iron, and in those cases where the austenite to pearlite change occurs at the normal Ar1 point martensite is not an intermediate product. The hardening which takes place when an austenitic steel is tempered is ascribed to the association of the carbide with the resulting occurrence of the allotropic change. Electrolytic separation of the carbide in nickel-chrome steel in which the transformation has not taken place at the normal temperature shows a complete absence of chromium, and the abnormalities of nickel-chromium steels are probably due to iron carbide which has been rendered more stable by the special elements present in solution. When quenched from 1000° C. the iron in plain carbon steels is entirely in the α -state; in alloy steels γ -iron is present in varying proportions, particularly when the carbon content is high, while in extreme cases α -iron is absent. A novel form of dilatometer used in the work at high temperatures is described.—F. C. Th.

Steels; Embrittling effects of cleaning and pickling upon carbon —. S. C. Langdon and M. A. Grossman. Trans. Amer. Electrochem. Soc., 1920, 305—338. [Advance copy.]

REMOVAL of grease by organic solvents or alkali, even when the steel was electrolytically cleaned (as cathode) in a solution of alkali, did not affect the strength of the steel, but vigorous pumice brushing caused a reduction in strength due to the scratching of the surface. Sand-blasting caused appreciable reduction in the ability of rods in the "raw" state and after being "normalised" to withstand the alternating stress test, particularly with steels of low carbon content. Steel with 0.98% C when hardened and tempered was, however, improved by sand-blasting. Cold-rolled strip was only slightly reduced in ductility. The effect of sand-blasting is probably one of hardening due

to cold work. Pickling for 5 min. in 2N sulphuric acid at 50° C., or in nitre-sulphuric solution, hydrochloric acid, or hydrofluoric acid, of equivalent acid concentration, produced in all cases similar embrittling effects. With rods, stock as received was rendered increasingly brittle with increasing carbon content up to about 0.54% C. In high carbon steel the effect was less marked. The brittleness produced in normalised rods decreased, but in hardened and tempered rods increased with increasing carbon content. The effects were almost independent of size from $\frac{1}{8}$ in. to $\frac{1}{2}$ in. in diam. With cold-rolled strip steel the brittleness produced was greater in proportion to the amount of "work" which the specimen had received, and increased with the thickness of the strips. In general, increasing the temperature of the pickling bath from 0° C. to 50° C. caused increased brittleness, but above 50° C. increasing the temperature had little effect. Brittleness increased with the time of immersion markedly up to 5 min., after which the increase was more gradual. Nitric acid produced practically no brittleness. Electrolytic pickling caused about the same degree of brittleness as was produced by simple immersion in the same acid for the same period. Electrolytic "pickling" as anode or cathode in a neutral solution of sodium sulphate produced no brittleness, although hydrogen was evolved vigorously upon the cathode surface. Specimens which had been rendered brittle by pickling became less brittle on standing, slowly at the ordinary temperature and more rapidly at higher temperatures. It is concluded that pickling produces a permanent effect, caused by the roughening of the surface, and a temporary effect, due probably to occlusion of hydrogen.—T. St.

Chromium steels; Structure of —. J. H. G. Monypenny. Iron and Steel Inst., May, 1920. [Advance proof.] 26 pages.

In the case of many of these steels it is the carbide of the cementite which first goes into solution at the change point on heating, the pearlite carbide passing into solution at a higher temperature, which increases as the chromium and carbon contents of the steel are raised. The carbon contents at the eutectoid points are much lower for the chromium steels than for plain carbon steel, the following figures illustrating the influence: 2% Cr, 0.66% C; 6% Cr, 0.5% C; 10% Cr, 0.35% C; 15% Cr, 0.2% C. In the case of a steel with 1.1% C and 2.8% Cr, in the quenched condition, when etched with sodium picrate two distinct carbides are to be seen; one of these, presumably cementite, is darkened, while the other is comparatively unattacked. Austenite in these steels is transformed into martensite by cold-work and by tempering, but attempts to obtain martensite directly by slower cooling were only partly successful, mixtures of martensite and troostite always being obtained. Steels quenched from 1200° C. are austenitic if they lie to the right of a straight line running from 0.6% C and 15% Cr to 1.05% C and 5% Cr and completely martensitic if to the left of a parallel line from 0.45% C and 15% Cr to 0.8% C and 5% Cr. Between these two lines the structure is austenite-martensite. A steel rendered austenitic by quenching, if it lies near the limiting line, is spontaneously transformed in part into martensite. In the austenitic steels the effect of tempering is comparatively small up to 500° C. when a sudden and considerable increase of hardness sets in. There do not appear to be any distinct areas in the diagram in which different double carbides exist (cf. Murakami, J., 1919, 257 A). The effect of increasing chromium content is progressive. The rate of diffusion of carbides in chromium steel is much slower than in plain carbon steel, with the result that much longer time is required at a given temperature to produce equilibrium.—F. C. Th.

Iron-chromium-carbon steels; Properties of —. 1. *Thermal analysis.* C. A. Edwards, H. Sutton, and G. Oishi. Iron and Steel Inst., May, 1920. [Advance proof.] 44 pages.

In presence of chromium the carbon change point on cooling is raised considerably to an extent depending on both the chromium and the carbon contents of the steel. With a constant carbon or chromium content the highest A₁ point is reached when the ratio of the chromium to the carbon is about 10:1. The annealed steels contain a definite double carbide, Fe₃C.Cr₃C₂. It is concluded that a carbide Cr₃C does not exist, but in the high chromium steels in the annealed state the carbide Cr₃C₂ is probably present. The steels can be divided into two groups—first, those with Cr/C less than 10, which possess similar hardening properties to ordinary steels; and, secondly, those with a higher ratio of chromium to carbon which have lower critical cooling velocities and which can therefore be air-hardened. The boundary line is not sharply defined and depends on the content of silicon and manganese in the steel. Possibly the air-hardening properties of these steels may be due to the dissociation of Cr₃C₂ into Cr₃C₂ and free chromium.

—F. C. Th.

Chromium steels. II. Effect of heat treatment on electrical resistivity. C. A. Edwards and A. L. Norbury. Iron and Steel Inst., May, 1920. [Advance proof.] 36 pages.

In the annealed condition the addition of chromium to steel has little or no influence on the electrical resistance until the amount is increased above 4.3 times the carbon content. It is concluded that within this range of composition virtually all the chromium is in combination with the carbon, the carbide existing as isolated patches in a chrome-free ferrite. With higher contents of chromium the resistance is increased by 3.75 microhms for each 1% Cr. The change occurs at a composition corresponding to the formula Fe₃C.Cr₃C₂, and this double carbide is probably present in these alloys. Excess of chromium above that required to form this carbide is present partly in solid solution in the ferrite and partly as a new carbide, probably Cr₃C₂. Quenching experiments show that there is a second critical range of temperature for these steels at about 1000° C., and the maximum resistance is not attained until this has been exceeded. During tempering the loss of resistance takes place in two stages around 200° C. and 500° C. respectively. At the lower temperature the carbide which falls out of solution may be the ordinary iron carbide, whereas that which is deposited at the higher temperature may be either Cr₃C₂ or Cr₃C₂. So far as the chromium in solid solution is concerned, an increase of 1% raises the resistivity by 5.1 microhms per cm. cube.—F. C. Th.

Nickel-chrome and other steels; Brittleness in —. F. Rogers. Iron and Steel Inst., May, 1920. [Advance proof.] 5 pages.

The paper contains the data on which the earlier conclusions were based (J., 1919, 774 A).—F. C. Th.

High-speed steel containing chromium and tungsten; Constitution, hardening, and tempering of —. K. Honda and T. Murakami. Iron and Steel Inst., May, 1920. [Advance proof.] 11 pages.

In the annealed state a high-speed steel containing about 5% Cr and 18% W with 0.6% C consists of iron with tungstide in solution, free tungstide and the carbides Cr₃C and WC in the free state. If the steel be heated above the A₁ point the carbides dissolve in the austenite and undergo the following changes: 2Cr₃C=Cr₃C₂+5Cr and 2WC+3Cr+4Fe=2Fe₃W+Cr₃C₂. The higher the temperature the

greater the extent to which these changes proceed from left to right, but on normal cooling from the high temperature the reverse change takes place but slightly, with the result that the carbides and tungstide remain in solid solution. The separation of the carbides on tempering produces an increase in the magnetisation, whence this property affords a ready means of studying the changes in the solution. The tempering takes place in two stages at about 400° C. and 700° C. The self-hardening and the resistance to tempering depend principally upon the quantity of Cr_3C_2 in solution, and hence these properties increase with the carbon and chromium jointly. The maximum temperature attained, since it determines the extent to which the changes have occurred, also has an influence. Tungsten in high-speed steels diminishes the temperature at which hardening begins on cooling. Above 12% tungsten exists as fine globules of Fe_3W , and these, acting as saw teeth, increase the cutting efficiency rather than the hardness of the steel. The globules of tungstide are darkened by etching for 10 to 15 secs. in a cold alkaline solution of potassium ferricyanide. As cast the tungstide is found as a eutectic, but in the tool the globules must be uniformly distributed.—F. C. Th.

Assaying; Volatilisation losses in —. F. P. Dewey. Amer. Inst. Min. Eng., Feb., 1920. Chem. and Met. Eng., 1920, 22, 797—802.

Losses of gold, silver, and lead in assaying are not ascribable to volatilisation except in rare cases due to excessive temperature. They are largely due to dusting and other mechanical causes, the working up of very fine slimes tending to promote loss of this kind. Only minute quantities of precious metals are traceable in the fumes condensed in muffle flues. The drag exerted by one metal on a less volatile one, in cases where true volatilisation takes place, is slight.—W. J. W.

Micrometallurgy. [Copper-silicon and iron-silicon alloys.] S. Bogdan. Bul. Soc. Chim. Rom., 1919, 1, 60—72.

A MICROGRAPHIC study of copper-silicon and iron-silicon alloys showed that when the silicon content is very small the product is a homogeneous solid solution, but when the silicon content exceeds 1% crystalline compounds, probably silicides, are always obtained embedded in the solid solution. When the percentage of silicon present exceeds 4 two crystalline silicides can be isolated as well as silicon itself in both the crystalline and the amorphous state. Prolonged heating of the alloys at high temperatures facilitates the separation of their components. As the proportion of silicon present increases still further, the mass becomes more crystalline, the hardness increases, and the alloy becomes more brittle.—W. G.

Duralumin; Heat treatment of —. P. D. Merica, R. G. Waltenberg, and H. Scott. U.S. Bureau of Standards, Sci. Paper 347. Nov. 15, 1919. 41 pages.

WHEN duralumin (Cu 0—4.5%, Mg 3.5—0%, commercial Al the remainder) is quenched from between 250° and 500° C., and then aged between 0° and 200° C. the hardness and, especially at the lower ageing temperatures, the ductility increase; the actual value of these properties also increases with the temperature of quenching up to 520° C. This increase corresponds to the increase of CuAl_2 in solid solution in the aluminium. At 520° C. this compound melts as a eutectic, and the metal is spoilt. In order to obtain the best mechanical properties the alloy should be quenched from temperatures as near this as possible. By heating for 10—20 mins. at 510°—515° C., quenching in boiling water, and allowing to age at 100° C. for 5—6 days, maximum values for tensile strength and ductility

are obtained. To develop a higher proportional limit the alloy should be aged at 150° C. for 2—4 days. The composition of the most satisfactory alloy is Cu 3—4.5%, Mg 0.4—1%, Mn 0—0.7%, Al (99%) remainder. The alloy should be preheated to 500° C. before rolling and rolled at 450° C. It is suggested that the hardening observed on ageing is due to gradual precipitation of the compound CuAl_2 from solid solution in a colloidal form dispersed throughout the mass.—A. R. P.

Alloys of aluminium and magnesium with copper, with nickel, and with manganese; Mechanical properties and resistance to corrosion of rolled light —. P. D. Merica, R. G. Waltenberg, and A. N. Finn. U.S. Bureau of Standards. Tech. Paper 132, 1919. Pp. 13.

ALLOYS of the three series Al-Mg-Ni, Al-Mg-Mn, and Al-Mg-Cu were prepared with varying amounts, up to a total combined content of 4.5%, of the hardening metals, and cast into slabs 3.5 in. thick. These were then rolled down to 0.032 in. thick, the final rolling being in the cold, after annealing at 400°—450° C. Hardness and tensile tests were made on the sheets in the cold-rolled condition, after annealing at 422° C., and after heat treatment consisting of quenching from about 500° C. and allowing to stand or "age" for several days either at 110° C. or at room temperature. The Al-Mg-Cu alloys gave better tensile results in all conditions than those of the other series, and showed improvement under appropriate heat treatment, as compared with the annealed specimens. The Al-Mg-Ni alloys also were improved by heat treatment, but not to the same extent as the Al-Mg-Cu alloys. The Al-Mg-Mn specimens were not improved by heat treatment. In the annealed condition, and for equal additions, either with or without magnesium, copper had the greatest hardening effect, and manganese was more effective than nickel. The Al-Mg-Mn alloys in general resisted corrosion by salt water best, the heat-treated Al-Mg-Cu alloys being but little inferior. The latter alloys, in the cold-rolled and annealed states, were, however, the least resistant to corrosion of any of the alloys tested. Hard-rolled commercial aluminium corroded much more than any of the alloys. Annealed aluminium was more resistant than the hard-rolled metal, but did not compare favourably with most of the alloys.—T. St.

Zinc muffles; Colouring matter formed in old fragments of — and the changes due to muffle and furnace gases. O. Mühlhaeuser. Z. angew. Chem., 1919, 32, 53—56.

IN spite of the enveloping silicious skin, gases penetrate the walls of zinc muffles through pores and cracks. The various colours observed in the retort material and glaze are associated with the changing character of the gases and with the materials contained in the binding clay. The various colours observed and their causes are described.—T. H. Bu.

Lead; Hardening effects of various elements upon —. C. O. Thieme. J. Ind. Eng. Chem., 1920, 12, 446—448.

AN alloy of lead with about 1% of calcium has a Brinell hardness of about 15, but appears to lose its hardness when re-melted several times owing to oxidation of the calcium. Addition of sodium to lead increases the Brinell hardness up to about 8 at 0.8% Na (the maximum dissolved by the lead); the alloy loses its hardness after re-melting several times. Arsenic (0.5 to 1%) hardens and increases the fusibility of lead. An alloy of lead with 2% Ni is resonant. Alloys of lead with antimony in amounts exceeding 18—20% are too brittle for practical use. Up to the eutectic point (13% Sb) the hardness increases approximately one Brinell number for each 1% Sb. An alloy of 90% Pb and

10% Sb has a hardness of about 14, and an alloy containing 18% Sb a hardness of about 16. By adding 1% phosphor-copper to this alloy the hardness is increased to about 24, but the product is more brittle, loses its lustre more rapidly than the lead-antimony alloy, and is difficult to cast, since it readily segregates. Magnesium also hardens the alloy of lead and antimony, 0.5% increasing the hardness to 17.5. This alloy soon loses its lustre. The addition of 5% tin to the lead-antimony alloy increases the hardness to about 19, and a further addition (up to 8%) gives a fine-grained alloy with hardness of about 20. It retains its lustre fairly well and takes a good polish. Each increase of 1% Sn increases the hardness by about 1 Brinell number, but such alloys should not contain more than about 10% Sn unless the antimony is correspondingly increased. An alloy of lead with about 0.5% Mg has a hardness of 15; it does not oxidise rapidly, and therefore retains its lustre and hardness on re-melting. The addition of mercury (up to 7%) increases the hardness of lead.—C. A. M.

Tungsten; Estimation of —. R. F. Heath. Chem. Trade J., 1920, 66, 629.

The finely powdered ore (0.5 g. if low grade or 0.2 g. if a concentrate) is fused with 5 g. of sodium peroxide in a nickel crucible. In the analysis of steels and mattes the sample is first dissolved in hydrochloric and sulphuric acids, the solution evaporated to dryness, and the residue fused with peroxide. In either case the melt is leached, the insoluble matter filtered off, the filtrate diluted, and an aliquot part treated with 6–7 c.c. of the test solution (10.3 g. of stannous chloride and 2.1 g. of stannic chloride dissolved in 100 c.c. of dilute acetic and 40 c.c. of syrupy phosphoric acid) and 3–4 c.c. of strong sulphuric acid; after standing 3–4 mins. the blue colour produced is compared with that of a standard solution of tungsten treated in the same way.

—A. R. P.

Tungsten; Analysis of —. J. Erlich. Ann. Chim. Analyt., 1920, 2, 102–103.

Two grms. of the sample is heated for 2 hrs. with 100 c.c. of hydrochloric acid and 30 c.c. of 30% ferric chloride solution; the insoluble portion is washed with ammonium nitrate solution, ignited, and weighed. The ignited residue is fused with sodium bisulphate, the melt dissolved in ammonia, the solution oxidised with bromine, treated with ammonium carbonate, and the precipitate (impurities) collected and weighed. The difference between this and the first weight gives the amount of tungstic oxide present. It may be found necessary to repeat the fusion with sodium bisulphate in order to separate all impurities from the tungstic oxide.

—W. P. S.

Amalgams; Electro-endosmosis and the preparation of solid alkali —. S. B. Frank and J. R. Withrow. J. Amer. Chem. Soc., 1920, 42, 671–675.

Solids amalgams of potassium and strontium are obtained by electrolysis saturated solutions of potassium chloride or sulphate or strontium chloride respectively between a platinum anode and a mercury cathode. A porous cell 40×80 mm. containing about 50 g. of mercury is suspended so that it dips about 5–10 mm. into the saturated solution, contacts are made in the usual way, and the current passed for about 1 hr., using a voltage of 25. (Cf. J.C.S., ii., 350.)—J. F. S.

Sodium amalgam; Preparation of — in flakes. A. D. Hirschfelder and M. C. Hart. J. Ind. Eng. Chem., 1920, 12, 499.

Hot liquid sodium amalgam is slowly poured into a jar containing xylene or kerosene which is being rapidly stirred by an electric stirrer. The fine flocculent deposit is dried on a porcelain plate in a current of air, and may then be readily pulverised.

—C. A. M.

[Hardness of metals.] “Ball test.” T. Baker and T. F. Russell. Iron and Steel Inst., May, 1920. [Advance proof.] 16 pages

If L is the load in the Brinell test, V the volume of the indentation, and d the diameter of the impression, then $L = bV^m$ and $L = ad^n$ where a , b , n , and m are constants. It is shown that n is a measure of the cold-work put upon the material, and in the completely work-hardened state is equal to 2. As this condition is approached the curves expressing the relation of the Brinell number to the load used in the test becomes more and more horizontal. By plotting in the stress-strain curve the stress against the contraction of area divided by the reduced area a curve is obtained which is free from the arbitrary character of the ordinary test. When this is done there is no point of inflexion in the upper part of the curve comparable with that usually shown. Where it is not possible to use the 10 mm. ball, the following procedure may be adopted. If the ratio of the load to the square of the diameter of the ball is kept constant, $D, d/d$, is the diameter of impression under standard conditions where D is the diameter using a ball of diameter d , and d is the normal 10 mm.—F. C. Th.

Tin and antimony. Kling and Lassieur. See XXIII.

PATENTS.

Carbonising of steel; Mixtures for the — during the case hardening process. The District Chemical Co., Ltd., and J. B. Hoblyn. E.P. 141,802, 20.1.19.

The mixture contains in addition to the commonly used ingredients oxalates of the alkalis or alkaline earths and organic nitrogen compounds, e.g., primary, secondary, or tertiary aromatic amines.

—T. H. Bu.

Steel; Manufacture of —. G. A. Jarvis. E.P. 141,956, 4.6.19.

The main portion of scrap is preheated to about 800° C. and then introduced into a furnace containing a smaller portion of scrap, having an excess of carburising material to give the desired carbon content to the full charge.—T. H. Bu.

Steel scrap; Method of utilising —. G. M. Muntz and E. M. Roubieu, Assrs. to Tropenas Co. U.S.P. 1,336,236, 6.4.20. Appl., 25.6.17.

In melting steel scrap in a cupola, a melt is first produced with metal of lower fusing point than the steel scrap, and then successive charges comprising steel scrap largely in excess of metal of lower melting point are melted, the sizes of the charges being so proportioned that there is a practically continuous rain of molten metal at high temperature to the hearth of the cupola.

Mercury; Process for decomposing — and obtaining radium and gold. G. B. Perez. E.P. 126,961, 12.5.19. Conv., 15.5.18.

MERCURY is introduced into a central depression in a vacuum tube similar to a Crookes tube and subjected to the electric discharge from an induction coil between two pointed electrodes arranged a short distance above the mercury and inclined downwards towards its surface. The mercury is said to be decomposed into metallic gold and a radio-active substance.—T. H. Bu.

Iron; Bessemerising —. R. S. McCaffery. U.S.P. 1,338,655, 27.4.20. Appl., 13.11.18.

MOLTEN iron, without basic additions, is bessemerised in a converter having an acid lining except

at the part where basic products may be formed during the process.—W. E. F. P.

Iron; Production of — in an electric furnace. G. J. Stock. U.S.P. 1,338,881, 4.5.20. Appl., 13.2.20.

ORE is fed steadily to the furnace at a rate sufficient to balance the electrical energy consumed in melting the charge and raising it to the required temperature, whereby the latter is maintained substantially constant.—T. St.

Iron resistant to cutting; Process for the production of —. Deutsch-Luxemburgische Bergwerks- und Hütten A.-G., and H. Lütke. G.P. 317,671, 6.6.18.

IRON containing phosphorus (Thomas iron, old cast iron etc.) is smelted with scrap in a basic furnace under a layer of slag, the phosphorus content of which has been increased to such an extent that it can absorb no more from the melt. In the preparation of the melt the phosphorus content can be regulated by the addition of more slag on the one hand, or of lime on the other. Since the molten metal is covered during the whole process with a layer of slag, no injurious gases can be absorbed from the flue gases; and further, since the smelting process is carried so far that the phosphatic slag is in equilibrium with, and therefore non-reactive towards, the iron, a product is obtained particularly free from gas and suitable for casting. The metal is especially resistant to cutting and suitable for nuts etc.—G. F. M.

Iron and steel; Determination of the content of non-ferrous constituents, especially carbon, in —. B. D. Enlund. G.P. 319,255, 22.11.18.

A MEASURE of the total non-ferrous elements in iron and steel is given by the electrical resistance of the fully hardened specimen, and of the carbon content by the difference in electrical resistance of the specimen in the hardened and unhardened states. The increase in resistance with hardening is greater when the iron contains only small amounts of silicon and manganese, and standards with varying amounts of these elements are therefore necessary when the carbon content is high.

—T. St.

Nickel salt; Method and apparatus for electrolysing a solution of a —. C. Heberlein. E.P. 141,766, 29.10.18.

NICKEL is dissolved from ore, matte, or the like, by means of an acid until a liquor of not more than 2% acidity is obtained, which is circulated as catholyte through a number of electrolytic vats in series. As an anolyte nickel salt solution is used. When the acidity of the anolyte is sufficient for dissolving nickel from fresh ore, matte, etc., it is withdrawn to be used for preparing fresh nickel salt solution. The catholyte is returned into circulation with fresh nickel salt solution, the acidity being not more than 2%. The process is thus continuous and cyclic. Insoluble stationary anodes and stationary cathodes are used.—T. H. Bu.

Nickel; Process for recovering — from cupro-nickel alloys, scrap, and the like. A. McKechnie, and McKechnie Bros., Ltd. E.P. 142,310, 10.5.19.

THE charge of alloy or material containing metallic copper or nickel is rapidly melted on the hearth of a basic reverberatory furnace and maintained under heat while air is blown through; or the molten charge may be bessemerised in a basic converter. The treatment by either method is continued until the whole or part of the nickel content is volatilised. The nickel is recovered from the furnace fume in the form of nickel oxide.—J. W. D.

Nickel; Preparation of finely divided — for catalytic purposes by reduction. H. Schlink and Co. G.P. 318,177, 5.8.16.

THE material to be reduced is passed downwards in the form of powder over a number of electrically heated plates arranged in tiers, whilst the reducing gas, for example hydrogen, is led in from below.

—E. H. R.

Lead; Dezincing of —. H. Harris. E.P. 142,315, 16.5.19.

MOLTEN lead is brought into intimate contact with a molten mixture of either an alkali chloride and caustic alkali, or an alkali chloride and zinc chloride in presence of oxygen.—J. W. D.

Lead; Refining of —. H. Harris. E.P. 142,398, 16.5.19.

MOLTEN lead partially oxidised by means of an air spray is charged into a distributor from which it issues in the form of thin streams and is passed through a suitable molten reagent (e.g., common salt) until the impurities most easily removed by the reagent (e.g., copper) disappear from the lead. The molten metal is then passed through a fresh molten reagent to remove other impurities (e.g., arsenic, tin, and antimony).—J. W. D.

Metallurgical method and apparatus. Production of lead. G. F. Greenwood. U.S.P. 1,338,439 and 1,338,440, 27.4.20. Appl., 19.7. and 23.7.18.

LEAD sulphide ores are roasted in an electric furnace so constructed that the sulphur dioxide is withdrawn by suction and air is drawn through the charge. The roasted ore is transferred to an electric reducing furnace and reduced by carbon and heat with exclusion of air, at a temperature and pressure less than atmospheric, favouring the production of carbon monoxide. The carbon monoxide is removed and burned to carbon dioxide in a gas engine, which is used to generate an electric current for heating in the reducing reaction.

—J. W. D.

Alloy; Electric-resistance —. W. A. Scheuch, Assr. to Western Electric Co. U.S.P. 1,337,276, 20.4.20. Appl., 3.8.17.

THE alloy contains more than 50% Ni, $1\frac{1}{2}\%$ or more Co, and titanium.—T. H. Bu.

[Alloy.] *Composition of matter and process of making same.* H. B. Blumenberg, jun., Assr. to F. Blumenberg. U.S.P. 1,338,279, 27.4.20. Appl., 23.2.17.

A METAL and a compound of an element of the nitrogen group, bismuth and nitrogen excepted, capable of generating a volatile hydride on contact with water, are fused together by means of an electric current of low voltage.—J. W. D.

Alloy. F. Milliken, Assr. to F. Milliken, S. F. Weaver, and J. M. Repplier. U.S.P. (A) 1,338,517 and (B) 1,338,518, 27.4.20. Appl., 11.12.18.

THE alloys contain 50–60% Cu, 26–34% Ni, 4–8% Fe, 7–11% Zn, the metals being in a deoxidised state, and in addition (A) contains tungsten and (B) chromium.—T. H. Bu.

Alloy. G. L. Van Allen, Assr. to Baker and Co., Inc. U.S.P. 1,339,009, 4.5.20. Appl., 21.9.18.

THE alloy contains Au 64%, Ag 18.75%, Cu 9%, Pd 8%, and Al 0.25%.—B. M. V.

Metals; Process of melting and melting and reducing —. M. H. Bennett, Assr. to Scovill Manufacturing Co. U.S.P. 1,337,305, 20.4.20. Appl., 9.2.20.

NON-FERROUS metals of high thermal conductivity are melted in an electric furnace having a closed furnace chamber. An electric current of high

amperage is delivered to the charge through a number of electrodes in such manner that a magnetic field is set up in the melting metal which is thereby caused to circulate. The distribution and adjustment of the current are designed to promote uniform heating, undue volatilisation of the metal around the electrodes being prevented by the circulation of the charge. Any metal volatilised is condensed and returned to the melting chamber.

—W. E. F. P.

Metallic-arc welding. J. Churchward, Assr. to Wilson Welder and Metals Co. U.S.P. 1,337,543, 20.4.20. Appl., 14.1.20.

An electrode for electric arc welding consists of a homogeneous austenitic steel containing more than 12% Mn.—T. St.

Furnace; Metallurgical —. S. M. Howell. U.S.P. 1,337,703, 20.4.20. Appl., 1.7.19.

A combustible mixture of gas or hydrocarbon vapour and air is passed first through a mixing chamber containing metallic chips, then through a perforated plate of refractory material which retains the chips in place, into the furnace chamber.

—B. M. V.

Melting-furnace; Electrically heated —. L. C. Harvey. U.S.P. 1,337,839, 20.4.20. Appl., 11.11.19.

The melting chamber is substantially cylindrical in shape, and has an open upper end and a closed curved bottom. A heating element extends into the open end. The chamber is supported in an inclined position, and means are provided for rotating it about the longitudinal axis.—T. St.

Open-hearth furnace. J. C. Cromwell and W. M. Mehlhorn. U.S.P. 1,338,288, 27.4.20. Appl., 26.6.17.

THREE regenerator units are situated at each end of the furnace, the flue connexions and controlling means for the connexions being arranged to permit either one or both of two units to be connected in series with the third unit, or to be connected parallel with each other to the exclusion of the third unit.—J. W. D.

Roasting furnace; Gas-fired —. Donnersmarchhütte Oberschlesische Eisen- und Kohlenwerke A.-G. G.P. 317,039, 17.7.18. Addn. to 310,283 (J., 1919, 641 a; 1920, 69 a).

A NETWORK of horizontal girders is arranged in the zone where the water of constitution is expelled to obviate irregularities in the working of the furnace. By the retention of the sloping plates the dust can again be separated in the upper chambers. To facilitate the passage of the ore a space is provided above the top layer of girders in which a movable stirrer can be operated.—W. J. W.

Metals; Recovery of — from their ores. H. V. Welch, Assr. to International Precipitation Co. U.S.P. 1,338,271, 27.4.20. Appl., 11.8.17.

THE ore is heated with a suitable reagent to produce a fume containing the metal, the gases containing the fume are brought into contact with a liquid capable of dissolving the metal, and the latter is precipitated electrically from the fume. The metal is subsequently separated from the solution and the liquid used to act on a fresh quantity of fume.—J. W. D.

Zinc alloy. H. Goldschmidt and K. Müller, Assrs. to The Chemical Foundation, Inc. U.S.P. 1,338,826, 4.5.20. Appl., 19.4.17.

A ZINC alloy containing from 0.25 to 4.0% Mn and at least 6.9% Cu+Al, the ratio Cu:Al being about 3:1.—W. E. F. P.

Coating metals; Process for — by spraying with finely-divided metal. Method of obtaining adherent deposits by the spraying process. Process for improving the adhesion of metal coatings. Metalliserator G.m.b.H., in liq. G.P. (A) 318,460, 23.3.15, (u) 318,461, 10.4.15, and (c) 318,462, 1.12.15.

(A) The metal to be powdered and sprayed is first given a coating of fat or vaseline containing reducing substances in a colloidal condition, e.g., gelatin, glue, or "oidlag" or "aquadag" (colloidal graphite). (u) The metal to be coated is first given a thin layer of an easily fusible metal, such as tin or Wood's alloy, and the hot coating metal is then sprayed on to this, so that it melts and thus forms a binder. (c) The metal to be coated is heated to such a temperature that it is alloyed with or welded to the spraying metal. For example, vessels to be coated with lead are heated to 320° C., and iron is heated to 250°–300° C. or copper to 500° C. prior to coating with zinc.—A. R. P.

Non-oxidising atmosphere; Method of heating in a — with avoidance of the production of mill scale. F. K. Meiser. G.P. 319,277, 3.1.18.

THE gaseous fuel used consists substantially of nitrogen and carbon monoxide, and is obtained by the removal of carbon dioxide from semi-producer-gas.—T. St.

Reheating furnaces; Construction of walls or partitions, more especially for use in continuous recuperative —. Wellman Smith Owen Engineering Corp., and A. V. Kemp. E.P. 141,981, 18.7.19.

Castings; Manufacture of [sound] —. H. Wade. From A. J. Griffith. E.P. 142,201, 4.2.19.

Ores and the like; Grading or concentration of —. F. G. Gasche. E.P. 142,246 and 142,389, 14.3.19.

Heat treatment of metals and apparatus therefor. F. G. Brettell. From Shead and Co. Iron Works. E.P. 142,358, 5.7.19.

Steel; High-speed —. High-speed tool-steel. W. L. E. Eilender, Assr. to The Chemical Foundation, Inc. U.S.P. 1,337,209-10, 20.4.20. Appl., 6.3.17.

SEE G.P. 309,175 and E.P. 104,670 of 1916; J., 1919, 908 a; 1917, 967.

Magnet-steel. K. Honda, Assr. to Sumitomo Chukosho, Ltd. U.S.P. 1,338,132-4, 27.4.20. Appl., 22.10.17.

SEE E.P. 118,601-2 of 1918; J., 1919, 726 a.

[Steel alloy] objects having great strength and great resistance against the action of acids. B. Strauss, Assr. to The Chemical Foundation, Inc. U.S.P. 1,339,378, 4.5.20. Appl., 25.6.13. Renewed 6.8.19.

SEE E.P. 13,415 of 1913; J., 1914, 697.

Alloys. Cooper Research Co., Asses. of H. S. Cooper. E.P. 120,565, 15.10.18. Conv., 15.10.17. SEE U.S.P. 1,251,987 of 1918; J., 1918, 186 a.

Ores; Treatment of fine —. Dorr Co., Asses. of D. S. McAfee. E.P. 135,844, 24.11.19. Conv., 16.8.18.

SEE U.S.P. 1,305,817 of 1919; J., 1919, 511 a.

Electrochemical precipitation of metals. E. R. Holden. Reissue 14,849, 27.4.20, of U.S.P. 1,269,565, 11.6.18. Appl., 30.10.19.

SEE J., 1918, 589 a.

Zinc solutions; Purification of —. S. Field, Assr. to The Metals Extraction Corporation, Ltd. U.S.P. 1,331,334, 17.2.20, and 1,337,058, 13.4.20. Appl., 21.5.19 and 26.9.19.

SEE E.P. 138,946 and 138,954 of 1918; J., 1920, 371 A.

Ores; Concentration of —. E. Edser and S. Tucker, Assrs. to Minerals Separation North American Corp. U.S.P. 1,337,548, 20.4.20. Appl., 3.7.18.

SEE E.P. 121,303 of 1917; J., 1919, 78 A.

Solder; Manufacturing —. E. E. P. J. P. S. J. de St. Laurent, Assr. to Aluminium Solder Co. U.S.P. 1,333,966, 4.5.20. Appl., 22.9.16. Renewed 11.11.19.

SEE E.P. 13,856 of 1915; J., 1916, 1264.

Coke for blast-furnaces. G.P. 319,550. See 11. A.

Oxidising tin and zinc. E.P. 142,157. See VII.

Zinc wire. G.P. 317,355. See XI.

XI.—ELECTRO-CHEMISTRY.

Standard cell; A lead —. M. G. Mellon and W. E. Henderson. J. Amer. Chem. Soc., 1920, 42, 676–689.

CELLS of the general type Pb.Hg/MSO_4 (sat.)/ $\text{Hg}_2\text{SO}_4/\text{Hg}$ in which M is sodium, nickel, manganese, cobalt, or zinc are constant and reproducible, and possess all the characteristics necessary for a standard cell. The EMF is nearly the same whatever sulphate is employed. The following values are recorded: sodium sulphate, 0.96466 volt; nickel sulphate, 0.96430; cobalt sulphate, 0.96478; manganese sulphate, 0.96478; and zinc sulphate, 0.96477 volt. (Cf. J.C.S., ii., 348.)—J. F. S.

Potassium ferricyanide. Brown and others. See VII.

Electrolytic potentials. Pick. See VII.

Alkali amalgams. Frank and Withrow. See X.

PATENTS

Electrolytic reduction of organic compounds. Farbenfabr. vorm. F. Bayer und Co. G.P. 303,303, 20.3.17.

IN the electrolytic reduction of organic compounds using a cathode of lead or a lead alloy, the cathode is arranged in a horizontal plane with the active surface pointing downwards. The "lead oil," instead of remaining on the surface and causing loss of efficiency, falls to the bottom of the vessel, and is either oxidised in the anode chamber to lead oxide, or may be removed from the cell.—L. A. C.

Electrical conductor [zinc wire]; Producing an alteration of structure of an — by heating during winding. A.-G. Brown, Boveri und Co. G.P. 317,355, 11.12.17.

THE brittleness of zinc wire is reduced by passing a suitable electric current through the wire during the winding process.—J. S. G. T.

Electric furnaces; Control system for —. F. Thornton, jun., Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,338,408, 27.4.20. Appl., 29.6.18.

Electric heating furnaces; Polyphase —. E. C. R. Marks. From Armour Fertilizer Works. E.P. 142,283, 22.4.19.

SEE U.S.P. 1,317,328 of 1919; J., 1919, 912 A.

Electrolysing apparatus. C. N. Riiber, Assr. to Norsk Alkali A./S. U.S.P. 1,337,724, 20.4.20. Appl., 27.3.19.

SEE E.P. 125,935 of 1918; J., 1919, 945 A.

See also pages (A) 437, *Electrical precipitation* G.P. 316,790; *Electro-osmotic dehydration* (G.P. 317,403). 439, *Gaseous fuel* (U.S.P. 1,339,225). 440, *Substitute for mineral oils* (G.P. 299,691). 456, *Nickel solutions* (E.P. 141,766); *Electric resistance alloy* (U.S.P. 1,337,276). 457, *Metals from ores* (U.S.P. 1,338,271).

XII.—FATS; OILS; WAXES.

Coconut oil; Rancidity of Philippine —. G. A. Perkins. Philippine J. Sci., 1919, 15, 463–474.

THIRTY samples of edible coconut oil were kept for two years under varying conditions of light, air, etc. Oxygen was found to be necessary for the development of rancidity, but not of acidity. The action of light increased the hydrolysis of the fat in sealed bottles, but, on the other hand, all samples exposed to the air in darkness became rancid. Enzymes soluble in fat (but not those insoluble) appeared to have a slight effect, especially in increasing the acidity, but sterilisation of the oil had little, if any, beneficial effect. In the second stage of rancidity the free fatty acids were oxidised to an extent depending upon the amount of hydrolysis. This oxidation was accelerated by light and moisture, but light was not an essential factor. An oil with a low initial acidity remained sweet after exposure to air and light for two years.—C. A. M.

Saponification value [of fats]; Butyl alcohol as a medium in the determination of —. A. M. Pardee, R. L. Hasche, and E. E. Reid. J. Ind. Eng. Chem., 1920, 12, 481–482.

THE use of butyl alcohol (cf. J., 1920, 305 A) gives practically the same values as ethyl alcohol in the determination of the saponification values of the more readily saponifiable oils and fats, but in the case of substances which are more difficult to saponify (e.g., beeswax, spermaceti, and wool fat) the results are higher and more trustworthy after saponification for 30 mins. when butyl alcohol is used. Saponification is complete in 5 mins. in the case of cod liver oil when *n*-butyl alcohol is used as the medium.—C. A. M.

Fatty acids from cottonseed foots; Heat balance of a distillation plant for the recovery of —. J. Alsberg. J. Ind. Eng. Chem., 1920, 12, 490–493.

FRESH fatty acid stock was distilled with superheated steam in a coal-fired still, the volume in which was kept constant. Measurements of the temperature at the different points were made at intervals of 5 mins, the amounts of fatty acids and water in successive portions of the distillate weighed, the cooling water weighed, and an allowance made for surface evaporation; the steam was passed over a barometric condenser, and fixed gases were removed by means of a vacuum pump. In calculating the heat lost by radiation from the shell of the condenser the mean specific heat of the fatty acids was taken as 0.46. The heat balance was calculated from the data obtained, and the total heat above 32° F. per lb. of fatty acids distilled in one experiment was found to be 315.3 B.Th.U.; whence heat of vaporisation per lb. of fatty acids at 24.51 in. vacuum = $315.3 - 0.46(433.8 - 32) = 130.5$ B.Th.U. (433.8° F. is the temperature of the fatty acid vapour).—C. A. M.

Fatty acids; Preparation of — for soap from petroleum oil fractions of high molecular weight. L. Ubbelohde and S. Eisenstein. Mitt. deutsch. Forschungsinst. Textilstoffe, 1918 [4]. Chem. Zentr., 1920, 91, II., 22—23.

PARAFFIN w-x or heavy mineral oil or distillate is slowly oxidised when heated with air or oxygen below 200° C., and much more rapidly in presence of a catalyst such as finely-divided manganese dioxide or stearate. Purified paraffin wax heated for 12 hrs. with 1% of manganese stearate in the presence of oxygen and 2% of water yielded a nearly colourless product with saponif. value 200, and unsaponifiable matter 18—20%. It contained butyric, valeric, and probably caprylic acids, but no known solid fatty acids could be identified. It yielded good soaps.—C. A. M.

Catalyst; New — for hydrogenation [of oils]. W. D. Richardson. Chem. and Met. Eng., 1920, 22, 793—796.

FINELY-DIVIDED nickel, prepared mechanically by abrasion, constitutes an active catalyst for the hydrogenation of oils. The nickel in the form of shot is subjected to the action of abrasives such as ground quartz, pumice, carborundum, diatomaceous earth, etc., Abbé tumbling or pebble mills lined with porcelain being suitable for the operation. Adulteration with portions of the lining is innocuous, but a lining of nickel plates would be preferable. A semi-drying oil was first used in the mill, but water is better, as it produces a more finely-divided catalyst, is less liable to contain catalyst poisons, and forms a film of sub-oxide on the nickel which probably increases the activity if kept down to the narrowest limits. The activity curve of the catalyst shows a considerable rise at the commencement of hydrogenation, which lasts for some time before it reaches its peak and declines. Exposure of the nickel before grinding to temperatures as high as 1470° C does not appear to destroy the catalytic activity. Recovery of the nickel is effected by filter-pressing, then burning to remove fat, followed by mixing with a flux and melting in a furnace. The nickel is then recovered in the form of shot.—W. J. W.

Hydrogenation. Rideal. See XX.

PATENTS.

Oils; Process of removing or decreasing objectionable odours from treated —. J. E. Booge, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,337,339, 20.4.20. Appl., 13.6.18.

BLOWN cottonseed oil having an objectionable odour is blown with steam at 105°—130° C. under reduced pressure.—A. de W.

Fats and oils; Extraction of —. H. Bollmann. G.P. 303,846, 28.9.16.

THE raw materials, such as cereal grains, are placed in a number of containers having perforated bottoms arranged in a closed chamber. The containers are moved through the chamber in one direction, and the solvent falls freely through the containers in succession in the opposite direction, so that its fat content gradually increases.

—A. J. H.

Catalysts which have been used for fat-hardening; Revivifying —. C. and G. Müller, Speisefettfabr. A.-G. G.P. 319,332, 15.12.18.

A CATALYST prepared, e.g., from nickel borate, is regenerated after use by stirring for ½ hr. in warm refined olive oil, removing the oil first by mechanical means and then by heating with a fat-solvent such as ether, and finally heating the residue for ½ hr. at 430°—440° C. in hydrogen.—L. A. C.

Washing and cleaning; Process for —. K. Horkenbach. G.P. 317,796, 6.11.17. Addn. to 317,402 (J., 1920, 376 A).

FRESHLY-CUT peat is treated with a mineral acid and heated with simultaneous agitation in a current of air or oxygen containing also carbon dioxide, if necessary; the detergent is then finally produced as described earlier. The structureless lower layers of peat give a better product than that obtained from the upper layers.—D. F. T.

Detergent; Production of a pasty —. Chem. Werke München O. Barlocher. G.P. 318,151, 4.5.18. Addn. to 311,909 (J., 1920, 198 A).

SODIUM bicarbonate, suspended in water, is introduced slowly in a fine stream into a mixing or conveying apparatus in which a mixture of magnesium hydroxide and water-glass is circulated very rapidly. The sodium bicarbonate may be introduced, if necessary under pressure, into the suction pipe of the mixing apparatus in close proximity to the latter. Carbon dioxide may be substituted for sodium bicarbonate.—J. H. L.

Hydrogenating oils; Apparatus for —. G. Martin. U.S.P. 1,333,328, 9.3.20. Appl., 10.6.19.

SEE E.P. 139,239 of 1918; J., 1920, 305 A.

Oil from vegetable seeds, nuts, and the like; Extracting —. C. Downs, R. A. Bellwood, and T. W. Turnill. U.S.P. 1,338,909, 4.5.20. Appl., 21.1.19.

SEE E.P. 121,856 of 1918; J., 1919, 377 A.

Tyre-filling compositions. E.P. 142,416. See XIV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Resin from species of Xanthorrhoea not previously examined. E. H. Rennie, W. T. Cooke, and H. H. Finlayson. Chem. Soc. Trans., 1920, 117, 338—350.

THE Xanthorrhoea resins from Kangaroo Island and W. Australia, in common with other species, contain p-coumaric acid, either free or as an ester, and p-hydroxybenzaldehyde, and by steam distillation from a strongly alkaline solution the following additional substances were obtained:—From a red resin from Kangaroo Island—a fragrant liquid of vanillin-like odour and paeonol (2-hydroxy-4-methoxyacetophenone). From a yellow resin from X. Tateana—hydroxypaeonol in addition to the above two substances. From a red resin from X. Preissii—a fragrant liquid not identified, l-citronellol, paeonol, hydroxypaeonol, and a compound which was possibly methoxydiphenyl ether.—G. F. M.

PATENTS.

Coating substances to protect them or render them non-porous; Producing materials for —. C. A. Cleghorn, and The Gayner Pneumatic Co. E.P. 141,414, 10.1.19.

“TURKISH birdlime” (prepared from the fruits of Cordia myxa and Cordia latifolia) is dried at a temperature not exceeding 180° F. (82° C.) preferably in a vacuum, and then pulverised, conveniently at the same temperature. It can then be spread in a thin coating on canvas or other material to render it fluid-proof or applied as a liquid-tight jointing material; the powder is finally treated with water.—D. F. T.

Ink compound; Solid —. M. Tsutsumi. E.P. 141,631, 11.9.19.

EXTRACT of logwood and galls (60 pts.) is mixed with

sodium salicylate (0.2 pt.), potassium ferrous tartrate (30 pts.), purified gum arabic (7 pts.), and a suitable proportion of "Soluble Blue" or "Fast Blue." The powder may be pressed into cakes.

—D. F. T.

Ferric oxide; Manufacture of pure — F. Hemingway. U.S.P. 1,337,402, 20.4.20. Appl., 28.8.19.

AN intimate mixture of a ferrous salt and a dry alkaline substance is ground continuously so that the iron is largely converted into the ferric condition, and the resulting powder is calcined at the temperature necessary to produce the desired shade of colour.—D. F. T.

[*Pigment*] *colours; Manufacture of* — G. B. Palmer. U.S.P. 1,339,219, 4.5.20. Appl., 14.6.19.

THE "sulphonic acid-derived pulp known as worms" is filtered and then incorporated with a pigment carrier.—A. de W.

Phenolic condensation product and process of manufacturing. L. V. Redman, A. J. Weith, and F. P. Brock, Assrs. to Redman Chemical Products Co. U.S.P. 1,339,134, 4.5.20. Appl., 6.6.18.

A MIXTURE of a filler, a phenolic body, and a methyleneamine body adapted to "react anhydrously" therewith, is mixed with a fusible phenolic condensation product, together with sufficient methyleneamine substance to complete the conversion to a hard resistant product, and then subjected to moulding and heat treatment to produce a hard resistant body.—A. de W.

Surfaces or the like; Process for rendering — capable of being easily wetted. C. H. Boehringer Sohn. G.P. 318,217, 2.10.17.

A CHOLATE is added to a solution to be used for coating or silvering surfaces or to water colours, bronze colours, inks, or the like, to facilitate the wetting of the surface under treatment.—L. A. C.

Lamplack; Plant for manufacturing — E. Viertel, Assr. to The Chemical Foundation Inc. U.S.P. 1,338,268, 27.4.20. Appl., 17.8.15.

SEE G.P. 295,517 of 1914; J., 1917, 296.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Vulcanising natural and artificial caoutchouc and caoutchouc-like substances. L. Gaisman and J. L. Rosenbaum. E.P. 141,412, 10.1.19.

THE colour bases of basic coal tar dyes are capable of accelerating the vulcanisation of rubber with sulphur, and the vulcanised products are, in some cases, coloured throughout their mass.—D. F. T.

Tyre filling compositions. W. G. Wright. E.P. 142,416, 17.12.19.

SOYA bean oil (25½ pts.), cooled to about 32° F. (0° C.), is mixed thoroughly with calcined magnesia (2 pts.) and Venetian red (½ pt.); sulphur chloride (6½ pts.) is then added quickly, the resulting chemical reaction raising the temperature to 300°–400° F. (about 90°–150° C.). After a few minutes the mixture, having attained the necessary consistency and colour, is poured into moulds and cooled uniformly.

—D. F. T.

Rubber; Method and apparatus for treating [drying] — E. A. Wullenweber, Assr. to Morgan and Wright. U.S.P. 1,337,536, 20.4.20. Appl., 5.10.17.

RUBBER is dried by subjecting it to the action of segregated volumes of heated and humidified gas in circulation, which are successively removed when

excessively humid and replaced by freshly-conditioned volumes of gas, so that the rubber remains moist at the surface until it reaches a uniform, nearly dry condition. The process is carried out in a chamber in which gas is circulated intermittently until saturated, when it is exhausted intermittently and fresh gas supplied intermittently. Automatic means are provided for maintaining the supply and exhaustion of gas, and for maintaining the humidity and temperature inside the chamber.

—J. H. J.

Guttapercha substitute. G.P. 317,145. See V.

XV.—LEATHER; BONE; HORN; GLUE.

Tanning materials; Preparation of lightly chromed hide powder for the analysis of — G. Baldracco and S. Camilla. J. Soc. Leather Trades' Chem., 1920, 4, 88–92.

ORDINARY liming and subsequent deliming of the skins with hydrochloric acid has many disadvantages. The skins are given a preliminary unhairing, washed in running water for 24 hrs., then treated with a weak solution of ammonia, again washed, and sliced with a razor. The product is dried at ordinary temperatures, ground in a special mill to give a non-chromed hide powder, which is chromed with 2 g. of crystallised chromium chloride per 100 g. of powder for 15 mins., and pressed to eliminate water. The powder cake is broken up, dried in a current of air, ground, and used for the modified method of analysis (J., 1920, 123 A).—D. W.

PATENTS.

Leather; Process for rendering — resistant to chlorine. P. Askenasy. G.P. 299,075, 19.11.16.

THE leather is treated with such chlorinated oils, waxes, etc., as contain less than 30% Cl, or a mixture of such products. These are more suitable than compounds containing more chlorine because they are less resinous in character.—D. W.

Chrome leather waste; Process for detanning — E. Immendoerfer. G.P. 305,598, 8.8.17.

THE waste is treated with aldehydes or quinones, and then submitted to successive strong solutions of alkalis and acids. The aldehyde or quinone renders the felt more resistant to the action of the acids or alkalis.—D. W.

Deliming hides; Process for — R. Haberer und Co. G.P. 317,804, 28.9.18.

WASTE liquors obtained in the removal of the bitter principle of lupins, especially by methods in which no caustic alkali or lime is used, are very suitable for deliming hides, as they do not hydrolyse or dissolve the hide substance even when highly concentrated, and the ammonium salts they contain react favourably on the pelt. The waste liquors may be neutralised with alkalis or alkaline salts before use.—D. W.

Waterproof vegetable-tanned leather; Process for manufacturing — W. Rechberg. G.P. 317,965, 22.2.18.

THE tanned leather whilst still wet is treated with a solution of asphalt, petroleum pitch, rosin, or the like in a volatile organic solvent, dried, and then given a fresh treatment with the impregnating medium.—D. W.

Adhesives; Process of preparing flexible — unaffected by hot water. G. Hoffmann. G.P. 316,604, 17.7.17.

HOT solutions of known adhesives in anhydrous organic solvents are mixed with anhydrous acids and softening agents, and the mixtures cooled and

treated with a small amount of a hardening agent. For example, a solution of cellulose in acetone and amyl acetate is treated with oil and acetic acid, and formalin added to the mixture; or, a solution of anhydrous glue or gelatin in acetic acid is treated with glycerin, and subsequently, when cold, with formalin.—C. A. M.

XVI.—SOILS; FERTILISERS.

Soils; Acidity and acidimetry of —. *The Hopkins and Pettit method for determining soil acidity.* H. G. Knight. J. Ind. Eng. Chem., 1920, 12, 340—344.

In the Hopkins and Pettit method the soil is extracted with potassium nitrate solution and the extract titrated, using phenolphthalein as indicator. The author finds that identical results are obtained when the soil is extracted with *N*/1 potassium nitrate, potassium chloride, sodium nitrate, sodium chloride, or calcium chloride solutions. The acidity of the salt extract of an acid soil is independent of the temperature from 25° to 90° C., but the acidity of the first portions of the extract increases with increase in concentration of the salt solution. The difference in absorption of calcium and potassium from solutions of their bases may be accounted for by precipitation effects. There is a marked basic exchange when a neutral salt solution is added to an acid soil, and alumina goes into solution; this, however, does not account for the total acidity of the solution. Exchange of acid radicles does not take place to any appreciable extent. When an acid soil is extracted with potassium acetate solution, acetic acid may be distilled from the extract.—W. P. S.

Kjeldahl method for crop and soil analysis; Boric acid modification of —. F. M. Scales and A. P. Harrison. J. Ind. Eng. Chem., 1920, 12, 350—352.

The use of 4% boric acid solution (Winkler, J., 1913, 485) for absorbing the ammonia distilled in the Kjeldahl method is trustworthy. Bromophenol-blue is a suitable indicator for the subsequent titration.—W. P. S.

Manganese in plant ashes. Wester. See XXIII.

PATENTS.

Fertilisers; Manufacture of compound —. E. Reinau. G.P. 300,697, 25.3.16. Addn. to 299,001 (p. 449 A).

PHOSPHORITE and similar minerals are decomposed by the mother liquors obtained by the action of nitric acid on mixed sulphates (G.P. 299,006; p. 449 A), yielding fertilisers containing an alkali, ammonia, nitric acid, and phosphoric acid in any desired proportions.—W. J. W.

Saccharifying cellulosic materials. G.P. 316,696. See XVIII.

XVII.—SUGARS; STARCHES; GUMS.

Sugars; Determination of — *by inversion.* Hildt. Ann. Chim. Analyt., 1920, 2, 103—106.

The inversion of sugars by enzymes and by organic and inorganic catalysts is discussed; benzenesulphonic acid is particularly useful for the inversion of sugars in the analysis of condensed milk (cf. J., 1919, 51 A), since its prolonged action does not affect the reducing powers of the sugars produced.—W. P. S.

PATENTS.

Separating solids from liquids [e.g., starch from gluten]; Process and apparatus for —. J. Y. Conte. E.P. 128,944, 25.6.19. Conv., 27.6.18.

The precipitated starch is washed off the inclined troughs on which it accumulates by a powerful jet of water without the use of scrapers, and the water used for this is circulated round and round by a pump, so that the milky liquid may be obtained in a convenient state of concentration.—W. H. C.

Saccharifying cellulosic materials. G.P. 316,696. See XVIII.

Syrup. G.P. 317,165, 317,530, and 318,795. See XIXa.

XVIII.—FERMENTATION INDUSTRIES.

Malling; Changes produced in starch and nitrogenous substances during —. F. Tombeur. Bull. Assoc. Anc. Elèves de Louvain, 1920, 20, 72—85.

A HIGH-GRADE Moravian barley was steeped for 72 hrs. with change of steep water every 12 hrs., malted for 6 days, allowed to "felt" for 1 day, and cured for 48 hrs., first at 45°—50° C. and then at 105°—110° C. The change in the 1000-corn weight (dry) showed a malting loss of 14.36%. The loss of starch during steeping and germinating (due to formation of sugars) was high; the loss during curing (due to formation of decomposition products as well as sugars) was comparatively small. The maximum yield of extract (77.4%) was attained after 12 hours on the kilo, diminishing subsequently (to 75.3%), and it is concluded that the amount of diastase formed during germination is directly proportional to the duration of the process. The total nitrogen-content decreased very slightly during malting, but the soluble nitrogen increased greatly to the first stage of curing and decreased thereafter, thus showing that the amount of nitrogenous matter in wort may be lessened by suitably regulating the kiln temperature. Of the total nitrogen the proportion of soluble nitrogen was 19.17% in the barley and 34.18% in the finished malt. The proportion of coagulable soluble nitrogen decreased to less than one-half between the steeping stage and the end of curing, whilst the proportion of non-coagulable nitrogen increased fourfold.—L. E.

Acidity of worts, beers, and other similar liquids; New methods for determining the —. H. W. Windisch and W. Dietrich. Woch. Bran., 1919, 36, 379—381, 387—390. (See J., 1920, 128 A, 293 A.)

THE presence of both primary and secondary phosphate in a solution ensures the latter against either excessive acidity or excessive alkalinity, i.e., it serves as a "buffer" system. Thus in a solution of primary and secondary potassium phosphates the H-ion concentration changes from 0.64×10^{-3} to 0.61×10^{-8} as the ratio of primary to secondary salt is changed from 32:1 to 1:32. The proportions of free acid and of primary and secondary phosphate in mixtures may be determined by the method previously described (*loc. cit.*) by using capillary active organic bases as indicator. Of the bases tried only eucupine (0.1% solution of the dihydrochloride) proved sufficiently sensitive; it is less convenient than the acid indicator since about 5 mins. must be allowed after each addition of alkali in order that the liquid may acquire its "permanent" viscosity; and further, the viscostagonometer outlet is liable to be rendered greasy by the free base and needs to be cleaned frequently with chromic acid. On adding successive portions of standard alkali to a mixture of free acid and primary phosphate in presence of eucupine

dihydrochloride, the surface tension remains practically constant until the free acid is completely neutralised, at which point it falls rapidly to a value which again alters very slowly until tertiary phosphate begins to be formed, when there is a further rapid fall. The liquid must not contain more than about 1 c.c. of $N/10$ primary salt, otherwise the secondary salt formed during titration will lower the surface tension so much as to render the observation of the formation of tertiary salt rather difficult.—L. E.

Hop bitter acids; Analysis of — and their changes during hop boiling. H. Lüers and A. Baumann. Z. ges. Brauw., 1920, 43, 65–67, 73–76, 81–84, 89–82, 97–101.

THE hop bitter acids lower the surface tension very considerably. Surface tension measurements with the dropping pipette (stalagmometer or viscositagonometer) of Traube were made on hop extracts prepared by boiling 1 g. of the hops with 1 l. of 2% Rochelle salt for 1 hr. under a reflux condenser. There is a certain parallelism between the results and those obtained by the determination of the bitter substances of hops by Adler's method (J., 1912, 1003), and the surface tension method is advantageous in following the degree of extraction during hop boiling, since it shows differences during the later stages of extraction which cannot be detected by Adler's method. Surface tension measurements, ultra-microscopic examination, and ultra-filtration experiments show that the α -bitter acid (humulone) is, in aqueous solution, intermediate between a suspensoid and an emulsoid, its degree of dispersion being affected by the presence of electrolytes or of other colloids. Humulone lowers the surface tension of water to a greater degree in presence of acid than in presence of alkali. Since it lowers the surface tension of water strongly, humulone tends to accumulate at the surface of separation between the water and air or other phase. This property is also possessed by proteins and dextrans, and in conjunction with the high internal friction of the dextrans and products of protein-hydrolysis imparts head-retention to beer. During hop boiling the humulone undergoes a chemical change (resinification) corresponding with which there is a rise of surface tension; the change, which is accompanied by increase of bitter flavour, increases with duration of boiling and with the acidity of the solution. This change is less pronounced when the humulone is boiled with wort than when boiled in aqueous solution, whence it seems that the wort protein protects the humulone to some extent. The β -hop bitter acid (lupulone) and the γ -resin also possess the property of lowering surface tension though to a much less degree than humulone.—L. E.

Beer wort; Conditions requisite for head-formation by —. W. Windisch and V. Bermann. Woch. Brau., 1920, 37, 109–111, 121–125, 129–132, 137–139, 145–147, 153–155.

By using membrane filters (cf. Zsigmondy and Bachmann, J., 1918, 453A) differing in fineness of porosity, it is possible to obtain an approximate measure of the degree of fineness which colloidal particles must possess in order to affect the head-formation of wort, etc. If wort which gives a persistent head is filtered through a membrane filter of sufficient fineness, the ultra-filtrate thus obtained gives, on shaking, an abundant but non-persistent head; in this respect the ultra-filtrate resembles a solution of albumoses. If the residue on the filter is washed free from sugars and salts, dried, and treated with water, part of it dissolves (reversible colloids) and part is insoluble (irreversible colloids). If the ultra-filtrate is treated

with the soluble part of the filter residue it regains the power of head-retention. The soluble part of the filter residue consists in part of non-coagulable protein, but chiefly of non-protein substances, and it appears that whilst the non-coagulable proteins are able to produce head, it is the non-protein substances which render the head persistent. These non-protein substances are of a carbohydrate nature. The proteins which produce head have a high capillary activity, whilst the non-proteins which render the head persistent have a low capillary activity. The authors have employed the "iron value" (analogous to the "gold value" of Zsigmondy, J., 1902, 192) for characterising the colloids which are responsible for head-retention. This "iron value" is measured by the concentration of sodium chloride requisite just to produce turbidity or flocculation of a ferric hydroxide sol in the presence of the colloid. Of the non-proteins which cause head-retention, barley gum appears to be the most active, and there appears to be an optimum proportion between the protein head-producer and the non-protein which causes head-retention. By the addition of a capillary active substance (e.g., amyl or octyl alcohol) which lowers the surface tension of wort, the latter loses the power of head-formation when its surface tension is sufficiently reduced, viz., to 38 or 40 dynes per cm.—L. E.

Yeast; Influence of sudden cooling, during or after fermentation, on —, especially in respect of subsidence. H. Will. Z. ges. Brauw., 1920, 43, 49–51, 57–58.

IN a brewery where, inadvertently, the yeast was kept below 0° C. for 34–36 hours, great difficulty was subsequently experienced with regard to subsidence, the beer remaining quite turbid with suspended yeast; the flavour of the beer also was very prejudicially affected. Experiments showed that the subsidence is not affected by low temperature (0° to –19° C.) either during or immediately after fermentation or 3 or 6 weeks after fermentation, and the difficulty encountered at the brewery must have been due to some other cause.—L. E.

Beer; Use of Bac. Delbrücki in the preparation of stable pale —. H. Lüers. Z. ges. Brauw., 1920, 43, 51–53, 58–61.

ACIDIFICATION of the mash and wort by means of *Bac. Delbrücki* (Windisch, J., 1913, 986), in the preparation of pale beer, improves the colour and flavour of the beer, especially where brewing water of high carbonate-content is used. To restrict the protein-content of the beer as far as consistent with head-retention and palate-fulness, it is advisable to acidify the wort rather than the mash. A small proportion of wort of about 14° Bllg. is treated at 50° C. with a pure culture of the organism, an acidity equivalent to 1.5–1.6% of lactic acid being obtained in 12 hrs. Sufficient of this acidified wort is added to the main mash to neutralise the carbonate of the water, and water softened by boiling is used for sparging. The bulk of the acidified wort is added gradually to the main wort during the whole drainage process, the amount so added (8 hl. per brew of 180 hl. of 12° wort) being such that the wort, after coagulation of the protein, has an acidity, $p_H=5.2$, at which maximum protein coagulation is obtained. The finished beer has a slightly higher acidity ($p_H=4.0-4.2$) than beer prepared in the ordinary way ($p_H=4.3-4.4$) and is characterised by brilliance, good head-retention, and flavour. It also possesses very high mechanical as well as biological stability, being unaffected by strong mechanical agitation. The loss of extract involved in acidification renders the process too costly for ordinary use.—L. E.

Saccharin and dulcin in beer; Detection of —.

A. Baumann. *Z. ges. Brauw.*, 1920, 43, 137—139.

For the detection of saccharin and dulcin in beer the method proposed by Tortelli and Piazza (J., 1910, 1327) gives satisfactory results, but the saccharin isolated is liable to be contaminated with impurities which prevent its crystallisation; this difficulty is avoided as follows:—The residue of saccharin (from 1 l. of beer) left on distilling the ether-petroleum spirit solution (*loc. cit.*) is dissolved in alcohol and treated with a few drops of potassium hydroxide solution and then with a few c.c. of sodium chloride solution and two or three times this quantity of water, shaken with petroleum spirit, and the solution is distilled until free from alcohol, cooled, acidified with 3—4 c.c. of 10% sulphuric acid and again extracted 3 or 4 times with the mixture of ether and petroleum spirit. This extract is washed with a little water to remove acid and filtered; on evaporating the filtrate *in vacuo* at the ordinary temperature the saccharin is obtained crystalline and almost white. The authors have isolated saccharin and dulcin from beer to which 90 mg. saccharin and 10 mg. dulcin or 10 mg. of saccharin and 90 mg. dulcin had been added per litre.—L. E.

Wines; Treatment of the blue "casse" of —. A. Piedallu, P. Malvezin, and L. Grandchamp. *Comptes rend.*, 1920, 170, 1129—1131.

OXYGEN in an extremely finely divided condition, obtained, for instance, by forcing it through a porcelain filter candle under pressure, when passed through wine damaged by blue "casse" is capable of rapidly oxidising the ferrous salts present in such wine to ferric salts. By this means the iron may be eliminated, being precipitated in its ferric state by the tannins present, and such wine after clarification is not liable to further "casse."

—W. G.

Alcohol and water; Vapour composition of mixtures of —. W. K. Lewis. *J. Ind. Eng. Chem.*, 1920, 12, 496—499.

THE results obtained by Wrewsky (*Z. physik. Chem.*, 1912, 81, 1) have been recalculated and extended by extrapolation, and the data expressed graphically in diagrams so as to be applicable to the conditions of industrial distillation. (*Cf. J.C.S.*, July.)—C. A. M.

Fermentation; Sensitiveness of yeast — to the p_H value. H. von Euler and S. Heintze. *Ark. Kemi*, 1917, 7, No. 21, 1—21.

YEAST fermentation of sucrose and dextrose occurs with a maximum velocity in solution of acidity represented by $p_H=5$. In the presence of yeast water or asparagin the maximum velocity is at the same point, but is very much greater than in solutions which contain no nutritive nitrogen compounds. Oxalic acid in solutions up to 0.025N is not a yeast poison nor is chromic acid in dilute solutions. (*Cf. J.C.S.*, July.)—J. F. S.

Saccharase [invertase]; Influence of temperature and acidity on the formation of —. H. von Euler and O. Svanberg. *Ark. Kemi*, 1917, 7, No. 23, 1—32.

THE optimum formation of invertase from yeast occurs at 26°—30° C.; at 35° there is no further formation. At acidity $p_H=2$ the formation of invertase is disturbed, but at $p_H=6-7$ there is a strong formation which amounts to 90% of that under the optimum conditions of acidity.—J. F. S.

Saccharase [invertase]; Temperature sensitiveness of —. H. von Euler and I. Laurin. *Ark. Kemi*, 1917, 7, No. 24, 1—30.

THE activity of invertase is decreased by one half

on heating to 59° C.; at this temperature it has an inactivation constant, $k=5 \times 10^{-3}$. The minimum temperature sensitiveness of invertase lies at an acidity $p_H=4-5$, and this point is the position of optimum enzyme activity. The temperature sensitiveness of separated invertase is a little less than that of the non-separated enzyme due to a protecting action of the cell. Invertase from bottom yeast has a smaller temperature sensitiveness (about 2°) than that from top yeast. (*Cf. J.C.S.*, July.)—J. F. S.

Catalase from Phyllostachys mitis, Riv. Chemical kinetics of catalase I. E. Yamasaki. *Sci. Rep. Tôhoku Imp. Univ.*, 1920, 9, 13—58.

A CATALASE prepared from an edible sprout of a species of bamboo, *Phyllostachys mitis*, Riv., decomposes hydrogen peroxide with a velocity which steadily decreases during the reaction. The enzyme is prepared by crushing the plant with water saturated with toluene and after 24 hrs. pressing and treating the filtrate with absolute alcohol. (*Cf. J.C.S.*, i., 453.)—J. F. S.

Methyl alcohol. Schryver and Wood. See XX.

PATENTS.

Soft drinks [non-intoxicating beverages]; Process of producing —. A. L. Straus. U.S.P. 1,337,027, 13.4.20. Appl., 14.11.19.

BEER that has undergone alcoholic fermentation is boiled to remove alcohol. The residue is mixed with water and treated with an agent to produce a bitter flavour and with a foam-producing material, or it is treated with sufficient citric acid to produce a pungent taste. In either case it is further treated with a carbohydrate sweetening material, salt, and hops. The product is fermented to an alcohol content of less than 0.5%, fined, carbonated, and filtered; it may be carbonated and filtered a second time, if desirable, and is finally carbonated again.—L. E.

Beverages; Process for making non-intoxicating —. W. Wilhelmy, sen. U.S.P. 1,338,803. 4.5.20. Appl., 17.5.19.

A MIXTURE of about 500 lb. of ground, pale malt and about 300 lb. of ground caramel malt is mashed with about 18 brls. of water at 77° F. (25° C.) and boiled for a short time. After settling the wort is drawn off, mixed with about 85 brls. of boiling water, boiled, treated with pre-determined quantities of hops during boiling and sufficient water to make 100 brls. after boiling. The liquor is then drained (from the spent hops etc.), cooled to about 68° F. (20° C.), pitched with yeast (4 lb. per brl.), and allowed to ferment for about 36 hrs. The brew is allowed to cool and stand for a specified period, treated with a body-giving substance, and then carbonated.—L. E.

Yeast; Manufacture of — with little or no production of alcohol. Verein der Spiritus-Fabrikanten in Deutschland. G.P. 304,213, 24.4.15. Addition to 303,222 (J., 1920, 315 A).

THE nutrient solution in which the yeast is pitched contains a mineral salt from which the acid is liberated by the yeast, and this acid combines with the alkali (e.g. ammonia) or alkaline salts present in the inflowing nutrient solution.—J. H. L.

Cellulosic materials; Saccharification of — with simultaneous recovery of citrate-soluble phosphate. Chem. Fabr. Rhénania A.-G., F. L. Schmidt and G. A. Voerkelius. G. P. 316,696, 25.12.17. Addition to 305,120 (J., 1919, 896 A). (See also Ger. Pat. 304,400; J., 1920, 309 A.)

THE hydrolysis of the cellulose ester and the solution of the phosphate are effected by a short boiling in concentrated solution, e.g., with 2—4 parts of

water for one part of wood originally taken. A 10% solution of sugar is thus obtained. Preferably only so much phosphate is used as will yield the amount of free phosphoric acid necessary to saccharify the wood dextrins.—J. H. L.

XIXA.—FOODS.

Milk analysis. G. Ambühl and H. Weiss. Mitt. Lebensmittellunters. Hyg., 1919, 10, 53—75. Chem. Zentr., 1919, 90, IV., 191—192.

Preparation of serum for refractometer measurements.—The reagent is prepared by dissolving 125 g. of mercuric chloride in about 75 c.c. of warm 36% hydrochloric acid and then diluting the solution to 100 c.c. with the same strength acid; 0.3 c.c. of the reagent mixed with 30 c.c. of water should give a refractometer reading of exactly 20°. Milk serum is obtained by mixing 30 c.c. of milk with 0.3 c.c. of the reagent, and filtering the mixture. The quantity of added water in a milk calculated from the refraction of the serum agrees with the amount actually present if this does not exceed 35%; with from 40 to 60% of added water the results are 2.5—4.5% too low. The refractions of serums prepared with mercury chloride are usually 3.0°—3.4° higher than those of serums prepared with calcium chloride, but the difference is less in the case of sour milks.

Detection of nitrates in milk.—The method described by Tillmans and Splittgerber (J., 1912, 249) is trustworthy, but not that of Tillmans (*ibid.*, 1911, 44). The test may be applied to the mercury chloride serum obtained as described above; nitrates are decomposed gradually in milk and about one-half of the quantity may disappear within one month. The reagents themselves and the filter paper used must be tested for nitrates.

—W. P. S.

Butter and margarine; Detection of coal-tar dyes in —. G. Van B. Gilmour. Analyst, 1920, 45, 173.

ABOUT 1 c.c. of the clear filtered fat is heated in a test-tube at 185° C. and shaken occasionally; in the absence of coal-tar dyes the fat will become colourless within 10 mins., but in their presence the fat remains coloured. The fat must be separated previously at a moderately low temperature since butter fat separated (from curd, etc.) at above 100° C. does not become colourless when heated at 185° C.—W. P. S.

Cellulose as cattle fodder; Use of —. F. Scurti and G. Morbelli. Staz. Sper. Agrar. Ital., 1919, 52, 233—265. Chem. Zentr., 1919, 90, IV., 1112—1113.

MAIZE cobs contained 38.6% of crude cellulose, 1.75% of crude proteins, and 46.3% of substances soluble in dilute hydrochloric acid. By digesting the cellulose for 2 hrs. with 10 pts. of 0.125—2.5% sulphuric acid 45.2—53.3% of the cellulose was dissolved, of which 32.96—47.5 consisted of reducing sugars calculated as dextrose. The residue consisted mainly (up to 74%) of cellulose. The best results were obtained when the quantity of acid was 4% of that of the material. Reduction of the amount of liquid or of the acidity, or increase of the pressure to 5 or 10 atm. caused reversal of the hydrolysis. The yield of sugars was also reduced by reducing the period of hydrolysis to 1 hr. or prolonging it beyond 2 hrs. The soluble carbohydrates contained dextrose, arabinose, and xylose.

—C. A. M.

Rice chaff. Scurti and Zay. See II B.

Mineral waters. Baughman and Skinner. See VII.

PATENTS.

Preserving organic materials [e.g., foodstuffs]. R. Pape. E.P. 142,169, 24.1.19.

ORGANIC material, e.g., a foodstuff such as milk, is heated at 70° C. or at 98°—100° C., and at the same time is subjected to a gaseous pressure of 2 atm. or more during the whole period of heating. During the heating the material is kept in a state of agitation to increase the amount of gas dissolved and to prevent superheating. In order to maintain the original taste, smell, and colour of the material the heating may be conducted at 40°—60° C. and the gases introduced heated to 250°—350° C., or, conversely, the material may be at a high temperature—say 120° C.—and the gases introduced at a low temperature—say 20° C.—J. H. J.

Eggs; Method of preserving —. H. J. N. H. Kessener and N. L. Söhngen. G.P. 312,505, 17.3.17.

Eggs are coated with a solution of cellulose acetate in ethyl acetate; the coating does not affect the taste or vitality of the eggs.—W. P. S.

Beverage extract; Solid soluble — and process of manufacturing same. J. K. Lippen, Assr. to Postum Cereal Co. U.S.P. 1,338,231, 27.4.20. Appl., 1.4.18.

SORGHUM grains are flaked and the flour is separated, dextrinised, and re-mixed with the flakes. Dextrinised wheat and barley flours, rye middlings, and molasses are added, and the mixture is roasted at a caramelising temperature. The product is extracted with water, and the solution evaporated to dryness.—J. H. J.

Fruit-juices; Process of treating —. F. L. Dunlap and R. A. Kuever. U.S. Pat. 1,338,684, 4.5.20. Appl., 24.9.18.

A PROTEOLYTIC enzyme active in slightly acid media is added to citrus fruit juice, which is then heated sufficiently long to enable the enzyme to digest the proteins of the juice.—J. H. J.

Drying fruits, vegetables, and other substances; Method of and apparatus for —. G. H. Benjamin. U.S.P. 1,339,092, 4.5.20. Appl., 16.4.17.

SUBSTANCES to be partially dried are subjected simultaneously to the action of radiant heat, electric light rays of controlled intensity, and a moving atmosphere of controlled temperature and humidity.

—J. H. J.

Dried potatoes; Process for preparing —. J. Carpozow. G.P. 318,980, 7.5.15.

POTATOES are washed, peeled, finely-divided, covered with a layer of vegetable mucilage, and dried at a temperature which will not affect the starch. The coating prevents darkening of the potatoes.—C. A. M.

Syrup; Preparation of non-crystallising edible —. R. Schomann. G.P. (A) 317,165, 27.6.18, and (B) 317,530, 3.8.18.

(A) A NON-CRYSTALLISING syrup is made by concentrating below 100° C. starch milk to a starch content of 5—10%, and mixing with beet molasses, etc., in such proportion that the mixture contains about 66% of soluble solid matter. Both the starch and the pectin of the potatoes impart consistency to the syrup. (B) Potato shavings or potato meal are added to the molasses and the starch is converted into starch paste by heating at 65°—70° C.

—G. F. M.

Syrups; Production of non-crystalline table —. R. Schomann. G.P. 318,795, 4.7.18. Addition to 317,165 (preceding).

POTATOES are steamed and so strongly pressed as to

yield a juice containing 5–10% of starch. The juice, after evaporation at a temperature below 100° C. if necessary, is treated with such a quantity of beet sugar, or concentrated juice or molasses, that the mixture contains not more than 66% of soluble dry substance.—J. H. L.

Leguminous vegetables; Process for rendering — more easily cooked. H. Lüthjo. G.P. 318,568, 18.11.16.

PEAS etc. are treated at the ordinary temperature or far below the boiling temperature with a solution of caustic alkali or ammonia, or with gaseous ammonia, and the water content then reduced to that of ordinary commercial products.—C. A. M.

Cacao, chocolate, or the like; Apparatus for grinding or mixing —. C. Postranceky. E.P. 13,512, 3.6.14.

Cocoa and chocolate; Process and apparatus for manufacture of —. F. E. Whitham. E.P. 141,953, 31.5.19.

Preserving animal and vegetable substances; Method and apparatus for —. K. G. Falk and E. M. Frankel. E.P. 123,073, 30.1.19. Conv., 30.1.18.

SEE U.S.P. 1,309,357 of 1919; J., 1919, 651A.

Milk product; Condensed — S. M. Dick. E.P. 141,763, 8.3.18.

SEE U.S.P. 1,258,996—7 of 1918; J., 1918, 320A.

XIXB.—WATER PURIFICATION; SANITATION.

Sewage purified by the activated sludge process; Action of the bacteria of the flora of — on carbohydrates. P. Courmont and A. Rochaix. Comptes rend., 1920, 170, 1131–1135.

The bacteria of the flora of sewage purified by the activated sludge process exert a marked fermenting action on carbohydrates, the activity varying with the species.—W. G.

PATENT.

Scale and the like on citrus trees; Process of destroying —. H. Blumenberg. U.S.P. 1,237,538, 20.4.20. Appl., 26.12.16.

SCALE-INFESTED trees are treated with a compound of hydrogen and an element of the phosphorus group, except nitrogen and boron.—J. H. J.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Alkaloids; Rapp's method for determination of —. A. Heiduschka and L. Wolf. Süddtsch. Apoth.-Zeit., 1920, 60, 142–143. Chem. Zentr., 1920, 91, 11., 581–582.

Low results by Rapp's method for estimation of alkaloids (J., 1919, 336 A) caused by partial absorption of the chloroform by the plaster of Paris paste, may be prevented by mixing sand or powdered glass with the paste. For the assay of cinchona bark, 3 g. is heated with 15 c.c. N/2 hydrochloric acid on the water bath for 10 mins., cooled, agitated for 5 mins. with 60 c.c. of chloroform and 3 c.c. of 20% sodium hydroxide, and then for 1 min. with 30–35 g. of plaster of Paris. The chloroform, after separation, is filtered and 50 c.c. is shaken for 1 min. with 12.5 c.c. of N/10 hydrochloric acid,

diluted with 12.5 c.c. of water, and 20 c.c. of the mixture finally titrated with N/10 potassium hydroxide; the percentage of quinine is given by $(10 - \text{c.c. } N/10 \text{ KOH})300 \div 2$. In the case of *Semen strychni* it is sometimes difficult to obtain a clear chloroform solution, even after repeated extractions with benzene. 100 g. of the alkaloid solution in ether-chloroform is evaporated to small bulk, treated with 25 c.c. of chloroform, and shaken with 20 c.c. of 2% sulphuric acid. After clarification, the solution is passed through a filter half filled with moistened plaster of Paris, 14 c.c. of the clear filtrate being mixed with 3 c.c. of 15% sodium hydroxide and shaken for 3 mins. with 70 c.c. of chloroform and 25–30 g. of plaster of Paris. 60 c.c. of this solution, after clarification, is filtered, shaken with 12 c.c. of N/10 sulphuric acid, diluted with 12 c.c. of water, and 20 c.c. of this mixture titrated with N/10 sodium hydroxide. The percentage of alkaloid is equal to $(10 - \text{c.c. } N/10 \text{ NaOH})3.61 \div 5$.—W. J. W.

Quinine; Determination of —. C. Bamberger. Pharm. Zentr., 1920, 61, 257–259, 267–270.

THE following method yields results which agree with those obtained by the Swiss Pharmacopœia method which is known to be trustworthy (the German Pharmacopœia method is unreliable):—25 g. of cinchona bark is warmed on a water-bath for 10 mins. with 2 c.c. of hydrochloric acid and 20 c.c. of water, then cooled and shaken with a mixture of 25 g. of chloroform and 50 g. of ether. 5 g. of sodium hydroxide is then added, the mixture shaken thoroughly for 3 mins., and 50 g. of crystallised calcium sulphate is added; when this has settled, 60 g. of the clear chloroform-ether solution is transferred to a separating funnel and shaken out twice with 5 c.c. of N/10 hydrochloric acid and twice with 10 c.c. of water. The united extracts are titrated with N/10 potassium hydroxide solution, using methyl-red as indicator. (Cf. Frerichs and Mannheim, J., 1915, 1070; Rapp and Dietrich, J., 1918, 336A.)—W. P. S.

Solvarsan; Composition of —. R. G. Fargher and F. L. Pyman. Chem. Soc. Trans., 1920, 117, 370–377.

SALVARSAN precipitated from methyl alcoholic solution by means of ether contains no combined methyl alcohol as suggested by Kober (J. Amer. Chem. Soc., 1919, 41, 442). When precipitated by means of acetone, however, it contains one molecular proportion of that substance. Commercial solvarsan contains a considerable proportion of sulphur, 2–3% in extreme cases, which is not disclosed in the reputed formula. The authors consider that, at least in part, this occurs in the form of a sulphamma-group, $-\text{NH}_2\text{SO}_2\text{H}$, whilst some may be directly attached to arsenic, or be present in physical association with the solvarsan. Pure solvarsan, as distinct from the commercial product, was obtained by the reduction of 3-nitro-4-hydroxyphenylarsinic acid in alkaline solution with just sufficient hydrosulphite to reduce the nitro-group. The resulting amine was purified by the method of Ehrlich and Berthelm (J., 1912, 407) and was further reduced to the arsenobenzene by means of hypophosphorous acid in presence of potassium iodide as a catalyst, the precipitated base being then converted into its dihydrochloride.—G. F. M.

Arsanilic acid; Preparation of primary —. H. C. Cheetham and J. H. Schmidt. J. Amer. Chem. Soc., 1920, 42, 828–829.

By heating a mixture of dry arsenic acid (4 g.-mol.) and aniline (1 g.-mol.) for 12 hrs. at 150°–160° C. a good yield of arsanilic acid (26% of the pure recrystallised substance) can be obtained and 90% of the excess aniline can be easily recovered.—J. K.

Arsenic; Organic compounds of —. A. McKenzie and J. K. Wood. Chem. Soc. Trans., 1920, 117, 406–415.

ETHOXYDICHOROARSINE, b.p. 145°–146° C. at 751 mm., and diethoxychloroarsine, b.p. 64°–65° C. at 20 mm., were obtained by the action of one and two mols. respectively of sodium ethoxide on arsenious chloride. Ethyldichloroarsine was obtained from arsenious oxide through ethyldiiodoarsine and ethylarsenious oxide. For the preparation of diphenylethanoarsine, diphenylchloroarsine was treated with sodium hydroxide solution, whereby an almost theoretical yield of diphenylarsenious oxide was obtained. This was heated at 120°–160° C., and dry hydrogen cyanide was passed into the molten material for 5 hrs. The yield of crude product is almost theoretical. The pure substance has m.p. 31°–32° C.—G. F. M.

Benzoic acid and its derivatives; Volatility of — in steam. N. V. Sidgwick. Chem. Soc. Trans., 1920, 117, 396–406.

THE volatility in steam of benzoic acid and some of its derivatives was determined by passing steam through a mixture of the acid and water contained in two 120 c.c. flasks heated in a glycerin bath and arranged in series with one another and with a Kjeldahl trap leading to the condenser. The steam connections, the upper parts of the flasks, and the trap were all lagged to prevent condensation. The following are some of the results:—

	Percentage in distillate.	Vapour pressure in mm. × 100.	Relative vap. pressure.
Benzoic acid ..	1.006	112.9	1.0
Phenylacetic acid ..	0.294	29.55	0.262
Toluic acid—ortho ..	0.964	97.0	4.49
—meta ..	0.604	60.7	2.81
—para ..	0.215	21.6	1.0
Hydroxybenzoic acid—ortho ..	0.400	39.7	1320.0
—meta ..	0.0015	0.149	5.0
—para ..	0.0003	0.030	1.0

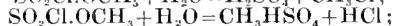
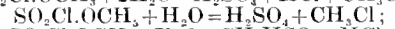
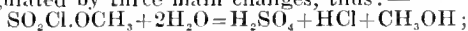
The relative vapour pressure above is referred to the *para*-acid as unity, except that phenylacetic acid is referred to benzoic acid.—G. F. M.

Chlorobenzenes; Analysis of industrial —. F. Bourion and C. Courtois. Comptes rend., 1920, 170, 1115–1117.

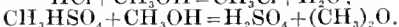
THE method previously given (J., 1920, 425 A) for the analysis of industrial chlorobenzenes is slightly modified to allow of a more accurate determination of the percentage of polychlorobenzenes. A sample of the chlorobenzene is distilled at 160° C. and the density at 40° C. of the residue which does not pass over is determined. By means of this value and formulæ given it is possible to calculate the percentage of polychlorobenzenes present.—W. G.

Methyl chlorosulphonate; Action of water on —. J. Guyot and L. J. Simon. Comptes rend., 1920, 170, 326–328.

THE action of water on methyl chlorosulphonate is regulated by three main changes, thus:—



there being under some conditions two subsidiary changes:—



The final equilibrium depends on the relative amounts of water and chlorosulphonate present. The primary change is the one which produces methyl hydrogen sulphate, but with excess of water this compound is further decomposed, this latter action being, however, reversible. When the amount of water diminishes the concentration of the acids becomes important, the esterification of the methyl alcohol, with the consequent formation of methyl chloride, becoming the most important change.—W. G.

Halogenhydrins; Preparation of —. J. Read and M. M. Williams. Chem. Soc. Trans., 1920, 117, 359–362.

CHLORINE or bromine water may be used as a source of hypochlorous or hypobromous acid for the preparation of halogenhydrins from ethylenic compounds. Ethylene bromohydrin, for example, is obtained by leading ethylene and air saturated with bromine vapour through two perforated bulbs fixed a short distance apart in ice-cooled water which is kept in continual agitation. The operation may be continued until the solution contains over 14% of bromohydrin. A proportion of ethylene dibromide, corresponding to about 27% of the bromine used, is formed as a by-product, but in the case of the chlorohydrin the amount of dichloride formed appears to be substantially less (*cf.* Gomberg, J., 1919, 923 A).—G. F. M.

Ethers; Catalytic preparation by the dry way of certain —. A. Mailhe and F. de Godon. Bull. Soc. Chim., 1920, 27, 328–330. (See J., 1919, 926 A; 1920, 247 A.)

ALLYL alcohol vapour when passed over calcined alum at 185°–190° C. gives a 30% yield of allyl ether. If the allyl alcohol is mixed with methyl alcohol or its higher homologues the main product is still allyl ether, although a certain amount of the mixed ether is obtained. (*Cf.* J.C.S., July.)

—W. G.

Amines; Preparation of — by catalysis. A. Mailhe. Comptes rend., 1920, 170, 1120–1123.

ALDAZINES undergo hydrogenation when passed with hydrogen over reduced nickel at 140°–160° C., giving the corresponding primary and secondary amines, the relative proportions of the two amines obtained varying with the temperature of hydrogenation and the aldzine used. The method has been applied to the preparation of ethyl-, isobutyl-, and isoamyl-amines.—W. G.

Hydrogenation; Catalytic — with protected hydrosols. E. K. Rideal. J. Amer. Chem. Soc., 1920, 42, 749–756.

PROTECTING colloids, such as gum arabic, function as peptising agents in catalytic hydrogenation by colloidal platinum and palladium. The addition of a little colloidal palladium to colloidal platinum greatly enhances the activity of the latter. (*Cf.* J.C.S., ii., 364.)—J. F. S.

Catalytic actions at solid surfaces. III. Hydrogenation of acetaldehyde and the dehydrogenation of ethyl alcohol in the presence of finely divided metals. E. F. Armstrong and T. P. Hilditch. Proc. Roy. Soc., 1920, A, 97, 259–264. (*Cf.* J., 1919, 780 A; 1920, 163 A.)

ACETALDEHYDE is converted into ethyl alcohol, when passed with hydrogen over finely divided copper or nickel at 120°–300° C. With copper the yield of alcohol is 87.6% at 200°, and 33.7% at 300° C., whilst with nickel at 120°–150° C. it is 53.6%. In the presence of copper at 295°–300° C. a yield of 95% aldehyde may be obtained from alcohol. The presence of a little water in the alcohol improves the yield of aldehyde relatively to that of hydrogen. Small quantities (1–2%) of by-products, *n*-butyric aldehyde, crotonic aldehyde, and ethyl acetate, are always obtained. The presence of small quantities of water protects aldehyde from hydrogenation and in some cases entirely prevents the reaction. (*Cf.* J.C.S., July.)—J. F. S.

Methyl alcohol; Determination of —. S. B. Schryver and C. C. Wood. Analyst, 1920, 45, 164–170.

THE method proposed consists essentially in de-

termining the concentration of ammonium persulphate necessary to destroy completely the formaldehyde formed in the initial stages of the oxidation process under specified conditions of experiment. The formaldehyde is detected by treating the solution with 1% phenylhydrazine hydrochloride solution, then adding potassium ferri-cyanide and concentrated hydrochloric acid; a brilliant red coloration is produced and the test will detect as little as 1 part of formaldehyde in 2,000,000. To determine methyl alcohol in aqueous solution, portions of 5 c.c. each of the solution (which may contain from 0.0005 to 1% of the alcohol) are placed in a series of test-tubes, 5 c.c. of ammonium persulphate solution of varying concentration (0.01 to 5%) is added, the mixtures are heated at 100° C. for 10 mins., and 1 c.c. of each mixture is then tested for formaldehyde. A second series of oxidations is then made, using 5 c.c. of the alcohol solution with 5 c.c. of persulphate solution in concentrations varying between the limits of concentration found in the first test at which the oxidation of the formaldehyde was complete and not complete respectively. For each concentration of methyl alcohol there corresponds a definite concentration of persulphate to oxidise all the formaldehyde; these concentrations are given in tabular form. If c is the concentration (%) of the persulphate and x that of the methyl alcohol, and k a constant, then $x = kc$; experiments with pure methyl alcohol show that $k = 0.059$. The method, with slight modification, is applicable to the determination of methyl alcohol in acetone; in this case the amount of methyl alcohol present must be not less than 4%. There is no preferential oxidation of methyl alcohol in a mixture of this with ethyl alcohol, but in such a mixture the methyl alcohol may be determined by oxidising the mixture partially with a relatively small amount of persulphate (the alcohols being in excess) and determining colorimetrically the formaldehyde formed under these conditions. Ten c.c. of the ethyl alcohol containing methyl alcohol is diluted with 50 c.c. of water and 5 c.c. of this solution is heated in a boiling water-bath for 10 mins. with 5 c.c. of 1% ammonium persulphate solution; 1 c.c. of the mixture is then mixed with 1 c.c. of phenylhydrazine hydrochloride solution, heated in a boiling water-bath for 5 mins., cooled, treated with 1 c.c. of 2.5% potassium ferri-cyanide solution and 3 c.c. of concentrated hydrochloric acid, and the coloration compared with standards prepared, under similar conditions, with ethyl alcohol containing known amounts of methyl alcohol.—W. P. S.

Acetates; Systematic detection of —. L. J. Curtman, D. A. Broggi, and V. Fourman. *Chem. News*, 1920, 120, 230—231.

To detect acetates in mixtures the authors recommend the following procedure: The solution is acidified with nitric acid, treated with silver nitrate, and filtered. The filtrate is made just alkaline with caustic soda, a solution of barium and calcium chlorides added, and the precipitate filtered off. The clear liquor is concentrated to 5 c.c. after addition of hydrogen peroxide. After filtering off the deposited crystals the solution is made just neutral to phenolphthalein with dilute hydrochloric acid, 1 c.c. of ferric chloride solution is added, and the colour compared with that of controls prepared with 1 c.c. of ferric chloride solution in 5 c.c. of water and known quantities of sodium acetate. A reddish-yellow colour is perceptible if as little as 2 mg. of acetic acid is present. Acetates may also be recognised by the odour of the vapour evolved on boiling 1 c.c. of the test solution with 2 c.c. of sulphuric acid (1:1). The test is sensitive to 2 mg., but nitrates seriously interfere. (*Cf.* J.C.S., ii., 393.)—A. R. P.

Juniperus oxycedrus oil; Chemical characters of —. R. Huero. *J. Pharm. Chim.*, 1920, 21, 347—352.

Juniperus oxycedrus oil has acid value, 0.46—I37; saponif. value, 2.9—5.0; iodine value (Hübl), 179; free alcohols (extracted by phthalic anhydride), 12—14; acetyl value, 38.7—30.5. Whilst the total amount of bromine absorbed by the oil varies with the excess of bromine added, the bromine combining as an addition product with the formation of hydrobromic acid is a more constant quantity and ranges from 34 to 38 g. per 100 g. of oil.—W. P. S.

Xanthorrhoea resin. Rennie and others. See XIII.

Halogen in organic substances. Van Winkle and Smith. See XXIII.

PATENTS.

Acetic anhydride; Manufacture of —. H. Dreyfus. U.S.P. 1,338,979, 4.5.20. Appl., 4.2.19.

In the process where gaseous sulphuric anhydride reacts at a low temperature with a dry acetate the sulphuric anhydride is mixed with an indifferent gaseous diluent. (*Cf.* E.P. 17,920 of 1915; J., 1917, 162.)—A. E. D.

Alkaline-earth salts of organic acids; Manufacture of water-soluble —. A. Herzfeld and G. Lénárt. G.P. 303,282, 19.2.16.

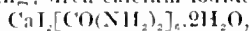
A MIXTURE of organic and inorganic acids obtained by the oxidation of a carbohydrate is treated with sufficient alkali hydroxide to neutralise the inorganic acid, and the organic acid is then neutralised by the addition of an alkaline-earth oxide or hydroxide. The alkaline-earth salt of the organic acid is then recovered from the solution by crystallisation. (*Cf.* J., 1920, 205A.)—L. A. C.

Tannin-albumin compound; Process for preparing a — from blood. Knoll und Co. G.P. (a) 305,693, 4.9.17, and (b) 317,675, 7.3.18.

(a) BLOOD is precipitated with a quantity of tannin equal to the total content of albuminous matter, including the hæmoglobin. The precipitate is drained, dried, and, if desired, subjected to a temperature above 100° C., and preferably treated with dehydrating agents, e.g., alcohol or hydrochloric acid. A light-coloured product is obtained which is free from both the colour and taste of the blood. (a) BLOOD is decolorised by means of a bleaching agent or coagulated and the albumins filtered off, washed, dried, and treated with a tannin solution, or they may be treated with tannin before filtration. In both cases the precipitate gradually adsorbs as much tannin from the solution as is contained in the tannin-albumin compound obtained as described under (a). The product has a light-grey colour.—D. W.

Organic derivatives of calcium iodide; Manufacture of —. W. Spitz. G.P. 318,343, 11.3.16.

STABLE, non-deliquescent compounds of therapeutic value are prepared by mixing aqueous or alcoholic solutions of calcium iodide and of a free or combined amino-acid or urea; double compounds of the two constituents separate from the solution by crystallisation or evaporation. Examples are: Glycocoll calcium iodide, $\text{CaI}_2(\text{CH}_2\text{NH}_2\text{COOH})_2 \cdot 2\text{H}_2\text{O}$, white, prismatic needles, decomp. above 275° C. without melting; urea calcium iodide,



white tables, m.p. 167.5° C.; glycylglycine calcium iodide, $(\text{C}_2\text{H}_5\text{N}_2\text{O}_2)_2 \cdot \text{CaI}_2 \cdot 2\text{H}_2\text{O}$, white needles, decomp. above 200° C.; alanine calcium iodide, $(\text{C}_3\text{H}_7\text{NO}_2)_2 \cdot \text{CaI}_2 \cdot 2\text{H}_2\text{O}$, m.p. 115° C.—L. A. C.

Paraldehyde; Production of —. Konsortium für Elektrochem. Ind. G.P. 319,368, 30.8.17.

AFTER the conversion of acetaldehyde into paraldehyde

hyde by acid catalysts, these acids are removed by treating the cooled reaction product with an excess of the solid salt of a weak acid, such as a fatty acid or boric acid. In this way the formation of aldol or crotonaldehyde can be avoided, whilst the paraldehyde and unaltered aldehyde can be removed and separated by distillation.—D. F. T.

Cymene; Process of treating [nitrating] —. Selden Co., and J. McC. Selden. E.P. 142,226, 20.2.19.

SEE U.S.P. 1,314,920 of 1919; J., 1919, 813 A.

Colourless organic compounds. E.P. 141,440. See III.

Naphthalene derivatives. G.P. 319,253. See III.

Electrolytic reduction. G.P. 303,303. See XI.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic science; Studies in —. H. J. Channon. Phot. J., 1920, 60, 164—173.

THE effect of intensity variation on contrast is not regular but is a function of the plate. With very weak intensities the maximum density obtainable with very prolonged exposures is a function of the intensity and of the plate, but is generally much smaller than the density obtainable by an equal exposure of short duration and high intensity. Reversal can be produced by continued exposure to a weak intensity after production of a latent image by normal exposure. A similar reversal can be produced by exposure to (not necessarily contact with) metallic zinc, with the difference that if reversal is produced by light action the plate sensitiveness is destroyed, but not if reversal is produced by zinc. The production of a positive by development in white light is shown to be partly due to the screening effect of the image first formed, and partly due to an actual loss of sensitiveness by the exposed and partially developed film. A number of experiments are described, for details of which the original paper should be consulted.—B. V. S.

Photographic sensitisers; Studies on —. I. The isocyanine dyestuffs. W. H. Mills and W. J. Pope. Phot. J., 1920, 60, 183—198.

SEVERAL members of the isocyanine class of dyes, produced by the condensation of 1 mol. of a quinaldinium alkyl-iodide with 1 mol. of a quinaldinium alkyl-iodide have found important use as colour-sensitisers of photographic emulsions, the best-known being Pinaverdol (British, Sensitol Green). Their general constitution is probably best represented by the formula $(R)N.C_6H_4(4):CH.(2)C_6H_4.N(R)I$ (see also Mills and Wishart, Trans. Chem. Soc., 1920, 117, 579—587). The preparation and properties of 20 isocyanines are described, all of them having been examined as to their sensitising effect on photographic plates by bathing in a solution of 1:75,000. The parent body (1,1'-dimethylisocyanine iodide) is a strong sensitiser, sensitising up to about 6400; the introduction of a third methyl into the 6-position gives a still stronger sensitiser (Pinaverdol). The replacement of the first two methyl groups by heavier groups (ethyl, *n*-propyl, and *n*-butyl) reduces the sensitising effect in increasing amount. The introduction of a third methyl group into the 6'-position reduces the sensitising effect, as does also the introduction of two more methyl group into the 2'- and 6-positions, or of a cyano or phenyl group into the 2'-position. The 1,1'-diethyl compound (Ethyl Red) is similar to the 1,1'-dimethyl compound, but a somewhat weaker sensitiser, and the addition of

other radicles into it has a similar effect. The introduction of the amino-group into the 5-, 6-, or 6'-position in 1,1'-dimethylisocyanine iodide increases the sensitising power to about the same extent in each case, but the introduction of the acetamino group has only a slight depressing effect; in the 6'-position, stronger in the 6-position, and very strong in the 5-position. The effect of the cinnamoyl-amino group in the 6-position is less than that of the acetamino group. The 1,1'-diethyl-6'-ethoxy-6'-methoxy compound is a strong sensitiser, corresponding with the German Pinachrome.

—B. V. S.

PATENTS.

Motion-picture film. W. V. D. Kelley, Assr. to Prizma, Inc. U.S.P. 1,337,775, 20.4.20. Appl., 8.7.18.

A FILM is provided on both sides with a sensitive emulsion containing a symmetrical design in insensitive material. Colour-sensation negatives are printed on both sides and the positives stained in suitable colours to give natural colours additively.

—B. V. S.

Actinometers. E.P. 141,825. See XXIII.

XXII.—EXPLOSIVES; MATCHES.

Nitroglycerin manufacture; Process for reducing the loss of nitric acid and glycerin in —. F. Hofwimmer. Z. ges. Schiess- u. Sprengstoffw., 1919, 14, 361—366, 381—383, 393—395. Chem. Zentr., 1920, 91, II., 620—621.

IN the production of nitroglycerin about 15% of glycerin is lost, owing to the solubility of the three-nitrates in the waste acid and wash-waters. Ten experimental runs were made in which the only variables were the proportions of glycerin and nitration acid. After a normal nitration the waste acid contains 0.7—0.8% of nitroglycerin. By mixing it with an equal quantity of 20% oleum it may be used for the manufacture of nitric acid. It is advisable to neutralise even the first wash water with soda in order to reduce the amount of dinitrate dissolved. Tables are given showing comparative figures for yields of tri- and dinitrate, temperatures, and amount of soda used. The neutralisation process showed a gain of 30—40% as regards time and consumption of compressed air, and of 60—80% of water. The life of the plant was also considerably extended.—W. J. W.

Nitrocellulose; German stability tests and a new hot storage test (75° C.) for —. F. Lenze and B. Plens. Z. ges. Schiess- u. Sprengstoffw., 1919, 14, 377—380, 395—399. Chem. Zentr., 1920, 91, II., 622. (Cf. J., 1920, 281 A.)

EXAMINATION by the manometric and the NO methods of 7 samples of collodion cotton and 6 samples of gun-cotton, which had considerably deteriorated after 13—15 years' storage in a magazine and 1—4½ years' exposure in the open gave no clear indication of their relative stability. The hot storage test, on the other hand, gave satisfactory results. The presence of unstable portions in gun-cotton cannot always be determined with certainty, but in this respect the hot storage test again is the most reliable.—W. J. W.

Mercury fulminate; By-products in the manufacture of —. A. Langhans. Z. ges. Schiess- u. Sprengstoffw., 1919, 14, 366—369, 399—402. Chem. Zentr., 1920, 91, II., 621—622. (Cf. J., 1920, 281 A.)

CRYSTALS are formed chiefly in the curved pipe leading to the coke-tower. They probably consist of a non-explosive double salt of mercurous nitrate

and an oxide of mercury. The condensation product in the receivers is similar to the crystals. White needles of variable composition are produced by precipitation of the condensation liquors with hexamethylenetetramine. It is not possible to separate the mercury entirely from the liquors. With 10% "formin" solution a compound of "formin" and mercurous nitrate is formed. In the estimation of the nitric acid in the liquors by means of nitron, the results differ by 2–3% from those obtained by titration. Greater accuracy is secured by a preliminary evaporation of the neutralised condensation liquors, by which means organic compounds, esters, etc., are precipitated.—W. J. W.

Recovery of nitrous gases. Walker. See VII.

PATENTS.

Cellulose; Nitration of —. C. Claessen. G.P. 298,418, 3.11.15.

CELLULOSE is mercerised by means of sodium hydroxide, washed, and dried, the material being then unravelled before nitration.—W. J. W.

Coloured smoke. E. C. Weisgerber. U.S.P. 1,339,142–50, 4.5.20. Appl., 10.1.19.

COLORLED smoke-producing compositions are composed of potassium chlorate and lactose together with ammonium iodide and iodine; iodine; litharge and iodine; lead iodide; copper iodide and iodine; ammonium iodide, iodine, and magnesium; mercury sulphide, cobalt oxide, and iodine; mercurous iodide; or naphthalene.

Nitrocellulose; Purification of —. J. Duclaux. E.P. 130,619, 2.8.19. Conv., 25.8.13.

SEE F.P. 461,785 of 1913; J., 1914, 222.

Explosives; Manufacture of —. G. A. Hedberg. E.P. 140,967, 28.8.19.

SEE U.S.P. 1,335,785 of 1920; J., 1920, 429 A.

Detonators; Composition for —. E. C. R. Marks. From E. I. Du Pont de Nemours and Co. E.P. 142,303, 7.5.19.

SEE U.S.P. 1,313,650 of 1919; J., 1919, 742 A.

Artificial clouds, fogs, or mists; Process and apparatus for production of intense —. P. Weiss and J. Verdier. U.S.P. 1,338,343, 27.4.20. Appl., 10.12.17.

SEE E.P. 127,289 of 1916; J., 1919, 553 A.

XXIII.—ANALYSIS.

Potassium chlorate as a standardising substance for solutions of alkali. H. B. Van Valkenburgh. J. Amer. Chem. Soc., 1920, 42, 757–760.

POTASSIUM chlorate is a suitable substance for standardising solutions of alkali. The chlorate is dried at 240° C. for 4–6 hrs., dissolved, the solution boiled in an Erlenmeyer flask and sulphur dioxide passed in for 30 mins., boiling being continued for a further 10 mins. to expel the excess of sulphur dioxide. Phenolphthalein is added and the solution titrated, either hot or cold, with alkali. The calculation of the strength of the alkali is based on the equation: $\text{KClO}_3 + 3\text{SO}_2 + 3\text{H}_2\text{O} = 3\text{H}_2\text{SO}_4 + \text{KCl}$.—J. F. S.

Carbon; Determination of — [in organic substances]. L. Lescoeur. J. Pharm. Chim., 1920, 21, 257–263.

A QUANTITY of the organic substance, e.g., sugar, oxalic acid, sodium acetate, urea, etc., containing 0.05–0.2 g. of carbon, is mixed in a covered silver crucible with 10 g. of a mixture of equal weights of sodium nitrate and potassium nitrate, and a

quantity of N/2 sodium hydroxide solution (free from carbonate) is added so that at least 10 mols. of NaOH are present for each atom of carbon. The mixture is dried and then fused until all carbon has been oxidised. After cooling the mass is dissolved in hot water, ammonium chloride and calcium chloride are added, and the precipitated calcium carbonate is collected, washed, and titrated with N/1 hydrochloric acid, using methyl orange as indicator.—W. P. S.

Manganese; Different methods of estimation of — and their use in the examination of plant ashes and similar products. D. H. Wester. Rec. Trav. Chim., 1920, 39, 414–422.

OF four colorimetric methods for the estimation of traces of manganese examined only Marshall's method (Chem. News, 1901, 76), in which the manganese is oxidised to permanganate by potassium persulphate, was satisfactory. This method is not vitiated by the presence of small amounts of nitric or sulphuric acid or silver nitrate, and the amount of persulphate used is without influence. During the process of oxidation the heating on a water bath should not exceed 30 mins. The presence of salts likely to be found in plant ashes does not interfere with the estimation, although it is preferable to remove chlorides by a preliminary evaporation with sulphuric acid. The presence of more than 0.05 g. of ferrie salts per 100 c.c. makes it impossible to carry out the colorimetric comparison.—W. G.

Potash; The De Roode-perchloric acid method for determining —. T. E. Keitt. J. Ind. Eng. Chem., 1920, 12, 276–277.

SULPHATES are removed by treating the hot solution of the sample with hydrochloric acid and a slight excess of barium chloride; the solution is then cooled, diluted to a definite volume, and an aliquot portion of the clear liquid is evaporated several times to dryness with the addition of *aqua regia*. The residue is dissolved in 20 c.c. of hot water, 5 c.c. of perchloric acid (sp.gr. 1.12) is added, the mixture evaporated on a hot-plate until copious fumes are given off, and then cooled. The residue should be solid when cold; it is again evaporated with the addition of water and perchloric acid, the residue, when cold, treated with 20 c.c. of 95% alcohol, stirred, and the insoluble portion collected after 30 mins. on a weighed asbestos filter, washed twice with alcohol containing 0.2% of perchloric acid, then twice with alcohol-ether (1:1), dried at 120° C., and weighed. The weight of the residue is multiplied by 0.34 to obtain the amount of K_2O present.

—W. P. S.

Iodic acid as a microchemical reagent for soluble and insoluble compounds of calcium, strontium, or barium. G. Deniges. Comptes rend., 1920, 170, 996–998.

If a drop of an aqueous solution of a soluble salt of calcium, strontium, or barium, or a suspension of a finely divided insoluble salt in a drop of water is placed on a microscope slide and one drop of a 10% aqueous solution of iodic acid is added a crystalline precipitate, characteristic for each of the metals, is formed and may be observed under a microscope. If barium is present as the sulphate, this salt should first be reduced to the sulphide in a reducing flame before applying the test. Calcium iodate or periodate should be ignited and the test applied to the resulting iodide.—W. G.

Chromium; Influence of atmospheric oxygen on the iodometric determination of —. O. Meindl. Z. anal. Chem., 1919, 58, 529–548.

ATMOSPHERIC oxygen reacts with the hydriodic acid-chromic acid mixture and causes the results obtained in the iodometric determination of chromium

to be too high. The error increases with increase in the time of contact.—W. P. S.

Tin and antimony; Separation of —. Estimation of tin by cupferron. A. Kling and A. Lassieur. *Comptes rend.*, 1920, 170, 1112—1114.

THE solution of the two metals (0.3–0.4 g. at the most) in hydrochloric acid and potassium chlorate is neutralised with sodium hydroxide in the presence of methyl orange. It is then acidified with hydrochloric acid, and 5–6 g. of tartaric acid is added and the solution warmed till perfectly clear. After cooling the solution is transferred to a conical flask lined with wax, treated with 10 c.c. of hydrofluoric acid, and after $\frac{1}{2}$ hr. 10 g. of sodium acetate is added, and the solution is diluted to 300 c.c. The antimony is then precipitated as antimony sulphide, which is filtered off, washed, dissolved in hydrochloric acid and potassium chlorate, and subsequently re-precipitated, collected, dried at 300° C. in a current of carbon dioxide and weighed. 10 g. of boric acid is added to the filtrate from the first precipitation of the antimony sulphide, and the solution is boiled with a little hydrogen peroxide. After cooling a 10% solution of cupferron is added, and the white precipitate which forms is filtered off, washed with cold water, dried, and calcined, the tin being weighed as stannic oxide.—W. G.

Cupferron; Use of — in quantitative analysis. G. E. F. Lundell and H. B. Knowles. *J. Ind. Eng. Chem.*, 1920, 12, 344–350.

ALTHOUGH cupferron (nitrosophenylhydroxylamine ammonium) has been used successfully for the separation of iron from manganese, and iron and titanium from aluminium and manganese, and for the determination of copper, iron, titanium, zirconium, thorium, and vanadium, the methods are subject to the action of so many interfering substances that they offer little advantage over the usual well-known methods.—W. P. S.

Nitrosophenylhydroxylamine (cupferron); Salts of —. Uranous salt. V. Auger. *Comptes rend.*, 1920, 170, 995–996.

IN neutral solution cupferron precipitates all the metals except the alkalis, the salts obtained being either simple salts, insoluble in chloroform and decomposed by dilute acids or complex salts, soluble in chloroform and more or less resistant to strong acids. Uranyl salts do not give a precipitate with cupferron, but if they are first reduced with zinc in acid solution then the whole of the uranium is precipitated as a uranous salt, $(C_6H_5N_2O)_4U$, crystallising from chloroform in brown prisms. By this means vanadium and uranium may be separated by successive precipitation.—W. G.

Uranium; Estimation of —. R. Schwarz. *Helv. Chim. Acta*, 1920, 3, 330–346.

AMMONIUM sulphide and ammonia are the simplest and most reliable precipitants for uranium. The former is best employed at 80° C. in presence of ammonium chloride; some ammonium uranate is always formed, and complete separation from alkali metals is only attained by repeating the precipitation. Precipitation with ammonia should be performed at 100° C. in presence of ammonium chloride, and preferably not in glass vessels owing to the tendency of ammonium uranate to carry down silica with it. The precipitates are best weighed as uranosouranic oxide, ignition being carried out in porcelain crucibles with access of air rather than in platinum. The uranyl phosphate method of separation from alkali metals is unsatisfactory owing to the fineness of the precipitate. Of volumetric processes, good results are obtained by the solution of uranosouranic oxide in sulphuric acid, but not by attempting complete reduction to uranous salts. Uranium in presence of aluminium

is best determined titrimetrically. Aluminium is incompletely precipitated by ammonium carbonate in presence of ammonium chloride, and some uranium is carried down; good results are, however, obtainable by this method by following a complicated procedure which is described. The separation of iron from uranium by ammonium carbonate and ammonium sulphide is quite effective. Chromium salts must first be oxidised to chromates, preferably by alkaline hypobromite, alternatively by ammonium persulphate. Sodium uranate, when precipitated in presence of chromates, always contains uranyl alkali chromates; the precipitation must therefore be repeated, and it is also advisable to redissolve the uranosouranic oxide finally obtained in nitric acid to separate it from silica. Evidence is given in favour of the existence of the oxide, U_2O_3 , and its formation by heating uranosouranic oxide at 112° C. in a stream of carbon dioxide. (*Cf. J.C.S.*, June.)—J. K.

Halogen in organic substances; Determination of —. W. A. Van Winkle and G. McP. Smith. *J. Amer. Chem. Soc.*, 1920, 42, 333–347.

THE sample is volatilised in a current of air, and the vapour is passed through a quartz tube heated for a length of about 25 cm. to 900°–1000° C. The products of combustion pass to an absorption tube through which is kept flowing a mixture of 25 c.c. of 5N sodium hydroxide solution and 10 c.c. of 2N sodium sulphite. The sulphite serves to reduce any oxyhalogen salts which may be formed. The time taken for a combustion is 1–2 hrs. The excess of sulphite in the absorbing solution is oxidised with potassium permanganate and the halogen is determined by the Volhard process. With ethyl bromide and iodide, ethylene chloride, allyl bromide, chloroform, chloropierin, bromobenzene, etc., results equal or superior to these obtained by the Carius method were obtained. A slightly modified form of the process can be used for less volatile substances, such as hexachlorobenzene, *m*-chloronitrobenzene, and *p*-bromoaniline.—E. H. R.

Nitrogen in organic compounds; Detection of —. Castellana's reaction. J. Flieringa. *Pharm. Weekblad*, 1920, 57, 3–4.

A POSITIVE reaction may be obtained with nitrogen-free compounds in Castellana's reaction (heating substance with alkali carbonate and magnesium and testing for cyanide; *Gaz. Chim. Ital.*, 1904, 34, ii., 357) if the magnesium is in excess owing to combination of atmospheric nitrogen. The proportion of sodium carbonate to magnesium should be 2:1, and the monohydrated carbonate should be used. Sugar should be added to the mixture to eliminate error due to combined atmospheric nitrogen.

—W. J. W.

Mineral constituents in organic substances; Determination of — especially in those containing phosphorus. J. Grossfeld. *Chem.-Zeit.*, 1920, 44, 285–286.

CERTAIN substances, *e.g.*, proteins, blood, etc., burn with difficulty, and the ash contains unburnt carbon. If, however, the substance is mixed previously with magnesium acetate solution and dried, it burns readily, yielding a white ash, and there is no loss of phosphorus. Allowance must be made for the quantity of magnesia added as magnesium acetate.—W. P. S.

Gas analysis; Automatic methods of — depending on thermal conductivity. E. R. Weaver, P. E. Palmer, H. W. Frantz, P. G. Ledig, and S. F. Pickering. *J. Ind. Eng. Chem.*, 1920, 12, 359–366.

THE method described depends on the fact that when a wire surrounded by a gas is traversed by a constant electrical current under conditions where

all loss of heat except by conduction through the gas is excluded, the wire attains a temperature equilibrium depending on the thermal conductivity of the gas. If the wire has a high temperature coefficient of electrical resistance, its resistance will have a value corresponding to the thermal conductivity of the surrounding gas and, therefore, to the composition of the latter. The apparatus, in which a balanced bridge is adopted, consists of two similar tubes, not more than 1 cm. in diameter, bored in a brass rod; each chamber thus formed has a platinum wire stretched down its centre, and the necessary electrical connexions and gas inlets and outlets are provided. One chamber serves for the gas under examination and the other for a comparison gas. The apparatus may be adapted for automatic recording.—W. P. S.

See also pages (A) 438, *Water in petroleum etc.* (Dean and Stark); *Sulphur in oils* (Waters). 446, *Sodium nitrate* (Butt); *Iodide and bromide* (Baughman and Skinner); *Alum* (Codwise); "*Oil*" *in sulphur* (Bushnell and Clark). 454, *Assaying losses* (Dewey). 455, *Tungsten* (Henth, also Erlich). 458, *Saponification value* (Pardee). 460, *Tanning materials* (Baldracco and Camilla). 461, *Acidimetry of soils* (Knight); *Kjeldahl method* (Scales and Harrison); *Sugars* (Hildt); *Worts etc.* (Windisch and Dietrich). 462, *Hop bitter acids* (Lüers and Baumann). 463, *Saccharin in beer* (Baumann). 464, *Milk* (Ambühl and Weiss); *Dyes in butter* (Gilmour). 465, *Alkaloids* (Heiduschka and Wolf); *Quinine* (Bamberger). 466, *Chlorobenzenes* (Bourion and Courtois); *Methyl alcohol* (Schryver and Wood). 467, *Acetates* (Curtman).

PATENTS.

Photometers, actinometers, and the like. F. J. Hargreaves. E.P. 141,825, 25.1 and 25.7.19.

A STANDARD light-source for use in photometers and actinometers consists of a fluorescent screen activated by a radio-active substance; the screen and radio-active material may be either as separate elements or in conjunction. Several forms of suitable holders etc. for the light source, and also a 4-leaved iris diaphragm for use with the apparatus are described.—B. V. S.

[Ball] *hardness testing machines.* W. and T. Avery, Ltd., and H. N. Cox. E.P. 142,323, 27.5.19.

Iron and steel. G.P. 319,255. See X.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Benson. Treating volatile compounds. 15,383. June 7.

Boby, Ltd., and Jennings. Apparatus for separating dust etc. from gases. 14,501. May 27.

Elektro-Osmose A.-G. 15,775. See XI.

Feldenheimer and Plowman. Catalysts and catalytic reactions. 14,397. May 26.

Fothergill. Evaporators. 14,988. June 2.

Goldschmidt A.-G. Preparation of solid substances for chemical operations. 15,479. June 8. (Ger., 25.7.18.)

Green and Lewis. Separating volatile liquid from solution. 14,785. May 31.

Hamilton and Co., Pile, and Stone. Pyrometers of thermo-couple type. 15,678. June 10.

Lilienfeld and others. 15,746. See XI.

Patton. Drying machines. 15,414. June 8.

Petree. Apparatus for separating impurities in suspension from liquids. 15,182. June 1.

Ramnaud. 15,019. See XVII.

Sharples. Separating substances from liquids. 15,577. June 9.

Soc. Gén. d'Evaporation Proc. Prache et Bouillon. Continuous and systematic lixiviation. 15,868. June 11. (Fr., 7.4.11.)

COMPLETE SPECIFICATIONS ACCEPTED.

3638 (1919). Fowler. Apparatus for subjecting air or gas to the action of liquid in the form of spray. (143,291.) June 2.

5606-8 (1919). Benjamin. Tunnel kilns. (143,652-3 and 143,974.) June 9 and 16.

8952 (1919). Allen. See VII.

9642 (1919). Jenkins. Absorbing gases. (143,365.) June 2.

12,335 and 12,338 (1919). Short and Watkins. Drying machinery. (143,707-8.) June 9.

13,367 (1919). Gombert. Drying processes and apparatus. (144,080.) June 16.

17,796 (1919). Zaeckel. Condenser for distilling apparatus. (143,428.) June 2.

22,286 (1919). Bruman. Intermixing liquids, gases, etc. (143,775.) June 9.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Blyth. Production of powdered fuel. 14,178. May 25.

Bone and Roberts. Manufacture of coke. 15,194. June 4.

British Thomson-Houston Co. (General Electric Co.). Filaments etc. 15,276. June 5.

Duckham and others. 14,379. See III.

Frazer. Gas scrubbing and washing apparatus. 15,266. June 5.

Granger, Mariller, and Soc. Anon. Exploit. Proc. Evaporatoires. Distilling crude mineral oils, volatile hydrocarbons, etc. 15,126. June 3. (Fr., 26.11.19.)

Iriy, Kaysser, and Löwenstein. Apparatus for semi-coking of coal, shale, etc. 15,778. June 10. (Ger., 28.10.18.)

Klönne. Regenerative chamber furnaces. 14,289. May 25. (Ger., 27.2.20.)

Lamplough, and Synthol, Ltd. Oil cracking. 14,288. May 25.

Lewis and Quain. Purification of oils containing sulphur compounds. 14,868. June 1.

Perry. Apparatus for distilling carbonaceous material. 15,341. June 7.

Poore. Retorts for destructive distillation of carbonaceous substances. 15,386. June 7.

Schöndeling. Quenching coke. 15,750. June 10. (Ger., 21.8.15.)

Standard Oil Co. Method of selecting grades of lubricants for internal-combustion engines. 15,209. June 4. (U.S., 10.1.20.)

Swinburne. Cracking hydrocarbon oils. 15,246. June 5.

Traube. Recovery of ethylene from gaseous mixtures. 15,192. June 4. (Ger., 14.7.19.)

Tully. Producing and utilising gas. 15,489. June 8.

Umpleby. Gas producers and/or retorts. 15,152. June 4.

Welford. 15,535. See VII.

COMPLETE SPECIFICATIONS ACCEPTED.

4339 (1919). Davies. Carbonisation and gasification plant. (143,602.) June 9.

5079 (1919). Berk and Co., and Hood. Purification of coal gas. (143,641.) June 9.
 7870 (1919). General Chemical Co. Treating gases containing carbonic oxide. (124,760.) June 16.
 8445 (1919). Smith. Briquet-carbonising furnace. (125,381.) June 2.
 8635 and 8638 (1919). Jackson (Twitchell Process Co.). Sulphonated products of mineral oils. (143,681-2.) June 9.
 8662 (1919). Smith. Gas-producers. (143,353.) June 2.
 9361 (1919). Corthesy and Castelli. Generating gas from coal etc. (143,361.) June 2.
 10,951 (1919). Roos. Production of explosive mixtures for use in internal combustion engines. (144,044.) June 16.
 11,413 (1919). West, Wild, and West's Gas Improvement Co. Retorts for distilling carbonaceous materials. (141,051.) June 16.
 11,418 (1919). Marks (U.S. Industrial Alcohol Co.). Liquid fuel. (144,052.) June 16.
 15,125 (1919). U.S. Industrial Alcohol Co. Motor fuel for aeroplane and other motors. (128,916.) June 16.
 31,688-9 (1919). Soc. Indus. de Prod. Chimiques. See VII.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

Anderson and Meikle. Treatment of blast-furnace tar. 14,567. May 28.
 Duckham, Morgan, and Thermal Industrial and Chemical Research Co. Stills for distilling tar, oils, etc. 14,379. May 26.
 Granger and others. 15,126. See II.
 Kinzberger and Co. Purification of anthraquinone. 14,457. May 27. (Austria, 20.9.17.)
 Kinzberger and Co. Purification of crude anthracene. 15,354 and 15,454. June 7 and 8. (Austria, 30.10.16 and 31.3.17.)
 Medsforth, and South Metropolitan Gas Co. Purification of anthraquinone. 14,275. May 25.
 Pestalozzi. Producing tar of aliphatic compounds or low-temperature tar. 15,651. June 9. (Switz., 20.6.19.)
 Stanier, and South Metropolitan Gas Co. Manufacture of naphthylamine sulphonic acids. 14,276. May 25.

COMPLETE SPECIFICATIONS ACCEPTED.

19,686 (1918). Barrett Co. Process of sulphonating benzene. (122,169.) June 9.
 8643 (1919). Jackson (Twitchell Process Co.). Alkali metal sulphonate. (143,683.) June 9.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

Badische Anilin u. Sodafabrik. Manufacture of yellow colouring matters for dyeing animal fibres. 15,457. June 8. (Ger., 14.4.14.)
 Bloxam (Act.-Ges. f. Anilinfabr.). Manufacture of ortho-oxy-azo dyestuffs. 15,515. June 8.
 Bloxam (Act.-Ges. f. Anilinfabr.). Manufacture of azo dyestuffs for wool. 15,637. June 9.
 Farbenfabr. vorm. F. Bayer und Co. Manufacture of azo dyestuffs. 15,133. June 3. (Ger., 16.7.18.)
 Imray (Monsanto Chemical Works). Manufacture of phthaleins. 14,767. May 31.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Arent. Treating materials to reduce inflammability. 15,946. June 12. (U.S., 26.6.19.)

Atomized Products Corp. Treating waste sulphite liquors. 14,387. May 26. (U.S., 16.11.18.)
 Bronnert. Manufacture of artificial silk. 14,632. May 28.
 Dreaper. Manufacture of artificial threads or filaments. 14,314. May 26.
 Dreyfus. Manufacture of cellulose products. 15,736. June 10.
 Levy. Production of artificial filaments. 15,780. June 10.
 Soc. Chim. des Usines du Rhône. Manufacture of cellulose esters. 15,104. June 3. (Fr., 20.6.19.)
 Sutherland. Making pulp board. 15,370. June 7.

COMPLETE SPECIFICATIONS ACCEPTED.

13,529 (1919). Kashtani. Treating cotton yarn or cotton cloth. (144,083.) June 16.
 31,188-9 (1919). Gillet et Fils. Treatment of vegetable fibres. (136,833 and 144,204.) June 9 and 16.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Bloxham (Act.-Ges. f. Anilinfabr.). Dyeing skins, hairs, feathers, etc. 15,636. June 9.
 Butterfield. Compound for stiffening and proofing textile fabrics etc. 15,502. June 8.
 Drey and Moseley. Detergents and bleaching compounds etc. 15,393. June 8.
 Jowett. Printing etc. processes for textile fabrics. 15,068. June 3.
 Poulson. Sizing textile fabrics etc. 14,820. June 1.
 Straub und Co. Production of coloured ornamental designs on fabrics. 15,677. June 10. (Switz., 1.3.20.)
 Taylor. Bleaching. 15,917. June 12.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

American Smelting and Refining Co. Making sulphur from sulphur dioxide. 15,121. June 3. (U.S., 9.9.18.)
 Badische Anilin u. Sodafabr. 15,458. See XVI.
 Berend, and Chem. Fabr. Dr. K. Albert. Preparing colloidal metals, metalloids, and compounds of same. 15,055. June 3.
 Carbic, Ltd., and Waterhouse. Manufacture of calcium carbide cakes. 15,603. June 9.
 Carpmal (Bayer u. Co.). Furnaces for manufacture of hydrochloric acid and sodium sulphate. 14,466. May 27.
 Carpmal (Bayer u. Co.). 15,191. See IX.
 Danckwardt. Producing anhydrous aluminium chloride. 14,944. June 2.
 Dutt and Dutt. Production of sulphur and calcium from gypsum. 14,827. June 1.
 Norsk Hydro-Elektrisk Kvaelfstofaktieselskab. Absorbing nitrous gases. 15,017. June 2.
 Swinburne. Manufacture of anhydrous chlorides. 15,245. June 5.
 Union Carbide Co. Manufacture of calcium carbide. 14,372-3. May 26. (U.S., 20.1.15 and 15.8.17.)
 Welford. Treatment of shales, residues, clay, or aluminous material. 15,535. June 9.

COMPLETE SPECIFICATIONS ACCEPTED.

5580 (1919). Ross. Obtaining copper sulphate from ores. (143,973.) June 16.
 5904 (1919). Rideal and Tarrant. Synthetic production of ammonia. (143,341.) June 2.
 8881 (1919). Damman. Process for purifying rock salt. (144,022.) June 16.

8952 (1919). Allen. Evaporating plant for brine etc. (144,023.) June 16.
 9784 (1919). Shadbolt and Grainger. Manufacture of ammonium sulphate. (144,030.) June 16.
 19,388 (1919). Soc. Indus. de Prod. Chimiques. Converting sodium monochromate into bichromate or chromic acid. (131,289.) June 2.
 19,978 (1919). Fairrie. Kilns for pyrites. (144,142.) June 16.
 31,688 (1919). Soc. Indus. de Prod. Chimiques. Recovering ammonium sodium sulphate from coke-oven and like gases by the use of sodium bisulphate. (136,833.) June 9.
 31,689 (1919). Soc. Indus. de Prod. Chimiques. Recovery of ammonia from coke-oven and like gases. (136,831.) June 16.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Bailey, and Oven, Kiln, and Saggars Committee of British Pottery Manufs. Fed. Plastic mixture for manufacture of saggars for firing pottery etc. 14,940. June 2.
 Foster and Sankey. Method of ascertaining contraction of earthenware, bricks, etc. during firing, and temperatures at which same were fired. 14,586. May 28.
 Ueda. Glass matting process. 15,973. June 12. (Japan, 30.11.17.)
 Welford. 15,535. See VII.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Carpmael (Bayer u. Co.). Manufacture of solutions of silicic acid and silicate cements, and silicate phosphate cements therefrom. 15,191. June 4.
 Clifton. Concrete. 15,804. June 11.
 Hyde. Treatment of cement, concrete, brick-work, etc. 15,955. June 12.
 Schol. Manufacture of stones etc. by compression from sand etc. 15,331. June 7. (Ger., 25.2.18.)
 Schol. Manufacture of artificial stones. 15,761. June 10. (Ger., 15.2.18.)

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Anderson and Meikle. 14,567. See II.
 Angel. Treatment and reduction of sulphides, refractory etc. ores. 14,334. May 26.
 Berend and others. 15,055. See VII.
 Braek and Stevens. Rotary furnaces for smelting etc. 14,884. June 1.
 Caspersson. Alloys of iron. 15,007. June 2.
 Cayo and Whiteley. Crucible etc. furnaces. 14,903. June 1.
 Cornelius. Producing zinc or zinc and lead. 14,484. May 27.
 Deuts.-Luxemburgische Bergwerks u. Hütten A.-G., and Klinkenberg. Manufacture of refined steel. 14,698. May 29. (Ger., 3.7.18.)
 Elektro-Osmose A.-G. Flotation process of concentrating ores. 14,625. May 28. (Ger., 29.7.18.)
 Gregory. Manufacture of iron, steel, manganese, ferro-manganese, etc. 14,631. May 28.
 Krupp A.-G. Extracting metals from ores, foundry products, furnace dust, etc. 15,884. June 11. (Ger., 3.6.19.)
 Lawson and Warren. Electro deposition of metals. 15,593. June 9.
 Liebig. Roasting fine granular sulphide ores. 14,597. May 28.
 Metallind. Schiele u. Bruchsalser. Welding aluminium to iron. 15,762. June 10. (Ger., 3.6.18.)

Miles. Alloy. 14,485. May 27.
 Moffat and Sutherland. Reduction of metallic sulphide ores. 15,519. June 8.
 Morgan, and Thermal Industrial and Chemical Research Co. Process for detinning iron. 15,516. June 8.
 Soc. Metallurgique de la Bonneville. Ternary alloys. 14,709. May 29. (Fr., 19.1.20.)
 Spafford. Manufacture of blister steel from wrought iron. 15,419. June 8.
 Trent. Treating ores etc. 15,732. June 10. (U.S., 10.7.19.)
 Tyrrell. Briquetting ores. 14,791. May 31. (U.S., 29.5.19.)
 Vautin. Recovering metals from slags. 14,858. June 1.

COMPLETE SPECIFICATIONS ACCEPTED.

4082 (1919). Stock. Manufacture of grey iron. (143,596.) June 9.
 4622-3 (1919). Wade. See XI.
 5381 (1919). Ayala. Solder for aluminium. (123,993.) June 16.
 5573 (1919). Hole. Electric smelting furnaces. (125,363.) June 16.
 5580 (1919). Ross. See VII.
 6210 (1919). Webster. Method of malleablising cast metal. (143,989.) June 16.
 7636 (1919). Cowper-Coles. Process and apparatus for sherardising. (143,674.) June 9.
 10,141 (1919). Comp. d'Appl. Méc. Steel particularly for ball and roller bearings. (126,289.) June 16.
 11,655 (1919). Musser. Centrifugal amalgamator and method of obtaining metal from ore. (143,383.) June 2.
 16,585 (1919). Luttenschlager. Cementation of iron and steel. (143,746.) June 9.
 16,813 (1919). Cohn. Alloys. (144,119.) June 16.
 21,038 (1919). Deuts. Molybdän-Werke. Production of alloys of iron and molybdenum. (131,896.) June 9.
 27,153 (1919). British Thomson-Houston Co. (General Electric Co.). Alloy steels. (144,185.) June 16.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Elektro-Osmose A.-G. Electro-osmotic separation of substances. 15,775. June 10. (Ger., 8.4.18.)
 Higgins, Pritchard, and United Alkali Co. Manufacture of graphite electrodes. 15,349. June 7.
 Jungner. Primary galvanic batteries and electrodes. 15,924. June 12. (Sweden, 17.6.19.)
 Kilburn (Norske Akt. for Elektrokemisk Industri). Electric furnaces. 15,441. June 8.
 Lawson and Warren. 15,593. See X.
 Lilienfeld, and Metallhank u. Metallurgische Ges. Electrical precipitation of solid materials from gases. 15,746. June 10. (Ger., 6.3.16.)
 Oldham and Oldham. Galvanic batteries. 15,205. June 4.
 Pechkrantz. Manufacture of metallic diaphragms of electrolytic cells. 15,861. June 11. (Switz., 12.6.19.)
 Pouchain. Electric accumulators. 14,500. May 27.
 Webb. Cathodes for electrolyzers. 15,286. June 5.

COMPLETE SPECIFICATIONS ACCEPTED.

3693 (1919). Moore. Electric furnaces. (143,292.) June 2.
 4622-3 (1919). Wade (Central Mining and Investment Corp.). Electrolytic recovery of metals from solutions. (143,619 and 143,620.) June 9.
 5573 (1919). Hole. See X.
 15,439 (1919). British Thomson-Houston Co. (General Electric Co.). Control for electric furnaces. (143,737.) June 9.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Durden. Apparatus for extracting oils from nuts, seeds, etc. 15,887. June 11.
 Elektro-Osmose A.-G. Decolorising liquids containing glycerin. 15,882. June 11. (Ger., 12.2.19.)
 Fry. Treatment of flax seed. 15,141. June 4.
 Schoonderwaldt. Preparation of substitutes for linseed oil. 14,358. May 26.
 Vakil. Refining oils and fats. 14,623. May 28.

COMPLETE SPECIFICATIONS ACCEPTED.

4366 (1919). Calvert. Treatment of oils, fats, etc. (143,321.) June 2.
 25,528 (1919). Bloxam (Arbini). Neutralising oils and fats. (144,176.) June 16.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Bloxam (Act.-Ges. f. Anilinfabr.). Manufacture of resinous substances. 15,774. June 10.
 Scholz and Tiedemann. Working up scrap linoleum into new linoleum. 14,693. May 29. (Ger., 24.1.20.)
 Schoonderwaldt. 14,358. See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

COMPLETE SPECIFICATIONS ACCEPTED.

4551 (1919). Wade (Goodyear Tire and Rubber Co.). Manufacture of rubber compounds. (143,610.) June 9.
 18,906 (1919). Burney (Westwood). Method of coagulating latex. (144,133.) June 16.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Badische Anilin u. Sodafabr. Manufacture of readily soluble tanning extracts or agents. 15,456 and 15,609. June 8 and 9. (Ger., 29.1.14 and 19.12.13.)
 Elektro-Osmose A.-G. Tanning or impregnating materials. 14,626 and 14,704. May 28 and 29. (Ger., 30.11.18 and 9.7.19.)

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Badische Anilin u. Sodafabr. Transforming ammonia into a salt for use as a fertiliser. 15,458. June 8. (Ger., 10.7.15.)
 Nitrum A.-G., and Schellenberg. Production of a solid neutral fertiliser containing nitrogen and phosphorus. 15,708. June 10. (Switz., 28.6.19.)

XVII.—SUGARS; STARCHES; GUMS.

APPLICATION.

Rambaud. Evaporating and concentrating apparatus for syrups etc. 15,019. June 2. (Fr., 10.6.19.)

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Clayton and Wissler. Brewing. 15,110. June 3.
 Klein. Making vinegar. 15,685-6. June 10. (U.S., 12.1.16.)
 Mapleton. Manufacture of non-alcoholic beer. 14,654. May 29.

COMPLETE SPECIFICATIONS ACCEPTED.

11,294 (1917). Geere and Geere. See XIX.
 13,338 (1919). Rogers and Bedford. Manufacture of alcohol. (144,079.) June 16.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Carpmael (Bayer u. Co.). Disinfecting seed. 15,373. June 7.
 Gould (Carolene Co.). Food products. 14,942. June 2.
 Imperial Trust, and Kidd. Preserving fruits, vegetables, etc. 15,976. June 12.
 Krizek. Apparatus for securing alcohol from baking bread. 15,757. June 10.
 Sgalitzer. Manufacture of food preparation from fresh blood. 14,624. May 28. (Ger., 15.1.16.)
 Shimizu. Manufacture of tea essence. 15,114. June 3.

COMPLETE SPECIFICATIONS ACCEPTED.

11,294 (1917). Geere and Geere. Manufacture of foods or stimulants for promoting growth of yeast. (143,938.) June 16.
 7236 (1919). Peyton and Ferguson. Preserving meat, fish, etc. (143,669.) June 9.
 10,489 (1919). Ames. Apparatus for aerating sewage etc. (143,369.) June 2.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

British Dyestuffs Corp., and Green. Manufacture of chlorides and anhydrides of organic acids. 15,103. June 3.
 British Dyestuffs Corp., and Green. Manufacture of symmetrical alkylated metaphenylenediamines. 15,864. June 11.
 Chem. Fabr. Griesheim-Elektron. Manufacture of acetaldehyde from acetylene. 14,516. May 27. (Ger., 10.8.16.)
 Du Pont de Nemours and Co. Producing tetra-substituted ureas. 15,621. June 9. (U.S. 11.9.18.)
 Soc. Chim. Usines du Rhône. Preparation of dialkylaminoethyl derivatives of theobromine. 14,465. May 27. (Fr., 1.3.20.)
 Soc. Chim. Usines du Rhône. Preparation of twice-substituted 2,4-diketo-tetrahydro-oxazols. 15,280. June 5. (Ger., 16.2.20.)

COMPLETE SPECIFICATIONS ACCEPTED.

4018 (1919). Soc. des Aciéries et Forges de Firminy. Producing acetic acid from paraldehyde. (124,195.) June 2.
 6094 (1919). Mailhe. Production of methyl derivatives of alklamino compounds. (124,219.) June 9.
 13,293 (1919). Thompson (Poulenc Frères, Senderens, and Aboulenc). Production of benzoic acid. (143,392.) June 2.
 20,572 (1919). Soc. des Aciéries et Forges de Firminy. Manufacture of ethyl acetate from paraldehyde. (131,600.) June 9.
 443 (1920). Weiss. Manufacture of oxyaldehydes and their ethers. (139,153.) June 2.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Bloch and Renwick. Photographic materials and processes. 14,398. May 26.
 Klein. Producing photographic negatives for colour printing, colour photography, etc. 15,226. June 4.

XXIII.—ANALYSIS.

APPLICATION.

Svenska Aktiebolaget Mono. Analysing gas-mixtures. 14,621. May 28. (Sweden, 28.5.19.)

I.—GENERAL; PLANT; MACHINERY.

Volatile substances; Manipulation of —. W. A. Stock, E. Kuss, and K. Somieski. Ber., 1920, 53, 751—758.

PRECAUTIONS to be taken and special devices found useful in the manipulation of volatile substances in the determination of vapour pressure, fractional distillation, etc., are described. Liquid propylene is a very suitable substance for cooling baths at temperatures in the neighbourhood of liquid air, since it remains highly mobile at this temperature and has a very low melting point. For extremely low temperatures, instead of a cooling bath, a massive aluminium block may be used, in which four holes have been drilled; one of these serves for the introduction of liquid air and is shielded from the remainder by a suitable vulcanite screen; the other holes serve for the introduction of thermometers, experimental vessels, etc. The block itself may be insulated by being suspended in a Dewar vessel. (Cf. J.C.S., July.)—H. W.

Aluminium vessels; Method of removing "scale" from —. J. Grossfeld. Chem.-Zeit., 1920, 44, 402.

SCALE deposited on aluminium vessels, such as kitchen utensils, etc., may be removed by strongly heating the aluminium below its melting point, followed by treatment with water, by which means the deposit is softened and can be readily brushed off. The metal does not become brittle or in any way deleteriously affected. The decomposition of the chalky deposit below its dissociation temperature (835° C.) is probably brought about by the presence of small quantities of organic matter, and may also be assisted by the action of traces of magnesium compounds.—W. J. W.

PATENTS.

Air, gas, or vapours; Apparatus for subjecting — to the action of liquid in the form of spray. C. H. Fowler. E.P. 143,291, 14.2.19.

TWO or more horizontal shafts revolve at high speed within a casing and are each provided with a number of thin discs spaced apart by collars; the sets of discs may or may not interleave, but in any event the discs on one shaft come opposite to the spaces between the discs on the neighbouring shaft. Washing liquid is supplied to the spaces between the discs by jets, and is also picked up by the discs from a sump, the amount of liquid supplied by the jets being only sufficient to carry off (through an overflow) the material washed out of the gas, which passes generally in an axial direction through the apparatus, but is guided by baffles so that it must pass through the spray. (Cf. E.P. 22,282/02, 27,391/07, and 17,696/10; J., 1903, 1121; 1908, 1196; and 1911, 942.)—B. M. V.

Furnaces [; Continuous annealing —]. T. Teisen. E.P. 142,710, 16.7.19.

IN a furnace consisting of a long chamber through which the objects and the heating gases pass in the same direction, the waste gases are drawn off at intervals along the length of the furnace through damper-controlled passages leading to a collecting chamber under the furnace and thence to an outlet or outlets. The goods are thus cooled gradually and the rate of cooling may be increased by cold air admitted at the exit end of the furnace. The combustible is preferably provided by a gas producer situated under the heating end of the furnace, and the air for combustion may be preheated by passing it through passages built in the walls of the furnace. —B. M. V.

Furnace. H. B. Kinnear and A. J. Townsend. U.S.P. 1,339,564, 11.5.20. Appl., 12.5.19.

THE goods are pushed upwards through one or more vertical tubes arranged within a heating chamber. —B. M. V.

Kiln. K. Himrod, Assr. to P. W. McKay. U.S.P. 1,339,705, 11.5.20. Appl., 30.8.16.

IS a kiln with inlet openings communicating with a flue and having an adjacent burner also in communication with the flue, a conduit is arranged between the burner and kiln through which a fluid may be delivered to the flue. Means are provided for generating and superheating steam, for mixing it and the fluid with the combustion products from the burner, and for directing this mixture into the kiln.—W. J. W.

Kiln. J. N. Silva. U.S.P. 1,340,633, 18.5.20. Appl., 28.2.19.

AN intermittent kiln comprises several round down-draught kilns, a feeding flue and an exhaust flue extending past them all, a manifold with its ends connected with the flues and its body encircling each kiln and communicating with the interior thereof, and a single flue common to the kilns and communicating with the central well of each kiln and with the exhaust flue. A branch exhaust flue draws the gases from the main flue and discharges them into the chimney and a pressure fan blows air into the feed flue.—A. B. S.

Kiln; Annular —. G. Zehner. G.P. 315,773, 23.2.16.

AN annular kiln has its crown supported by a series of upright pillars so arranged that there are alternate openings on opposite sides of the combustion channel which may be employed to convey the gases through the kiln in a zig-zag course. The kiln can thus be used alternately as an annular kiln and as a zig-zag oven, or it can be used partly as the one and partly as the other.—L. A. C.

Tunnel kiln. F. K. Meiser. G.P. 319,440, 28.9.18.

THE kiln is suitable for chemical reactions, for ignitions in different gas atmospheres, and for the recovery of vapours and gases from the material under treatment. The material is supported in the kiln in tanks, crucibles, or other vessels connected in series by detachable, movable pipes. The pipes of the individual vessels may dip in a liquid seal of a longitudinal conduit in which movable valves may be arranged so that a gas can be supplied to a definite part of the apparatus.—A. R. P.

Treating air or gases with liquids or vice versa; Apparatus for —. W. J. Bulgin, E. A. Hall, and G. Searle. E.P. 142,941, 17.2. and 29.7.19.

IN a rotary scrubber in which the rotating part dips into liquor within a cylindrical container, the working surface which retains a film of liquor consists of twisted flat strip which may be secured to the rotor in several ways, such as winding involutely between radial arms, or in several layers or zig-zag fashion on flat strip provided with projections to hold the twisted strip in place. Baffles of conical or *vena contracta* shape may be provided to ensure the passage of the air or gas through the effective portions of the rotor.—B. M. V.

Atomising more or less viscous materials; Method and means for —. A. Sonstbagen and E. H. Harberd. E.P. 143,289, 11.2.19.

THE material is spread in a thin layer on a smooth roller or belt moving comparatively slowly, and is removed therefrom by bristles or spikes projecting from another roller or belt which moves much faster, so that the material is thrown off from the projec-

tions by centrifugal force in the form of drops. The bristles or spikes project into the layer of liquid on the first roller or belt, but do not quite touch the roller itself, and just before they enter the liquid they may be bent back by a suitable obstruction and then suddenly released.—B. M. V.

Absorbing gases; Methods and means for — and their application to vacuum flasks for storing low-temperature liquids. H. C. Jenkins. E.P. 143,365, 15.4.19.

GRANULATED or powdered coal is used instead of charcoal for improving and maintaining the vacuum in vacuum flasks.

Condenser for distilling apparatus. J. H. Zaeckel. E.P. 143,428, 16.7.19.

THE vapour passes into a pipe, inclined upwards, from which vertical branches extend downwards into open vessels for the collection of distillates of various degrees of volatility. A number of similar pipe sections may be used, with the lower ends of the inclined pipes alternately on opposite sides.

—B. M. V.

Distillation of liquids; Apparatus and process for the continuous —. F. I. Gibson. U.S.P. 1,324,417, 9.12.19. Appl., 2.8.18.

A STILL is divided into several compartments with restricted communicating passages for the liquor, but all opening to one common vapour space. The liquid is fed continuously to the first compartment and the residue withdrawn from the last compartment.—B. M. V.

Evaporating apparatus. K. L. E. Thunholm. U.S.P. 1,324,417, 9.12.19. Appl., 2.8.18.

LIQUID to be evaporated is fed into the upper end of a vertical cylinder, divided by double horizontal partitions into a number of superposed chambers. The partitions are provided with central pipes, which pass through the upper and lower plates and through which a vertical shaft passes, having arms, carrying rakes which are rotated in each chamber. Passages are also provided through which the vapour passes upward to the outlet at the top and other sealed passages through which the liquid flows down from chamber to chamber to the concentrated liquor discharge at the bottom. Evaporation is effected by steam which passes through the double partitions, each of which is provided with an inlet and an exhaust connexion.—W. H. C.

Electrical precipitators; Method of regulating the velocity of fumes passing through —. W. W. Strong, Assr. to Research Corp. U.S.P. (A) 1,337,488 and (B) 1,337,489, 20.4.20. Appl., 25.9.17.

(A) DEVICES controlled automatically are placed in the different pipes of an electrical precipitator of the multiple pipe treater type in order to equalise the flow of gas therein. (B) A treater chamber has a discharge electrode extending through it and is provided with means for constricting the flow of gas adjacent to the electrode and also for preventing a disruptive discharge from the electrode to the constricting means at voltages below the normal critical one.—B. N.

Electrodes; Removing deposits from — [in electrical precipitators]. A. F. Meston, Assr. to Research Corp. U.S.P. 1,339,471, 11.5.20. Appl., 23.10.17.

MEANS are provided for striking the discharge electrodes of an electrical precipitator intermediate of the ends to cause vibration.—B. N.

[Electrically] separating suspended particles from gases. W. A. Schmidt, Assr. to International Precipitation Co. U.S.P. 1,339,480, 11.5.20. Appl., 27.8.17.

A CLEAN electrode surface, for receiving the precipitated material, is maintained by distributing a liquid in a film over a porous surface constituting the receiving electrode.—B. N.

Gases; Apparatus for the electrical precipitation of dust from —. Siemens - Schuckertwerke G.m.b.H. G.P. 315,931, 29.6.18.

DUST etc. is removed from blast-furnace gas or the like by passing the gas through a number of electrodes consisting of frames in which are mounted a series of pivoted plates arranged, either horizontally or vertically, in a manner similar to that of the laths of a venetian blind. The plates in alternate frames serve as precipitation electrodes and as high-potential electrodes respectively, and are inclined in opposite directions, so that repeated changes of direction are imparted to the gas.—L. A. C.

Dryer. W. B. Hutchinson, Assr. to A. Blackburne. U.S.P. 1,338,731, 4.5.20. Appl., 23.5.19.

In a rotary dryer with screw feed the blades are perforated to enable air to be blown through the screw and dryer in a contrary direction to the feed.—W. J. W.

Drying materials; Process for —. H. B. Lowden. U.S.P. 1,339,771, 11.5.20. Appl., 22.10.17.

THE material is caused to pass over a heated hearth by repeatedly cleaving it in a substantially vertical direction and at the same time imparting to it a slight forward motion.—W. H. C.

Dehydration of moist materials; Apparatus for [electro-osmotic] —. Elektro-Osmose A.-G. (Graf Schwerin Ges.). G.P. 316,443, 4.9.17.

In the apparatus previously described (G.P. 314,338; J., 1920, 53 A), the movable walls rest at their lower end in a bed provided with a run-off conduit for the separated liquid. A channel from one side of each wall, or channels from both sides, pass through the lower end of the wall and open into the discharge conduit.—W. J. W.

Pulveriser; Rotary —. E. Cortese, M. Crespi, and A. Squassi. U.S.P. 1,339,499, 11.5.20. Appl., 29.1.20.

A PULVERISING chamber provided with beater bars attached to wheels mounted on a rotating shaft and also with stationary pulverising means, has a blower, mounted independently on the shaft, which blows the powder and granular material from the beaters against a grid fixed over an opening in the casing.—W. H. C.

Refrigerating apparatus. R. B. Henderson and R. E. James. U.S.P. 1,339,622, 11.5.20. Appl., 13.5.18.

A GENERATING chamber, provided with means by which it can be heated and cooled, is connected by a gas passage and a liquid return pipe with a cooled dehydrating chamber, supported on the generator. The dehydrating chamber is connected with a cooled condenser by a pipe provided with an automatic and also with a manually operated valve. The refrigerant passes at intervals from the condenser to a refrigerating coil by a valved pipe and thence up a vertical pipe back to the generating chamber.—W. H. C.

Photochemical apparatus. W. O. Snelling. U.S.P. 1,339,675, 11.5.20. Appl., 1.3.17.

THE reacting fluids are caused to pass through a series of transparent vessels, each of which has a source of light opposite to it, the distance between

the source of light and the reaction vessel being decreased progressively in the direction of flow of the fluids.—W. H. C.

Separating device; Settling basin —. *Upward-current separating process and device. Slime-pulp thickener.* C. Allen. U.S.P. (a) 1,339,682, (b) 1,339,683, and (c) 1,339,684, 11.5.20. Appl. (a) (b) 3.7.17, (c) 5.10.18.

(A) To prevent irregular discharge at the outlet of a de-watering settler, a diaphragm or other obstruction is placed over the outlet in such a way as to leave an attenuated passage for the settling material for a considerable distance above the orifice. (B) The overflow lip of a settling device is adjustable as to circumferential length in order to vary the velocity of overflow. (C) The size of the outlet orifice of a pulp thickener is varied by means controlled by the level of a fluid of fixed density (e.g., clear water), which is in hydrostatic balance with the settling pulp. The clear fluid chamber is preferably arranged within the settler and provided with an inverted conical baffle underneath.

—B. M. V.

Catalysts; Manufacture of —. J. Boyce, Assr. to The American Cotton Oil Co. U.S.P. 1,339,686, 11.5.20. Appl., 2.6.13.

GRANULATED inert material is mixed with an adherent material so that the individual grains are coated. The coating contains a compound of the catalytic metal, and the coated material is treated to convert the compound to a catalytically active material, e.g., "nickel black."—B. M. V.

Fractionating compound substances containing ingredients of closely-related solubility. A. Seidenberg. U.S.P. 1,340,186, 18.5.20. Appl., 10.8.18.

A NUMBER of solvents are chosen such that the most volatile solvent dissolves the least soluble constituent most easily and the least volatile solvent dissolves the most soluble constituent most easily. The compound substance is dissolved in the mixed solvent, which is then gradually volatilised, so as to precipitate the constituents in the reverse order of their solubility.—B. M. V.

Heat exchanger. Method of exchanging heat. H. D. Hildebrand. U.S.P. (a) 1,340,266 and (b) 1,340,267, 18.5.20. Appl., 2.1.17 and 21.4.19.

TWO fluids, between which interchange of heat is desired to take place, are passed through elements consisting of a number of nested pipe sections, alternate sections of one element being connected with alternate sections of an adjacent element, so that the fluid to be heated flows through progressively smaller annular passages and the fluid to be cooled through progressively larger passages.

—B. M. V.

Crystallisation of salts; Utilisation of the heat liberated during the — from hot liquors. E. Roser. G.P. (a) 316,492, 1.3.18, and (b) 316,493, 20.3.18.

(A) HOT vapours liberated from liquors during crystallisation are conducted into an apparatus in which they are utilised for assisting the solution of the raw material. (B) Additional heating of the raw material and solvent may be effected by means of steam or hot gases in a second apparatus, from which any residual hot vapours are caused to pass into the first apparatus.—W. J. W.

Salt solutions; Apparatus for cooling hot —. Maschinenbau-A.-G. Balcke. G.P. 319,439, 26.6.18.

THE apparatus consists of a series of troughs provided with rotating cooling discs, which are fitted

with paddles, by the help of which the salt crystals and the solution are moved through the troughs.

—A. R. P.

Filtering gaseous substances; Apparatus and process for —. L. B. Fiechter. G.P. 317,254, 31.3.18. Conv., 2.11.17.

THE apparatus comprises a rotating flat screen over which the granular filtering material, e.g., sand, is distributed, and from which it is removed continuously or at intervals, elevated to a purification plant, and then, by means of a vertical shaft with guard rail, returned to the screen in a layer of the requisite thickness.—W. J. W.

Pneumatic separators. Williams Patent Crusher and Pulverizer Co., Assees. of A. J. Roberts. E.P. 139,749, 6.8.19. Conv., 1.3.19.

Separators for grading crushed rock and other materials. T. J. Sturtevant. E.P. 142,989, 9.4.19.

Purification of [used lubricating] oil and other liquids; Apparatus for —. King's Patent Agency, Ltd. From D. Stone. E.P. 142,703, 2.7.19.

Evaporators. K. L. E. Thunholm. E.P. 118,635, 23.8.18. Conv., 23.8.17.

SEE U.S.P. 1,324,417 of 1919; preceding.

Sludge press. W. Buckley. E.P. 143,478, 24.12.19.

SEE U.S.P. 1,316,419 of 1919; J., 1919, 806 A.

Distillation. G.P. 315,012. See III.

II.A.—FUEL; GAS; MINERAL OILS AND WAXES.

Gases of combustion of coal; Approximate determination of the amount of —. F. Castek. Feuerungstech., 1920, 8, 133—136.

IT is shown that the amount of gas produced by the combustion of coal can be calculated from the carbon content or the calorific value of the coal. Using the theoretical quantity of air, the volume of dry combustion gases (G) in cub.m. from one kg. of coal is one-tenth of the percentage of carbon in the coal (C). If v times the theoretical quantity of air is used, $G=0.1Cr$ and $v=18.65/x$, x being the percentage of CO_2 in the gases of combustion. If the calorific value of the coal is B, the carbon content is $B/97\%$.—A. G.

"Double gas" and its use in gas works. H. Strache. J. Gasbeleucht., 1920, 63, 230—235.

THE author advocates the adoption of a system of primary gasification followed by steaming, leading to the production of "double gas." This gas has a calorific value of 3330 cals. per cub.m., as compared with 5500 cals. for ordinary coal gas from the same coal. The amount of coal gas produced per 100 kg. of coal is 33 cub.m. and the amount of "double gas" 160 cub.m., the total calorific values being 425,100 cals. in the former case, assuming the coke to be used for the production of water-gas, and in the latter case 533,400 cals. The minimum calorific values of the gas and tar are given as 60.2% and 8.3% of the heat of the coal respectively. Advantages claimed for the process are:—Reduced floor-space, apparatus always ready for use, reduction in labour and low initial cost. The yield of by-products is greater than if coal- and water-gas are produced separately and then mixed. The tar (sp. gr. 1.012) contains: water, 33.7%, pitch, 18.9; paraffin, 1.5; light oils, 19.4; heavy oils, 24.3; ammonia, 0.91; insol. in benzol, 1.65; and ash, 0.28%.—A. G.

Gas producer plants operating with recovery of low-temperature tar and ammonium sulphate; Economics of —. E. Roser. Stahl u. Eisen, 1920, 40, 349—357, 387—395.

On the basis of tests with coal, lignite briquettes, and mixtures of coal and briquettes carried out on an installation of 15 Thyssen rotary-grate gas producers, the advantages of the gasification of fuel in by-product gas producers are set out in detail for varying conditions as to prices of raw materials and of products.

Gas producer plants operating with recovery of low-temperature tar and ammonium sulphate; Economics of —. Stahl u. Eisen, 1920, 40, 532—541, 610—621, 651—655, 685—689.

A FULL account of a discussion on two communications by Roser (preceding abstract) and Klingenberg (Stahl u. Eisen, 1918, 38, 2—11, 32—36, 46—51, 65—68).

Oxide [gas] purification; Practice and control of —. G. Weyman. Gas J., 1920, 150, 489—490.

THERE are three possible ways of removing hydrogen sulphide from coal gas by oxide purification. The first method is to form ferric sulphide, this being subsequently oxidised to free sulphur. The process is limited to the use of true bog ore or an artificial oxide consisting of precipitated hydroxide, and the presence of an alkali is necessary to neutralise any acid that may be formed. Ammonia is the best alkali to use as it can be distributed most efficiently. The development of heat must be restricted owing to increase in acidity with rise of temperature. For this purpose, as much water should be used as the oxide will carry without being wet. In the second process, ferrous sulphide is the intermediate product, and since acid conditions are favourable higher temperatures may be employed. Natural ores and burnt oxide can be used with good results, but the process should not be employed if the purifying area is below 0.5 sq. ft. per 1000 cub. ft. per 24 hrs. The oxide should be worked as dry as possible, and 50% of sulphur should be obtained without more than one removal. In the third process, which has not yet been applied to coal gas purifiers, most of the sulphur is oxidised direct to sulphuric acid and ferrous sulphate is produced which may be removed in solution. The temperatures used must be higher than usual and the conditions must be acid. Success in any of these methods depends on the material used, the plant available, and the temperature which can be maintained. Improved results have been attained by the passage into the purifiers of preheated air, and steam gives better results than water for moistening the oxide. The author advocates careful temperature control, determination of the ammonia and tar fog in the inlet gas and of the hydrogen sulphide at the outlet of the first two boxes. The liquid drainage should be examined regularly and the spent oxide tested for ammonia.—A. G.

Petroleum; Constitution of organic nitrogen bases of Californian —. C. F. Mabery and L. G. Wessen. J. Amer. Chem. Soc., 1920, 42, 1014—1020.

A RE-EXAMINATION of the basic constituents of Californian petroleum (J., 1900, 505) has shown them to consist mainly of alkylated quinelines (or isoquinolines). All the fractions boiling over intervals of 2°—4° covering a range of 130°—283° C. at 50—90 mm. gave pyridinepentacarboxylic acid and a methylpyridinetetracarboxylic acid on oxidation with potassium permanganate, and therefore contain at least three alkyl groups, of which the relatively simple nature is shown by the absence of

propionic or higher fatty acid from the oxidation products. 3-Methylquinoline was obtained by distilling with lime the product obtained by oxidising one fraction with chromic acid, and a medium fraction yielded a phthalone derivative by condensation with phthalic anhydride. (Cf. J.C.S., July.)—J. K.

Ammonium sulphate. Shewring. See VII.

Gas analysis. Ott. See XXIII.

PATENTS.

Coal or other mineral washing apparatus. A. France. E.P. 142,729, 15.9.19.

THE washing troughs are arranged in batteries in cascade form; the dense material discharged from the separators of one trough is subjected to another washing in a trough below, this process being repeated until the necessary grade is obtained.—A. G.

Coal; Process for treating —. J. N. Wingett, Assr. to W. A. Haggott. U.S.P. 1,337,496, 20.4.20. Appl., 3.5.15.

COAL is hardened, preparatory to shipment or storage, by heating it during its passage through a closed chamber, sufficiently to drive off certain volatile constituents adjacent to the surface only. The heavier oils are caused to move to the surface and dried, giving the coal a hard exterior, protecting it against exposure to the atmosphere, and preventing the formation of dust and slack.—B. N.

Coke; Manufacture of hard — from gassy coals. F. Saefel. G.P. 317,120, 9.5.18.

THE charge for coking consists of a mixture of the raw coal and coal which has been partly de-gasified.—W. J. W.

Coke oven with vertical heating flues. C. Otto u. Co. G.P. 317,962, 22.8.16.

A NUMBER of flues are built in immediate contact with the furnace side of the oven wall and others are built within the wall and communicate above with one another. Combustion takes place from below upwards in a number of narrow pipes, which communicate above with the heating flues by means of separate channels built into the upper portion of the oven wall, or by one continuous channel, in such manner that the products of combustion pass downwards through the heating flues with eddying motion.—J. S. G. T.

Chamber oven for gas production. H. Bröcker. G.P. 315,099, 23.3.18. Addition to 314,118 (J., 1920, 5 A).

THE oven comprises an upper and lower chamber separated by means of an inclined partition extending part of the way across the oven, and a slide which may be withdrawn when desired to afford communication between the chambers. The upper chamber serves as a carbonising chamber and can be put in communication with or cut off from the hydraulic main as desired. The lower chamber functions as a gas producer and is fed with hot coke from the upper chamber by withdrawing the slide.—J. S. G. T.

Coal; Gasification of — and obtaining by-products therefrom. C. H. Smith. E.P. 123,738, 17.2.19. Conv., 16.2.18.

CAKING coal is subjected to a preliminary distillation at temperatures below that at which substantial quantities of ammonia pass off, being agitated continuously and the gas and tar withdrawn until the coal loses its caking properties. The residue is subsequently treated in a by-product gas producer, to obtain producer gas or water gas, with recovery of ammonia. The distillation takes place at 800°—950° F. (about 430°—510° C.).—A. G.

Fuels; Gasifying of solid and liquid —. N. H. Freeman. E.P. 142,945, 19.2.19.

STEAM is heated to a temperature exceeding the ignition point of the fuel to be gasified and is applied to the fuel in this highly heated condition in a chamber in which the temperature is maintained at or above the ignition point of the fuel. In one type of apparatus oil is fed into a stream of steam concentrically through a jet, whilst if pulverised coal is used it is discharged through a hopper into the steam chamber.—A. G.

Carbon-hydrogen gas; Process of producing —. T. O'Connell. U.S.P. 1,310,495, 18.5.20. Appl., 10.9.19.

THE first layer of ignited coal in a gas producer is subjected successively to an air blast and a steam blow, both of equal length and directed from beneath the coal, to another air blast and steam blow similar to the former, to an air blast, to a steam blow directed from the top and lasting $\frac{1}{2}$ of the air blast, to a steam blow from the bottom lasting $\frac{1}{2}$ of the air blast, to a succession of two periods of operation, each period comprising an air blast and a steam up-blow of equal length, followed by three operations, each consisting of an air blast, a steam down-blow of $\frac{1}{2}$ of the blast period, and a steam up-blow of $\frac{1}{2}$ of the blast period. Coal is deposited in the centre of the fire after the last steam up-blow, and this is followed by two periods of air blast and steam up-blows of equal length and the process continued in the above described order, including the coaling, until the last coaling operation before cleaning of the fire has taken place and has been followed by a double air blast and steam up-blow, the coal thus prepared being subjected up to the point of cleaning to a series of operations consisting of air blasts, steam down-blows of $\frac{1}{2}$ of the air blasts, and steam up-blows of $\frac{1}{2}$ of the air blast periods.—A. G.

Gas-producer with annular charge. J. Schulte. G.P. 316,412, 3.1.17.

THE charge is contained within the producer in a tall annular chamber. By means of helical baffles the gas is caused to take a tortuous path in the spaces surrounded by and surrounding the annular chamber and is drawn off from the respective regions by separate pipes each provided with a separate cock and leading into a common pipe. Partitions are provided in the spaces within and without the annulus to force the gas into more intimate contact with the walls of the producer chamber.—J. S. G. T.

Gas producer. Maschinenfabrik Augsburg-Nürnberg A.-G. G.P. 317,220, 18.3.17.

THE stepped grate of the producer, which is relatively long in horizontal cross-section, is carried on a horizontal pipe which is turned about its axis alternately in opposite direction. The fuel is thereby broken up by being pressed alternately against one or other of the side walls of the producer.—J. S. G. T.

Ethylene; Compression, storage, etc. of —. A. A. Backhaus, Assr. to U.S. Industrial Alcohol Co. U.S.P. 1,339,431, 11.5.20. Appl., 6.6.19.

A CYLINDER suitable for commercial transportation has its interior free from absorbent filler and solvent and contains ethylene compressed to a high pressure, the amount of ethylene being more than $1\frac{1}{2}$ times the amount of hydrogen in a cylinder of the same size under the same pressure.—A. G.

Blast-furnace gas; Cleaning of —. N. K. Turnbull. E.P. 142,938, 15.2.19.

THE gas is filtered and is then purified, the cooled gas being passed in counter-current to the incoming

crude hot gas, thus being reheated and being in a more suitable condition for combustion in stoves, boilers, and the like or for use in gas engines.

—A. G.

Gases; Method and apparatus for cleaning —. F. R. McGee. U.S.P. 1,338,143, 27.4.20. Appl., 13.5.18.

THE cleaner comprises a gas-distributing chamber, a collecting chamber connected with the distributing chamber by a series of conduits and an impurity-receiving chamber provided with a gas outlet. The conduits have annular outlets opening into the impurity-receiving chamber and means in each conduit for imparting a whirling motion to the gases passed through them.—A. G.

Gasoline; Manufacture of —. J. W. Trotter. U.S.P. 1,339,727, 11.5.20. Appl., 14.11.18.

PARTLY refined hydrocarbon oil is converted into gasoline by spraying it through a "submerged catalytic spray" into a bath of oil under a pressure of not less than 75 lb., in the presence of superheated steam and at a cracking temperature.—C. A. M.

Ovens [for distillation of coal]; Method and apparatus for producing a regular progression of materials in vertical —. Soc. du Four Vertical Continu. E.P. 121,199, 3.3.19. Conv., 1.3.18.

Coke ovens; Discharging apparatus for continuous —. Soc. Anon. d'Ougree Marihay. E.P. 135,217, 15.11.19. Conv., 28.9.16.

Gas generators and the like; Preventing escape of gas through poke-holes of —. Façonseisen-Walzwerk L. Mannstaedt & Co. A.-G., and H. Bansen. E.P. 133,969, 16.10.19. Conv., 25.7.16.

Gas retorts [for domestic use]. E. A. Groundstroem. E.P. 142,689, 6.6.19.

Gas producers [for self-propelled vehicles; Automatic control of operation of] —. D. J. Smith. E.P. 143,353, 5.4.19.

[Coke] oven with regenerators. C. Berthelot. U.S.P. 1,340,104, 11.5.20. Appl., 15.8.19.

SEE E.P. 129,994 of 1919; J., 1920, 395 A

Fuel; Liquid —. E. C. R. Marks. From U.S. Industrial Alcohol Co. E.P. 143,017, 5.5.19.

SEE U.S.P. 1,313,158 of 1919; this J., 1919, 711 A.

Electrical precipitation. G.P. 315,931. See I.

Sludge acid. U.S.P. 1,339,519. See VII.

Ammonium sulphate. G.P. 299,622. See VII.

Purifying gases. G.P. 300,035. See VII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

PATENTS.

Asphalt rock, bituminous slate, etc.; Furnaces for the distillation of —. A. la Porta and R. do Bartolomeis. E.P. 142,511, 3.2.19.

A VERTICAL shaft furnace comprises a lower zone for the spent material and a middle combustion zone wherein part of the combustible matter of the rock etc. is burnt to provide the necessary heat for the distillation of the remainder in the third, or upper, zone, which is wider than the combustion zone but not so wide as the spent material zone.—A. G.

Decolorising carbon; Preparation of an active —. Zellstoff-fabrik Waldhof. G.P. 309,155, 22.6.17.

A DECOLORISING carbon with a greater efficiency

than wood or animal charcoal is obtained by carbonising the residues derived from the hydrolysis of cellulose or cellulosic materials.—W. J. W.

Gases from anthracite and petroleum coke; Treatment of — [in manufacture of carbon electrodes]. F. K. Meiser. G.P. 317,179, 19.6.17.

IN the manufacture of electrodes from anthracite and petroleum coke the gaseous products are mechanically freed from alkaline vapours at a temperature sufficiently high to prevent condensation of the tar. They may then be employed for heating the furnace without risk of attacking the firebrick.—W. J. W.

Electric discharge tubes and the like; Methods of and apparatus for automatically removing gases from —. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 141,913, 30.4.19.

THE operation of an incandescence cathode vacuum device is controlled during evacuation by means of a high-potential transformer which supplies operating current from its secondary to the device, with a resistance in the primary circuit. A low-potential transformer for heating the cathode is supplied with current from the primary circuit of the high-potential transformer, and the gases disengaged during the action are removed from the container.—B. N.

Incandescence bodies; Manufacture of —. W. Heseler, G.m.b.H. G.P. 319,613, 28.7.18.

INCANDESCENCE bodies in tubular or massive form are obtained by pouring artificial silk or like material into appropriate moulds. The incandescence material may be admixed with the silk or incorporated subsequently to casting.—J. S. G. T.

III.—TAR AND TAR PRODUCTS.

Chlorination of benzene; Kinetics of the —. F. Bourion. Comptes rend., 1920, 170, 1319—1321.

IN the chlorination of industrial benzene in the presence of iron as a catalyst the yield of monochlorobenzene increases with the velocity of chlorination, but decreases slightly with rise in temperature. For a given concentration and a given time the molecular proportion of benzene converted into monochlorobenzene is 8.5 times greater than the molecular proportion of chlorobenzene converted into *p*-dichlorobenzene.—W. G.

Benzene separated from industrial chlorobenzenes; Impurities in —. F. Bourion. Comptes rend., 1920, 170, 1181—1184.

THE chief impurity present in the benzene obtained by the process of enrichment from industrial chlorobenzene (J., 1920, 425 A), is chloroform, minor impurities being hexane and heptane.—W. G.

p-Phenylenediamine and aniline; Preparation of — from the corresponding chlorobenzenes. A. J. Quick. J. Amer. Chem. Soc., 1920, 42, 1033—1042.

THE catalytic influence of copper salts on the reaction between ammonia and chlorobenzene (G.P. 204,951; see E.P. 3396 of 1908; J., 1908, 1147) or *p*-dichlorobenzene (G.P. 202,170; J., 1908, 1147) is not connected with the formation of copper ammonium compounds, since nickel, cobalt, and zinc salts, which form similar compounds, have no influence. Cuprous salts are more effective than cupric salts, which probably only become effective when reduced; and water cannot be replaced by alcohol as a solvent in the reaction. The reaction with dichlorobenzene is best carried out above 200° C.; with 5—6% solution of ammonia the reaction is very

incomplete, and an amorphous red product is also produced. Addition of iron filings or activated charcoal to the cuprous salt improves the appearance of the final product, but with concentrated solutions of ammonia they, particularly the former, tend to retard the reaction. The action of ammonia on chlorobenzene under the conditions prescribed gave only a 39% yield, as against 80% claimed by the patent. Increase in the concentration of ammonia beyond 14% or in working temperature beyond 200° C. does not improve the yield, although the latter is necessary for complete decomposition of the chlorobenzene. Cuprous iodide has no catalytic value for this reaction. Diphenylamine could not be obtained from bromobenzene and aniline in presence of cuprous iodide under the conditions prescribed in G.P. 187,870 (F.P. 374,385; J., 1907, 756).—J. K.

PATENTS.

Dehydration of liquid mixtures [aqueous phenol] by distillation. K. Kubierschky. G.P. 315,012, 9.5.18.

THE method is applicable to mixtures of liquids which are not completely soluble in water, and in the early stages of distillation give distillates which separate into two layers—one containing much water, the other little water. Two distilling columns, with draw-off pipes for liquid on their lower parts, are connected with a common condenser. The liquid mixture to be dehydrated—e.g., a mixture of water and phenol—is supplied to one of the columns and is rectified, water-free phenol being run off below and water along with a certain amount of phenol passing to the condenser, where the distillate condenses and separates into two layers, a lower containing little phenol and an upper rich in phenol; these are discharged respectively to one or other of the columns and the process continued until finally pure phenol is run off from one and water from the other, keeping pace with the supply of the mixture to the still.—J. S. G. T.

2-Aminoanthraquinone; Process of making —. M. Fischer. Assr. to J. R. Geigy S.A. U.S.P. 1,340,603, 18.5.20. Appl., 8.3.19.

SEE E.P. 127,223 of 1919; J., 1919, 529 A.

Sublimation. E.P. 142,902. See XX.

IV.—COLOURING MATTERS AND DYES.

1-Hydroxy-4-chloroanthraquinone. F. Ullmann and A. Conzetti. Ber., 1920, 53, 826—837.

THE preparation of various derivatives of 1-hydroxy-4-chloroanthraquinone, m.p. 192° C., obtained from 1-hydroxyanthraquinone by the action of sulphuryl chloride in the presence of nitrobenzene at 100° C. is described. N-1-Hydroxyanthraquinonyl-4-anthranilic acid, bluish-violet shining needles, m.p. 272° C. (decomp.) is obtained by boiling a solution of 1-hydroxy-4-chloroanthraquinone and anthranilic acid in amyl alcohol with anhydrous potassium acetate in the presence of copper acetate and copper powder; it is converted by treatment with acetic anhydride and pyridine and subsequent action of *p*-toluenesulphonic chloride on the acetyl derivative thus produced into 4-hydroxyanthraquinone-2.1-acridone, shining blue leaflets, m.p. 321° C., which gives blue shades on cotton from a wine-red vat.—H. W.

Glyoxaline nucleus; Some properties of the —. [Glyoxaline-azo dyestuffs.] F. L. Pyman. J. Soc. Dyers and Col., 1920, 36, 107—112.

A GENERAL survey of the simple glyoxaline derivatives is given. Arylazoglyoxalines dye wool yellow to brown shades; the colour produced by

2-benzeneazoglyoxaline is readily removed by dilute acids, but that obtained by means of 2-*p*-sulphobenzeneazoglyoxaline-4,5-dicarboxylic acid is comparatively fast to washing and to acids. Some of the derivatives of glyoxaline should be suitable as dyestuffs similarly to those of the isomeric compound pyrazole.—L. L. L.

Sulphur; Removal of — from organic compounds by means of arsenic trioxide. W. Herzog. Z. angew. Chem., 1920, 33, 140.

ORGANIC compounds, and especially thioureas and their derivatives, may be readily freed from sulphur by boiling them in an inert solvent (xylene) with arsenic trioxide beneath a reflux condenser. The method is particularly suitable for the preparation of carbodiphenylimide (for use in Sandmeyer's indigo synthesis) from symmetrical diphenylthiourea.—C. A. M.

Picric acid. Doll. See XXII.

PATENTS.

Indophenolic compounds; Manufacture of —. F. à Brassard and Crawford, and E. A. Buckle. E.P. 143,014, 3.5.19.

IN the condensation of *p*-nitrosophenol and the product of which it is desired to make the indophenolic compound it is ensured that these are always reacting in equimolecular proportions by grinding them together before effecting the reaction in presence of concentrated sulphuric acid.

—A. J. H.

Dyestuffs and process of making same. A. Angel; M. L. Angel, extrix. U.S.P. 1,340,696, 18.5.20. Appl. 16.7.18.

SEE E.P. 121,347 of 1917; J., 1919, 101 A.

Photosensitising dyes. U.S.P. 1,338,346 and 1,338,349. See XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cotton; Constituents of raw —. II. E. Knecht and F. V. Fernandes. J. Soc. Dyers and Col., 1920, 36, 43—47 (Cf. J., 1911, 1007; 1919, 7 A.)

TABLES are given showing the percentage of matter extracted from Egyptian and American cotton by different solvents, the effect of heat on the extracts, and their nitrogen content. In samples of untreated and extracted cotton dyed with Methylene Blue there was no diminution in intensity after the cotton had been successively extracted with benzene, alcohol, and water; on the contrary, the colour became, if anything, brighter. An albuminoid substance has been obtained from cotton which yields a coloured lake with Methylene Blue. The amount of tannic acid absorbed by cotton diminishes as the impurities are successively removed. A table is given showing the tensile strength of extracted cotton in the conditioned state and dried at 100° C.—L. L. L.

Wool fibre; Influence of humidity upon the strength and elasticity of —. J. I. Hardy. J. Agric. Res., 1920, 19, 55—62. (Cf. J., 1918, 728 A.)

THE tensile strength of wool increases with decrease in the diameter of the fibres. The breaking strength of fine wool varies with the diameter; that of coarse wool varies with a figure between the first and second powers of the diameter. Increase of humidity to 80% causes a decrease in the tensile and breaking strengths of scoured and unscoured wool, while above 80% a slight increase occurs. The elasticity of scoured and unscoured wool increases with the humidity to 80% and decreases from 80% to the saturation point.—J. I. J.

Wool and cloth; Action of atmospheric influences on —. A. Kertesz. Färber-Zeit., 1920, 31, 1—4. A. Grass. *Ibid.*, 61—68. (Cf. A. Kertesz, J., 1919, 496 A.)

IN reply to Von Kapff (Färber-Zeit., 1919, 30 [23]; cf. J., 1908, 329), who maintains that wool is weakened by chroming and cites in support the decreased durability of certain modern chrome-dyed Army clothing, Kertesz disputes the validity of Von Kapff's measurements, and attributes the weakness of the Army clothing to conditions of manufacture. He strongly supports the observation of Reebberg (G.P. 286,310; J., 1915, 1140; cf. G.P. 303,231; J., 1920, 61 A) that chrome-dyed wool better withstands exposure to air than pure wool. Grass points out that no conclusions as to the durability of dyed wool under ordinary conditions should be drawn from the behaviour of Army clothes since they were exposed to totally abnormal conditions. The value of the experimental work of Kertesz is lessened by the facts that the conditions employed did not correspond with those of industrial methods and the duration of the chroming was not sufficiently prolonged; moreover, it was not shown that the woollen fabric used in the tests was normal.—A. J. H.

Fabrics; Resistance of — to climatic conditions and to ultra-violet rays. L. Vignon. Comptes rend., 1920, 170, 1322—1324.

FABRICS of linen and of chappe silk weighing respectively 110 g. and 100 g. per sq. m. were submitted to varying conditions of light, heat, and moisture, and their breaking strength then measured. The results indicate that the tissues of animal origin (chappe silk) are more resistant than those of vegetable origin (linen) to variations in climatic conditions and to the action of ultra-violet rays, the probable explanation being that the protein molecule is more resistant to hydrolysis than the cellulose molecule.—W. G.

Fabrics coated with solutions of cellulose esters; Dynamometric examination of —. L. Clément and C. Rivière. Chim. et Ind., 1920, 3, 440—444.

THE strength of fabrics is increased by coating with cellulose acetate, a solution of the latter in tetrachloroethane giving a slightly stronger coating than a solution in acetone or benzyl alcohol; tetrachloroethane and acetic acid have no injurious effect on the strength of linen. Oil varnish should not be applied to fabrics coated with cellulose acetate, since it diminishes the strength of the material. A coloured coating is best produced by applying to the fabric first one layer of a tinted solution of cellulose acetate and then three coats of colourless solution. The weakening effect of sunlight on coated fabrics is marked during the summer months, but may be prevented by the use of suitable pigments in the coatings.—W. P. S.

Sulphate cooking process; Control of —. W. E. B. Baker. Pulp and Paper Mag., 1920, 18, 544—545.

CONTINUOUS records of gauge pressure and temperature must be made and gas pressure curves plotted from differences between gauge and steam pressures. Specific gravity determinations should be made at regular intervals on cooled samples. Total sulphur dioxide is estimated by iodine titration, the interference of "reversible sulphur dioxide" being overcome by taking the first blue coloration as the end point. In the determination of "half-bound" sulphur dioxide by plotting the differences between results of the iodine and soda titrations an error not exceeding 0.2% SO₂ is introduced, due to acidity other than sulphurous acid. The curve for changed sulphur concentration, plotted from the differences between total sulphur and sulphur titratable by iodine, is a rising one, the rate of ascent giving an indication of the progress

of the lignone reaction. Reversible or loosely combined sulphur dioxide is estimated by treating the liquor with excess of alkali, acidifying with hydrochloric acid, and titration with iodine, the curve being a rising one and sometimes oscillatory. For lime and magnesia estimations a sample is treated in a sealed tube; the curve falls and again rises, the minimum point being of importance.

—W. J. W.

Lignin and reactions of lignin. P. Klason. Ber., 1920, 53, 706—711.

The naphthylamine ligninsulphonate described previously (J., 1919, 570A) has been investigated further. Its formation is considered to indicate the presence of the group $R.CH:CH.CHO$ in lignin. It is formed in greater quantity from old than from fresh sulphite liquors, and it would appear that the group $CHR:CH.CH_2OH$ is present in the latter, and that the aldehyde, in part, at any rate, is gradually formed in them by atmospheric oxidation. It is concluded that coniferyl aldehyde and coniferyl alcohol are the principal constituent groups at any rate of the lignin of conifers.—H. W.

Lignin in cellulosic materials; Direct estimation of —. E. Becker. Papierfabr., 1919, 17, 1325—1327. Chem.-Zentr., 1920, 91, II., 347.

ONE gram of the finely divided material is mixed with 10—20 c.c. of 72% sulphuric acid by means of a glass rod. When the mass becomes gelatinous a further 80—100 c.c. of 72% sulphuric acid is added, and the mixture allowed to stand for 24 hrs. It is then diluted with 1—1.5 parts of water, passed through a Gooch filter, washed with hot water, dried to constant weight, ignited, and weighed, the difference between the two weights giving the amount of ash-free lignin.—W. J. W.

PATENTS.

Silk; Manufacture of —. J. Korselt. E.P. 108,489, 1.8.17. Conv., 31.5.16.

THE strength and elasticity of unloaded, raw, or boiled silk fibres is increased, and the subsequent weakening of the fibres is avoided or reduced by the use of catalase. In order to accelerate the catalytic action, or to keep the catalase efficient for a longer time, substances such as creatinine may be added, which act as activators on the catalase contained in or added to the silk. The catalase may be obtained by extracting liver with water, precipitating the extract by alcohol, and again extracting the dried precipitate with water. The amount of catalase in the aqueous solution may be found by titration with hydrogen peroxide. The silk is treated in a solution of 0.1—2.0% of catalase at about 40° C. during 20—40 mins.; it is then centrifuged and dried.—L. L. L.

Wool, hair, or the like, and vegetable fibres; Manufacture of —. J. Korselt. E.P. 109,051, 18.8.17. Conv., 19.9.16. (Cf. ante.)

SKINS, wool, hair, feathers, artificial silk, cotton, and other vegetable fibres are strengthened and preserved by treatment with catalase and/or perhydridase. The fibres are treated with a dilute solution of catalase and/or perhydridase for 20—40 mins., and then dried. "Dead" wool may be dyed like normal wool after treatment. Cotton or other vegetable fibres are increased in strength, elasticity, brilliancy, and absorbent power for dyestuffs. The treatment may take place at any desired stage of manufacture—for instance, before spinning, before, during, or after dyeing, or during or after finishing. Perhydridase may be extracted from calf's liver with a solution of 1% sodium bicarbonate and 2% sodium fluoride; this solution may be applied directly to the fibres. The activity of catalase can

be increased by adding 1—5% (calculated on the weight of the wool) of creatinine to the bath.

—L. L. L.

Wool; Apparatus for drying —. S. C. Wilson. E.P. 139,278, 12.2.19.

IN a closed chamber through which hot air is circulated is supported a tier of perforated trays which receive a longitudinal reciprocating motion. The wool is fed on to the uppermost tray by a vibrating feed shoot, moved forward by a series of transverse rollers operating above the tray, and delivered from the end of the tray by means of hinged shoots to the one next below, the wool being opened by means of a roller whilst passing from one tray to the next.

—L. L. L.

Waterproofing fabrics and rendering the same impermeable to gases; Adhesive composition, more especially applicable for use in —. Soc. Anon. des Etabl. Hutchinson. E.P. 129,630, 20.6.19. Conv., 6.7.18.

A COMPOSITION consisting of, e.g., caoutchouc 20 pts., cellulose acetate 60 pts., tetrachloroethane 600 pts., is applied either hot or cold to fabrics for waterproofing them or rendering them impermeable. Sulphur and an accelerator of vulcanisation may be added to the composition, and the coating may be vulcanised. Loading agents or coloured materials may also be added. The material is particularly adapted for forming the envelopes of balloons and aeroplanes.—L. L. L.

Hop bines; Manufacture of textile fibres from —. H. Küchenmeister. G.P. 318,224, 17.11.18.

HOP bines are boiled for several hours with a weak caustic alkali solution and then washed. The process is repeated, and the material is then treated with very dilute acid, washed and dried. The hop bines may be subjected to a preliminary treatment for one or more days with a neutral alkali salt solution, at normal temperature, in order to remove the epidermis. An alkali sulphite may be added to the caustic alkali solution, and the second boiling may be followed by a short treatment under pressure with an alkali bisulphite, or replaced entirely by a similar treatment of longer duration. The wet fibres may be bleached, after treatment, by means of an ammoniacal hydrogen peroxide solution.

—W. J. W.

Fabrics for airships. Vickers, Ltd., J. McKechnie, and A. Ryan. E.P. 142,615, 25.3.19.

FABRIC of cotton or other fibre, having cellulose as its principal constituent, is converted partially or wholly into cellulose acetate; after treatment with a softening agent, such as triacetin or castor oil, and calendering, the fabric is doped and varnished.

—D. F. T.

Bast fibre material; Process of opening out and simultaneously dyeing —. R. Haller. G.P. 318,271, 25.1.19.

BAST fibre raw material is boiled in an alkaline medium with addition of vat dyestuffs such as indigo, thioindigo, N-dihydroanthraquinoneazaine (Indanthrene), and sulphur dyestuffs, which react with the pectins. Alkali sulphides may be added.

—W. J. W.

Wood; Recovery of organic products from —. H. F. Weiss, Assr. to C. F. Burgess Laboratories. U.S.P. 1,339,489, 11.5.20. Appl., 11.8.17. Renewed 21.10.19.

WOOD is ground in a current of water which serves to carry away the pulp; the water is separated from the pulp and used again, this cycle of operations being repeated until the concentration of the organic products in the water is sufficient for the subsequent recovery process.—D. F. T.

Sulphite liquor [for cellulose manufacture]; *Process for regulating [measuring] the acidity of —*. J. B. Crandon, Assr. to Crandon Mfg. Co. U.S.P. 1,340,849, 18.5.20. Appl., 23.11.17. Renewed 25.3.20.

THE liquor is caused to form the electrolyte of an electric battery and the current produced measured. —B. M. V.

Waste liquors from sulphite- and sulphate-cellulose manufacture; Removal of water from —. E. Oman. G.P. 316,592, 26.6.17. Conv., 5.4.17.

WASTE sulphite liquors are kept agitated and subjected to a temperature below their solidification point, any snow or ice crystals formed being removed by filtration or centrifuging. Portions of liquid adhering to the crystals are diluted. The low temperature of the crystals, as well as of the concentrated liquors, may be utilised for cooling further quantities of liquor. The concentration of the liquors may be increased to 40% by this method.

—W. J. W.

Cellulose acetates; Manufacture of —. J. O. Zdanowich. E.P. 139,232, 8.4. and 18.9.18.

THE substitution of organic acids, particularly mono-, di-, and trichloroacetic acids, for sulphuric acid in the production of cellulose esters has not given satisfactory results on account of their being weak acids. If, however, the chloro-acetic acids are employed in the nascent state by adding a halogen to the acetylating mixture, they react more energetically, even at low temperatures, and have no injurious effect on the cellulose molecule at high temperatures. After the production of the mono- and di-acetate of cellulose the esterification is too slow, but the addition of a small quantity of sulphuric acid or other strong condensing agent readily completes the esterification, the cellulose passing into a clear, colourless solution of extreme viscosity. This solution may be used directly for the production of films, artificial silk, horsehair, etc. The material precipitated in the usual way forms a transparent, glass-like, tenacious material, and is soluble in chloroform or acetone.—L. L. L.

Plastic [nitrocellulose] composition. H. H. Dow, Assr. to The Dow Chemical Co. U.S.P. 1,339,552, 11.5.20. Appl., 1.12.19.

NITROCELLULOSE is mixed with an aliphatic hydroxy compound containing from three to five atoms of carbon and with a halogen derivative of the next lower radicle of the same homologous series.

—D. F. T.

Cellulose acetate product; Manufacture of a — unaffected by cold. Knoll und Co. G.P. 298,806, 14.3.16.

CELLULOSE acetate mixtures containing resorcinol monoacetate (m.p. 50° C.) can be moulded when heated and maintain their shape on cooling.

—W. J. W.

Fibres for manufacture of pasteboard suitable for saturating with tar, for roofings, linings, or the like; [Mechanical] process for obtaining —. R. Kron. E.P. 135,466, 2.10.19. Conv., 19.11.18.

Cellulose acetate solutions; Production of —. T. Tyrer; P. A. Chambers and G. T. Feasey, exors. U.S.P. 1,339,728, 11.5.20. Appl., 28.3.19. See E.P. 130,402 of 1918; J., 1919, 714A.

Insulation. E.P. 143,280. See XI.

Carbohydrate in plants, etc. E.P. 123,326. See XVIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Scouring and milling of fancy woollen piece goods. S. Begg. J. Soc. Dyers and Col., 1920, 36, 38—43.

THE author discusses briefly different types of piece scouring machines, and the scouring, soap milling, and acid milling of piece goods. Many advantages are obtained by acid milling compared with soap milling, particularly in the selection of dyestuffs, time of milling, and saving of soap and water.—L. L. L.

Aniline Black; Method for producing ungreenable —. A. Scheurer. Sealed note 1414, 28.8.13.

Bull. Soc. Ind. Mulhouse, 1919, 85, 292. Report by M. Battagay, *ibid.*, 293—294.

THE mixture for producing the Aniline Black consists of 35 pts. of potassium ferricyanide, 35 pts. of potassium chlorate, 65 pts. of aniline, 105 pts. of hydrofluosilicic acid of 20° B. (sp. gr. 1.16), and 760 pts. of a 12.5% starch solution. This is quite stable and does not attack steel, nor tender the fibre. The colour may be developed by steaming or ageing at 45° C. for 2 days, and is quite ungreenable by sulphurous acid. Battagay reports that the process is in accordance with the modern view that greenable blacks are produced with a strongly acid mixture, and ungreenable blacks with a feebly acidic mixture.—A. J. H.

Anthocyan; Tinctorial properties of the —. A. E. Everest. J. Soc. Dyers and Col., 1920, 36, 47—53.

THE colouring matters of the anthocyan, anthocyanin, and anthocyanidin group have an affinity for tannin-mordanted cotton, and produce fine strong shades that are fast to light; this affinity is not affected by the number, nor by the position of the hydroxyl groups, nor by ethoxy or glucoside residues, but is dependent upon the presence of the pyrylium ring. These pigments will be practically useless for technical dyeing practice. The colours do not lack tinctorial power but lack fastness to washing and are sensitive to acids and alkalis.—L. L. L.

Drying of printed fabrics; Use of steam in the —. E. Boeringer. Bull. Soc. Ind. Mulhouse, 1919, 85, 298—411.

THE general conditions governing the evaporation of water are discussed and their application to the drying of fabrics is indicated. Capillary forces are shown to influence largely the drying of a wet fabric. The methods of drying by means of hot flues, tenter frames, hot air chambers, and the usual drying cylinders are examined critically with regard to efficiency and the theoretical principles involved. Various improvements are suggested.

—A. J. H.

Finishing processes; Influence of — on woven fabrics. E. Midgley. J. Soc. Dyers and Col., 1920, 36, 35—38. (*Cf.* J., 1919, 102A.)

THE author discusses the influence of crabbing on union and wool goods; the mechanical manipulation and chemical treatment of cotton goods as illustrated in mercerising and schreinerling; and defects developed during dyeing and finishing. Unbalanced strains are present in a woven fabric as it leaves the loom, and unless the material is reduced to a state of equilibrium there would be a great tendency for the fibres and yarns to shrink irregularly and give the fabric a wrinkled or cockled appearance and make it unsaleable. During crabbing the yarn swells and becomes plastic, and in this condition readily takes any position forced upon it; this position is permanent if maintained until the fibre becomes cool. The pressure during

crabbing is an essential factor, and consequently the tension during winding is important. The degree of crabbing must be varied according to the quality of the material, the structure of the yarn, the weave and structure of the fabric, and the type of finish required. The most difficult defect to account for is produced during dyeing and finishing, namely "crimps." It is produced where the wet processes develop the shrinking properties inherent in the fabric, and being due to uneven shrinkage and movement of the fibre, an absence of uniformity in either materials or treatment during the change from fibre to finished cloth is indicated. This type of defect is also produced by artificial conditioning of yarn on bobbins.

—L. L. L.

Tannins and cotton. Wisdom. See XV.

PATENTS.

Colour effects; Method of and means for producing certain — on textile and other fabrics and materials. The Calico Printers' Assoc., Ltd., E. Schofield, and F. Farnworth. E.P. 139,373. 24.4.19.

COLOUR (or a resist, discharge, etc., mixture) is applied to one side of the fabric in such a manner as to insure saturation and penetration to the other side, and subsequently another colour or colours are applied to the opposite side by allowing them to drip or drop on to the fabric. For example, the fabric is passed through a suitable machine and the first colour is applied to one face by means of a rotating brush or the like. After drying and brushing, the travelling fabric passes under regulated taps or equivalent appliances for supplying a second colour or colours, and if desired catching troughs may be automatically interposed between the taps and the fabric to produce an intermittent or "sarric" effect.—L. L. L.

Dyeing, scouring, and washing wool and other fibrous materials; Machines for —. J. and R. Whitaker. E.P. 142,602, 14.3 and 10.9.19.

IMPROVEMENTS are claimed in the side tipping discharge movement for the hinged inner chamber of machines of the type described in E.P. 101,060 and 108,809 (J., 1916, 961; 1917, 1045).

Hosiery-dyeing machine. Fabric-treating device. Fabric-treating machine. Dyeing device. H. M. Dudley. U.S.P. (A) 1,330,246, (B) 1,330,247, 10.2.20, (C) 1,331,743, and (D) 1,331,744, 24.2.20. Appl., (A) 19.7.18, (B) 21.1.19, (C) 18.2.18, (D) 27.2.18.

(A) A SERIES of dyeing chambers with non-perforated ends and foraminous sides are mounted on a common axis. Means are provided for revolving the chambers, for passing liquid through them in opposite directions, and for impinging the liquid evenly upon the foraminous sides of the chambers. (B) A rotary dyeing chamber contains a rotary fabric chamber. Means are provided for continuously revolving the two chambers and for passing a liquid and a gas through them. (C) A receptacle contains a series of rotary hollow foraminous reels upon which fabric may be wound. Means are provided for revolving any reel while the others are held stationary, and some of the openings of the reels may be closed by means of a sliding device. Provision is made for continuously forcing liquid outwards from the interior of each reel or for continuously drawing liquid into the interior of each reel through fabric wound thereon. (D) A series of dyeing chambers is provided with sectional foraminous cylinders upon which fabric may be wound. A resilient device is provided for forcing the foraminous cylinders against the inner faces of the rolled fabrics, and a device for forcing liquid through the fabrics wound upon the cylinders.—L. L. L.

Dyeing machine. H. M. Dudley. U.S.P. (A) 1,333,217 and (B) 1,333,218, 27.4.20. Appl., 25.1 and 6.3.18.

(A) WITHIN a dyeing machine is a series of fibre chambers, perforated at bottom and top (the relative positions of which can be varied), and each containing a sliding perforated plate. A liquid can be forced or drawn from a lower liquid chamber, through the fibre chambers and so into an upper liquid chamber. (B) A dyeing machine contains a series of dyeing chambers, the ends of each being perforated and movable so that their relative positions can be varied and the contained fibre be compressed. A liquid can be forced in one direction and drawn in the opposite direction through the dyeing chambers.—A. J. H.

Dyeing of furs and other materials; Electrical process and apparatus for — and products resulting therefrom. J. E. Bloom. U.S.P. 1,338,353, 27.4.20. Appl., 30.9.19.

FUR, or other fibrous material, is electrified with one sign only, by bringing it in a dry state, under insulated conditions, into physical contact with a conductor extension of one terminal of a direct current supply of suitable voltage and amperage, the other terminal being earthed, and with a suitable increased difference of potential between the electric source and the extension contact.

—B. N.

Hair; Process of treating —. H. R. Anders, Assr. to The Roessler and Hasslacher Chemical Co. U.S.P. 1,331,609, 24.2.20. Appl., 24.5.17.

HAIR is treated with a bath containing zinc dust and acetic acid, then washed with a solution of soap and sodium carbonate, and treated with hydrogen peroxide solution rendered alkaline with ammonia.—L. L. L.

Dyeing; Tablets for [domestic —]. H. Heymann. E.P. 142,899, 11.1.19.

Bleaching, dyeing, finishing, and like machines; Mechanism for stretching and guiding fabrics in —. J. Thornber and A. B. Henshilwood. E.P. 143,347, 25.3.19.

Silk, wool, etc. E.P. 108,489 and 109,051. See V.

Best fibres. G.P. 318,271. See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric acid; Manufacture of — by the chamber process. Kaltenbach. Chim. et Ind., 1920, 3, 407—421.

THE use of a series of tubes is recommended in place of lead chambers; the gases from the Glover tower are passed, in parallel, through the tubes in one or several series. Each tube is packed with a suitable filler and is surrounded by a water jacket. The necessary pumps and pipes are provided for circulating and supplying weak acid or water to the tubes and towers.—W. P. S.

Nitric acid; Production of concentrated — from nitrous gases. F. Foerster, T. Burchardt, and L. Fricke. Z. angew. Chem., 1920, 33, 113—117, 122—127, 129—132.

THE authors contradict their earlier contention (J., 1908, 1110) that only nitric acid with the lowest vapour pressure can be produced by the interaction of weak nitric acid with nitrogen peroxide, oxygen, and water. The process requires a lengthy time of treatment, however, as well as only slight excess of oxygen, and a low gas velocity. These latter factors tend to militate against

effective reaction, and under the most favourable conditions a concentration of only 80% is obtainable. Nitric acid of the highest concentration may be produced by treating the dilute acid with liquid nitrogen peroxide in sufficient excess, and then lightly agitating the mixture with oxygen.

—W. J. W.

Ammonia oxidation [; *Theoretical aspects of* —]. J. Baumann. Chem.-Zeit., 1920, 44, 145–146.

Of the two reactions $2\text{NH}_3 + \text{O}_2 = 2\text{NO} + 3\text{H}_2\text{O}$, and $2\text{NH}_3 + \text{O}_2 = \text{N}_2 + 3\text{H}_2\text{O}$, the latter is more exothermic, and undue heating of the apparatus beyond the contact chamber indicates that the reaction is taking place more in accordance with the second equation, resulting in a low yield. The exothermic character of the reaction is due to combustion of the hydrogen of the ammonia, and a hydrogen flame burning in a tube may give rise to the phenomenon of a "singing flame." The yield diminishes with increasing pitch of the note sounded.—W. J. W.

Nitrous acid; Action of — on coloured indicators.

C. Matignon and G. Gire. Bull. Soc. Chim., 1920, 27, 362–366.

From titrations of a mixture of nitrous and nitric acids in the presence of different indicators, it is shown that, contrary to the conclusions drawn from the heats of neutralisation (J., 1920, 188A), nitrous acid is a much stronger acid than the second acid function of phosphoric acid. This result is confirmed by electric conductivity measurements which indicate that the electrolytic dissociation constant of nitrous acid is 6×10^{-4} as compared with 1.9×10^{-7} for the second acid function of phosphoric acid.—W. G.

Arsenic acid; Iodometric determination of —.

P. Fleury. J. Pharm. Chim., 1920, 21, 385–391.

A QUANTITY of arsenate equivalent to about 50 c.c. of N/10 iodine solution is dissolved in 30 c.c. of water, 3 c.c. of hydrochloric acid (sp. gr. 1.171) is added, and the mixture heated on a boiling water-bath for 5 mins.; sufficient potassium iodide is then added to give a 25% solution, the heating continued for 5 mins., the mixture cooled, and the liberated iodine destroyed by the addition of thio-sulphate solution. Titration of the iodine does not give a reliable measure of the arsenic acid, because a small quantity of iodine is invariably liberated by the action of dissolved oxygen on the hydriodic acid. The colourless mixture is, therefore, treated with an excess of sodium bicarbonate and the arsenious acid titrated with N/10 iodine solution.

—W. P. S.

Solvay ammonia-soda process; Combination of — with processes for manufacturing nitrogen compounds. L. Hamburger. Chem. Weekblad, 1920, 17, 256–258.

AMMONIA produced by any nitrogen fixation process may be utilised for the Solvay process, the latter being worked to yield ammonium chloride, which may be employed for the manufacture of artificial manures. This method obviates the lime treatment in the Solvay process and eliminates the necessity for sulphuric acid in producing fertilisers. In the manufacture of urea from sodium cyanate an ammonium salt of a strong acid may be used.

—W. J. W.

Sulphate of ammonia; Manufacture of neutral —. F. Shewring. Gas World, 1920, 72, 453.

NEUTRAL ammonium sulphate may be obtained by passing ammonia from the weak ammoniacal liquor plant into the evaporating pans by means of a perforated lead pipe. In plants where ejectors are used for removal of the salt, it is important to keep the ammonium sulphate acid, as an alkaline

solution will cause blockages in the condensing plant. In the centrifuges the salt may be sprayed with weak ammonia solution to reduce the acidity, but an entirely neutral salt will not often result. To remove the acidity entirely, the salt is laid on a table with a false bottom, and while still moist is covered by means of a wooden hood provided with a vent pipe. Ammonia gas is passed through a lead pipe, with drilled holes and flattened end, laid beneath the false bottom, and is allowed to pass through the salt till neutralisation is complete.—W. J. W.

Ammonium sulphate; Melting point of normal —.

C. Caspar. Ber., 1920, 53, 821.

WHEN heated in an open tube, normal ammonium sulphate softens at about 310°C ., melts at 336°C ., 339°C ., and decomposes with evolution of gas at 355°C .; in a sealed tube it softens from about 360°C ., and has m.p. 417°C – 423°C . The datum, 140°C ., recorded in the literature is based on a misunderstanding, and really refers to ammonium bisulphate (cf. Watson Smith J., 1895, 629; 1896, 3).—H. W.

Calcium cyanamide; Yields in the manufacture of —. C. Pluvinage. Chim. et Ind., 1920, 3, 438–439.

THE conversion of calcium carbide into cyanamide is never complete under ordinary conditions of manufacture; a portion of the carbide remains unconverted, the nitrogen employed contains neon, argon, etc., and a quantity of cyanogen is formed, the amount varying with the temperature, pressure, and rate of reaction. Further, normal commercial carbide yields about 300 l. of acetylene per 1 kg., a quantity corresponding to 81.87% of pure carbide. If the calcium cyanamide obtained from such carbide contains 22.5% of nitrogen, 19% of the carbide has escaped reaction; if it contains 26% of nitrogen, 2% of the carbide has not reacted.—W. P. S.

Calcium carbide and nitrogen; Variety of carbon formed in the reaction between —. A. Remelé and B. Rassow. Z. angew. Chem., 1920, 33, 139–140.

SMALL quantities of carbonaceous matter remain after the decomposition of calcium cyanamide with water. When purified by fusion with potassium hydroxide or treatment with hydrofluoric acid this matter contained 98.15–99.4% C, 0.13–1.61% H, and 0.43–1.18% of residue. It had sp. gr. 2.250, ignited at 600°C – 610°C ., and had the characters of a pure graphite. (Cf. J.C.S., July.)—C. A. M.

Sulphites; Estimation of —. H. I. Waterman. Chem. Weekblad, 1920, 17, 196–197.

THE oxidation of sulphite solutions in air is especially rapid in presence of hydroxyl ions, for example, in solutions of normal sodium sulphite. Sulphite may be accurately estimated by dissolving the salt in a boiled 5% glycerol solution in a flask filled with carbon dioxide. A measured quantity of this solution is run into a known volume of acidified standard iodine solution under carbon dioxide. The excess of iodine is titrated with thiosulphate.—W. S. M.

Nitrites; Sensitive reaction for —. P. H. Hermans. Pharm. Weekblad, 1920, 57, 462–463.

TO 2 c.c. of the solution to be examined a few drops of glacial acetic acid are added and then 2 c.c. of 5% potassium oxalate solution, 1 c.c. of 5% manganous sulphate solution, and a few drops of 3% hydrogen peroxide. A red coloration indicates nitrate (cf. 1919, 574 A; 1920, 283 A). 0.02 g. of sodium nitrite per l. can be detected in this way.

—W. S. M.

Potassium phosphate; Reactions between primary — and increasing quantities of calcium bicarbonate during boiling. W. Windisch and W. Dietrich. *Woch. Brau.*, 1920, 37, 177—180, 187—189.

If solutions of primary potassium phosphate are boiled with increasing proportions of calcium bicarbonate the resulting precipitates show increasing percentages of calcium corresponding to a transition from secondary to tertiary calcium phosphate, and the increase in alkalinity of the filtrate (due to formation of secondary potassium phosphate) becomes less rapid as the proportion of bicarbonate is increased. The alkalinities of the filtrates (found from surface tension measurements with eucupine dihydrochloride as indicator; *J.*, 1920, 461 A) are lower than the calculated values, the differences being ascribed to entrainment of soluble alkaline compounds with the precipitates.—L. E.

Chromic chloride solutions; Action of neutral chlorides upon —. M. E. Baldwin. *J. Amer. Leather Chem. Assoc.*, 1919, 14, 10—19.

THE hydron concentration of solutions containing the same amount of chromic chloride but varying amounts of sodium, potassium, ammonium, lithium, and barium chlorides, increases directly with the amount of added salt. The power of increasing the hydron concentration was in the following order of increasing effect of chlorides:—K, NH₄, Na, Li, Ba, which are also in their order of increasing power of forming hydrated ions at infinite dilution. This explains their action on the hydron concentration, since the hydration of the neutral solute ions diminishes the amount of water functioning as solvent. The increased hydron concentration is, therefore, not due to further hydrolysis of the chromic salt. (*Cf. J.*, 1920, 123A.) —D. W.

Rare earths; Separation of the — by basic precipitation. W. Prandtl and J. Rauchenberger. *Ber.*, 1920, 53, 843—853.

THE authors suggest a modified process for the basic separation of the rare earths, based on the reversible reaction, $MCl_3 + 3NH_4OH \rightleftharpoons M(OH)_3 + 3NH_4Cl$, which can be employed at any dilution and which depends mainly on delaying the precipitation by the addition of a sufficiency of ammonium chloride. The equilibrium has been investigated for lanthanum, neodymium, and praseodymium in the presence of ammonium chloride solution in concentration varying from *N*/1 to 5*N* and for temperatures ranging from 15° to 100° C. The results show that the precipitates are composed of strongly basic chlorides of varying composition, the solubility of which increases to a maximum with 3*N* ammonium chloride solution and then diminishes. The earth salt is absorbed from its solution by the precipitate to a marked degree. Lanthanum is relatively easily separated from didymium by reason of its greater basicity, but neodymium and praseodymium differ little in this respect. The temperature of the solution is an important factor in the separation of the earth metals. Lanthanum may be comparatively readily separated from neodymium and praseodymium by the following method even when the latter are present in considerable amount. The impure oxide is dissolved in moderately concentrated hydrochloric acid so as to form a nearly neutral solution, an amount of ammonium chloride equal to the weight of the oxide is added, followed by sufficient water to make the solution 2—3*N* with respect to ammonium chloride. The solution is heated to 50° C. and a mixture of equal volumes of 4*N*-ammonia and 4*N*-ammonium chloride solution is slowly added with constant shaking. As soon as a moderate amount of precipitate has formed (about 5% of the original oxide), the operation is interrupted, the precipi-

tate is removed, and a second precipitation is effected in the clear filtrate. Alternate precipitation and filtration are continued in this manner until the filtrate no longer exhibits absorption lines. (*Cf. J.C.S.*, July.)—H. W.

Radium-barium solution; Crystallisation of a —. C. E. Scholl. *J. Amer. Chem. Soc.*, 1920, 42, 889—896.

PURIFICATION of radium salts from barium salts is more efficiently carried out by crystallisation of the bromides than by crystallisation of the chlorides. A saturated solution of the bromides in 0.33*N* hydrobromic acid deposits 1/3 of the dissolved solid on cooling from 100° C. to 0° C. Crystallisation of such a solution gives a rapid concentration of the radium: thus if the concentration of radium in the starting material be represented by 1.0 the 9th crystallising dish to the right has a concentration 62 and the 9th to the left 0.016. In the fractionation the crystals of any crystallisation are moved 2 dishes to the right and the liquors 3 dishes to the left. (*Cf. J.C.S.*, July.) —J. F. S.

Red phosphorus as a reducing agent. L. Rosenstein. *J. Amer. Chem. Soc.*, 1920, 42, 883—888.

RED phosphorus when boiled in acid solutions of various salts exerts a strong reducing action. Gold and silver salts are converted into insoluble phosphides; mercurous or mercuric salts are reduced to the metal; palladium and osmium salts are reduced either to the metal or to the phosphide; stannic, ferric, and iridic salts are reduced respectively to stannous, ferrous, and iridous salts. Selenates are reduced either to the element or an insoluble phosphide, molybdates to quadrivalent molybdenum salts, vanadates to trivalent vanadium salts, bichromates to chromic salts, and permanganates to manganous salts. Bismuth, lead, cadmium, antimony, and arsenic salts, arsenates and stannous salts are not reduced, whilst tellurates and chloroplatinates are only slowly reduced.—J. F. S.

Hydrogen peroxide; Acceleration of decomposition of — by colloidal rhodium. C. Zenghelis and B. Papaconstantinos. *Comptes rend.*, 1920, 170, 1178—1180.

THE decomposition of hydrogen peroxide by a colloidal solution of rhodium is a unimolecular reaction, the velocity of which is considerably accelerated by previously bubbling either hydrogen or carbon monoxide through the colloidal solution. —W. G.

Graphitic carbon [and graphitic acid]; Problem of determining —. W. A. Selvig and W. C. Ratliff. *Trans. Amer. Electrochem. Soc.*, 1920, 629—658.

STAEDENMAIER'S method (*J.*, 1898, 880; 1899, 716) is inapplicable in presence of a large proportion of amorphous carbon as it leads to explosion. Anthracite and bituminous coal give no graphitic acid by Brodie's method (*Phil. Trans.*, 1859, 149, (1), 249), but coke yields a small amount. The conversion of graphite to graphitic acid by this method is assisted by treatment with potassium permanganate and sulphuric acid after the green stage is reached. The oxidation of the graphite takes place more readily with fine powders, as in the case of Acheson graphite. Moisture in graphitic acid cannot be estimated by ordinary methods, as the substance is extremely sensitive to varying humidity. Carbon and hydrogen in natural and artificial graphites may be determined by careful combustion after an admixture of a non-combustible material such as copper oxide; a preliminary treatment for removal of volatile matter and carbonates may be carried out. For determining graphitic carbon by conversion to graphitic acid

0.5 g. samples of Ceylon, Alabama, Pennsylvania, and Acheson graphites were treated by the Brodie and Staudenmaier methods. Mixtures of 0.5 g. of graphite and 0.5 g. of amorphous carbon were also treated by the Brodie method. Staudenmaier's method gave figures about 1.5–2.5 higher than those obtained by Brodie's method, and both these methods showed a lower percentage of carbon than was indicated by direct combustion of the graphites. These differences were greatest with Alabama and Pennsylvania samples. The gases evolved during treatment were found to consist chiefly of carbon dioxide, which is probably the final product of a series of oxidations which the graphite undergoes on treatment with the oxidising mixtures. The oxidation of carbon to graphitic acid evidently takes place within narrow limits of oxidising agent, temperature, and time. Loss of carbon as carbon dioxide is greater with Alabama and Pennsylvania graphites than with Ceylon and Acheson samples.—W. J. W.

Graphitic acid—a colloidal oxide of carbon. G. A. Hulett and O. A. Nelson. Trans. Amer. Electrochem. Soc., 1920, 425–439.

THE moisture content of graphitic acid, as of other colloids, cannot be determined by the usual methods. A method used for determining the true moisture of coals (Amer. J. Sci., 40, 175; cf. J., 1917, 378) was applied, in which the substance is heated *in vacuo* and the moisture condensed at -78°C ., the other gases being pumped off by a mercury pump. By plotting moisture-temperature curves and noting when decomposition becomes measurable, the moisture content can be found by interpolation. Considerable amounts of water can be removed from graphitic acid without decomposition, but at 181°C . there are indications of slow decomposition, and at 218°C . explosion takes place. The vapour pressure of graphitic acid, on removing successive quantities of water, was determined. The results obtained at 25° and 56.5°C . indicate that the vapour pressure continually decreases with removal of water without suggestion of a break. Graphitic acid is assumed to be a colloidal oxide of carbon, the flakes being made up of an infinite number of exceedingly thin plates covered with absorbed water. This explains the relation of water to the substance and the divergent properties of different varieties caused by variable size of the plates. Graphitic acid appears to have a formula of the order C_xO or C_xO_y .—W. J. W.

Carbonic acid and silicates. Matignon and Marchal. See VIII.

Detection of nitrates. Groenewege. See XXIII.

PATENTS.

Sulphuric acid manufacture; Heat interchangers for contact process of —. P. Audianne. E.P. 142,522, 7.12.18.

A HEAT interchanger for use in sulphuric acid manufacture comprises a vertical steel cylinder containing a set of tubes, arranged close together, and throttled at their lower end by a device which consists of a screw with three threads, or other similar arrangement for imparting a rotary movement to the gas. At the top and bottom of opposite sides of the cylinder are two openings, and in a continuation of the cylinder below is a third opening at right angles to the other two. The upper portion of the apparatus terminates in a truncated dome with internal concentric conical portions spaced so as to allow equal volumes of gas to pass between, and having an opening at the top communicating with the contact chamber. The cold sulphurous gases enter the lowest opening and

pass up through the tubes and the dome to the contact chamber, after leaving which they enter the tube container at its upper end and pass round the tubes to the lower exit. The dimensions and number of the tubes are dependent on the output of the sulphur trioxide plant, their total surface being 5 sq. m. per ton of gas passing through the apparatus in 24 hrs. for a temperature of 220°C ., and 25 sq. m. for a temperature of 325°C . The total cross-sectional area of the tubes should be substantially equal to that of the space outside. The diameter of the cylinder is selected to give a linear gas velocity greater than 3½ m. per sec.

—W. J. W.

Sulphuric acid concentration. J. Patten, Assr. to S. M. Shoemaker. U.S.P. 1,310,290, 18.5.20. Appl., 30.12.18.

IN connection with a series of concentrators (U.S.P. 1,291,827; J., 1919, 361A), two valves are introduced for the feed, and two receivers are provided, from either of which the acid may be transferred to the other.—W. J. W.

Hydrochloric acid; Apparatus for producing — utilising sludge acid [from treatment of mineral oils]. H. M. Lasher, Assr. to The Kansas City Refining Co. U.S.P. 1,339,519, 11.5.20. Appl., 7.10.16.

A GENERATOR for hydrogen chloride is connected to an absorber, consisting of a conduit with opposite and alternate baffles, which communicates with an absorption tower packed with filling material. Water is admitted at the top of the tower, and flows out at the base into a chamber, whence it is conveyed to the first absorption apparatus, the latter being provided with a receiving tank to receive the acid solution.—W. J. W.

Nitric acid; Manufacture of concentrated — from dilute nitric acid. E. Reinau. G.P. 305,171, 30.9.16. Addn. to 299,001 and 299,007 (J., 1920, 449A.)

AN alkali nitrate and sodium bisulphate, produced from dilute nitric acid as previously described, are allowed to interact, baking of the mixture and formation of pyrosulphate being prevented by conducting the process under 400 mm. vacuum at the start, and at an initial temperature of 90° , rising to 140°C .—W. J. W.

Electrolysis of metal salt solutions. Fredriksstad Elektrokemiske Fabr. A./S., F.E.F. E.P. 138,862, 31.12.19. Conv., 13.2.19.

IN an apparatus for the electrolysis of alkali chlorides a sloping layer of loose granular material of the substance to be electrolysed separates the anode and cathode compartments, and is supported on a wire cloth cathode and superposed asbestos cloth, arranged at such an inclination that the granular material is distributed over the support by the action of gravity. The granular soluble substance is supplied as required, to compensate for the quantity dissolved, through an opening along the upper edge of the layer, and a partition, the lower edge of which dips into the electrolyte, prevents the passage of gas into the charging opening, directing it into a gas-collecting chamber. A number of inclined anodes are placed in the space above the separating layer, with their lower ends resting on a stationary wall, several cells being so arranged that the bottom wall of one cell forms the inclined cover of the adjacent cell.—B. N.

Ammonia; Synthesis of — at very high pressures. L'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude. E.P. 142,150, 21.9.18. Conv., 20.2.18.

FOR the synthetic production of ammonia at pressures above 500 atm. or even 2000 atm. (E.P.

130,086—7; J., 1919, 718 A). use is made of a single catalytic apparatus instead of several. After circulating round a tube containing the catalyst and attaining the temperature of reaction from the tube itself during working and by a heating coil at starting the gases are caused to enter the tube. Successive series of pipes conduct the ammonia formed from the interior of the tube to collectors provided with cooled walls and having an outer insulated tube communicating with the interior of the catalyst tube by means of which the gases freed from ammonia are returned to another portion of the catalyst. In a modification of the device the reacting gases are heated by traversing a central tube through the catalyst after which they enter the head of the catalyst tube, the products being withdrawn as before.—W. J. W.

Ammonia; Synthetic production of —. H. C. Greenwood. U.S.P. 1,337,903, 20.4.20. Appl., 15.2.18.

In the production of ammonia from hydrogen and nitrogen one of the gases is passed over an alkali amide before passing the mixture of gases over the catalyst.—W. J. W.

[Calcium] cyanamide; Process and apparatus for manufacture of —. A. Duchemin. E.P. 142,354, 30.6.19.

FINELY powdered carbide is placed in a sheet-iron crucible, having a perforated base covered with a layer of asbestos or the like. The crucible is enclosed in brickwork, which serves as a heat regenerator and is itself surrounded by an insulating jacket, consisting of two sheet-iron plates with asbestos between. A lid operated by a pivoted lever covers the whole device. Nitrogen enters the furnace at the base, passing through the asbestos in the jacket to the top of the brickwork, through openings in which it then enters the crucible and passes downwards to an exit. Initial heating of the carbide is effected by introducing a receptacle containing a mixture of two substances which react on each other, such as an alkali or alkaline earth metal and an oxide or halide of another metal, the reaction being started by a suitable priming, percussion cap, or an electric current. The brick casing serves to maintain the heat necessary for the interaction of the carbide and nitrogen.—W. J. W.

Calcium cyanamide; Apparatus for improving the grade of —. G. E. Cox, Assr. to American Cyanamid Co. U.S.P. 1,331,742, 24.2.20. Appl., 20.8.18.

A NITRIFYING apparatus for the manufacture of calcium cyanamide consists of a combustible container adapted to hold finely divided calcium carbide, a hollow central core for the container, and a hinged outer form for holding the container.

—L. L. L.

Crude calcium cyanamide; Process for continuous manufacture of —. M. Novak. G.P. 305,061, 18.10.16.

THE furnace consists of a single reaction chamber in which, after admission of the nitrogen, the material is at once heated to the reaction temperature, and from which it is removed whilst hot immediately the process is completed. The material may first be deprived of free oxygen by keeping it for a short time in an atmosphere of nitrogen in another chamber immediately preceding the furnace.

—W. J. W.

Crude calcium cyanamide; Process for electrically heating the reacting materials in the manufacture of —. M. Novak. G.P. 305,532, 22.10.16. Addn. to 305,061 (preceding).

THE iron rails on which run trolleys carrying the charge may be employed for carrying the electric

current to the furnace, from which it is conveyed by conductors arranged along the furnace walls.

—W. J. W.

Crude calcium cyanamide; Process for decomposing —. Chem. Fabr. Kalk Ges. G.P. 301,321, 2.8.16.

IN the decomposition of crude calcium cyanamide by means of water or steam, in presence of carbon dioxide, at a high temperature, and at normal or increased pressure, ammonium salts may be added in sufficient quantity to interact with the free or combined lime, ammonia being evolved.—W. J. W.

Cyanamides [and nitrides] of the alkalis and alkaline earths; Process of decomposing —. Chem. Fabr. Kalk Ges. G.P. 302,461, 13.2.17. Addition to 301,321 (preceding).

IN the decomposition of cyanamides and nitrides of the alkalis and alkaline earths by water or steam, in presence of carbon dioxide or solid or liquid inactive materials, ammonium salts may be added either in the solid state or in solution. By this means appreciable quantities of alkali or alkaline-earth salts are obtained.—W. J. W.

Hydrate of alumina and similar gelatinous precipitates; Process for depositing — from their salts. M. Buchner, Assr. to The Chemical Foundation, Inc. U.S.P. 1,337,191, 20.4.20. Appl., 27.1.15. Renewed 26.6.19.

NON-COLLOIDAL hydroxides of aluminium, and of other metals which form colloidal hydroxides, may be prepared by dry interaction between a salt of the metal and an alkali.—W. J. W.

Metallic hydroxides; Manufacture of —. M. Buchner, Assr. to The Chemical Foundation, Inc. U.S.P. 1,337,192, 20.4.20. Appl., 26.8.15. Renewed 26.6.19.

METALLIC hydroxides, which may be easily filtered and washed, are formed by treating salts of the metals with a large excess of ammonia, the reaction liquor being used for treating a fresh quantity of salt, in presence of ammonia.—W. J. W.

Sulphur dioxide; Apparatus for producing —. H. K. Lyon. U.S.P. 1,337,561, 20.4.20. Appl., 17.7.19.

A FURNACE for obtaining sulphur dioxide from ores comprises a fire-box with an air inlet, means for heating the fire-box and for preheating the ore above the fire-box before introducing it into the box, and a heating chamber above, and communicating with, the fire-box but separate from the pre-heater.

—W. J. W.

Sal-ammoniac (ammonium chloride); Metamorphous —. C. Pfanstiehl, Assr. to Special Chemicals Co. U.S.P. 1,337,665, 20.4.20. Appl., 19.2.19.

PRESSURE is applied to crystalline ammonium chloride in order to obtain a solid mass having the appearance and fracture of marble.—W. J. W.

Ammonium sulphate; Preparation of — from calcium sulphate and gas-liquor. C. Otto und Co. G.P. 299,622, 24.3.16.

ANY ammonia not converted into sulphate by the calcium sulphate is expelled from the liquor after the reaction by passing hot or cold purified coal gas through it, and the gas is then added to the crude coal gas leaving the retorts, the ammonia combining with carbon dioxide present in the crude gas.

—A. B. S.

Ammonium compounds; Recovery of — from urine. H. Wichelhaus and J. Angerstein. G.P. 316,757, 2.10.18. Addition to 313,271.

AMMONIUM chloride, obtained by treatment of the end liquors from the production of potassium salts

with urine (J., 1919, 901A), may be separated in the form of a double salt with magnesium chloride. The liquor remaining after complete conversion of the magnesium chloride into the double salt may be run to waste with impunity.—W. J. W.

Alunite; Process of treating —. W. F. Downs. U.S.P. 1,338,428, 27.1.20. Appl., 14.12.17.

ALUNITE is treated with a dilute mineral acid, then mixed with an alkali oxide compound, and heated to about 500° C. The sulphates and other water-soluble compounds are removed by dissolving in water and filtering, the insoluble portion is mixed with sodium carbonate and heated to 950° C. to convert the alumina into sodium aluminate, which is subsequently recovered by treatment with water.

—W. J. W.

Asbestos; Method of and apparatus for treating —. O. H. Cilley, Assr. to C. G. Sargent's Sons Corp. U.S.P. 1,338,613, 27.1.20. Appl., 9.1.20.

THE lighter impurities in asbestos are removed by dry dusting, and the heavier non-fibrous impurities by washing. The water is then removed by draining and pressing, and the fibres are finally opened up and dried.—W. J. W.

Asbestos; Process of treating —. E. H. Garcin. U.S.P. 1,340,535, 18.5.20. Appl., 5.12.19.

ASBESTOS fibres are separated and strengthened by treatment with water and steam, followed by crushing between rollers, after which the asbestos is subjected to an acid bath, washed, and mechanically treated. Crushing and acid treatment may precede the steam and hot-water washing, the asbestos then being pressed, mechanically picked while wet, and finally dried; or the fibres may be softened in hot water, passed through crushing rolls, subjected to an acid bath, washed with steam and hot water, freed from water by squeezing through rollers, mechanically picked, and dried.—W. J. W.

Zinc oxide; Treating —. F. G. Breyer and E. H. Bunce, Assrs. to The New Jersey Zinc Co. U.S.P. Burdick. U.S.P. 1,339,494, 11.5.20. Appl., 29.1.19.

(A) Zinc oxide is caused to gravitate in a thin layer through a highly heated atmosphere. (B) Zinc oxide is caused to slide by the action of gravity and without mechanical agitation through a chamber arranged to offer a certain resistance through friction of the walls, the material being strongly heated during its passage.—W. J. W.

Oxides of nitrogen; Process of and apparatus for treating gas mixtures containing —. C. L. Burdick. U.S.P. 1,339,491, 11.5.20. Appl., 29.1.19.

A GASEOUS mixture containing one or more oxides of nitrogen is submitted to catalytic oxidation; the resulting nitrogen peroxide is absorbed in an aqueous medium and any mist entrained in the issuing gases removed by precipitation; the apparatus for these operations consists of a series of connected units each including a precipitator and a catalytic oxidation chamber.—D. F. T.

Sodium cyanide; Process of making —. A. Kaufman, Assr. to Air Reduction Co., Inc. U.S.P. 1,339,706, 11.5.20. Appl., 2.5.17.

IN the manufacture of cyanide by interaction of an alkali metal compound with carbon and nitrogen under the influence of heat, and alkali compound is previously intimately mixed with a carbon mass containing a small proportion of carbonised material of high porosity and absorptive capacity. By this means such fusing of the reaction product as would cause solidifying on cooling is avoided.—W. J. W.

Sulphur monochloride; Preparation of —. Chem. Fabr. Rhenania. G.P. 298,581, 11.12.14.

CHLORINE gas is passed upwards through a

series of vessels containing fragments of sulphur and the sulphur monochloride formed flows out to a receiver, any dissolved sulphur being converted into sulphur chloride by the incoming chlorine. Any sulphur chloride present in the gases leaving the last chamber of the series may be utilised for preparing vulcanised oil (rubber substitute) or the like.

—A. B. S.

Gases containing sulphur dioxide and hydrogen sulphide; Process for purifying —. J. Terwelp. G.P. 300,035, 14.2.17.

THE gases are passed through the carbonate, oxide, or hydroxide of an alkaline-earth metal or of magnesium to remove sulphur dioxide, and the hydrogen sulphide is then absorbed by alkali carbonate solution. The process is especially applicable to producer-gas.—A. R. P.

Alkali azide; Preparation of —. O. Matter. G.P. 302,561, 24.11.16.

NITROUS oxide, preferably under pressure, is passed over molten alkali amide which is vigorously stirred until it solidifies. The mass is then powdered and again heated in the gas until no further absorption takes place. The yield of sodium azide is over 90% of the theoretical. The reaction product, which contains some alkali hydroxide, is dissolved in water and the solution concentrated under reduced pressure, when the pure azide, which is only slightly soluble in strong caustic alkali solutions, is precipitated.—A. R. P.

Gases containing carbon dioxide; Utilisation of impure —. F. Riedel. G.P. 315,019, 25.11.17.

THE gases are treated with an alkali carbonate solution, and carbon dioxide is recovered by strongly heating the bicarbonate solution produced, so that steam under pressure is generated simultaneously. The carbon dioxide is suitable for use as a fertiliser (J., 1920, 125A), any injurious gases being dissolved by the condensed water deposited in the mains.—W. J. W.

Calcium sulphide; Manufacture of —. Deutscho Petroleum A.-G. G.P. 319,651, 7.8.18.

CALCIUM sulphate and methane are caused to interact at 800°–1300° C., the temperature, pressure, and velocity of the gas being controlled so as to effect complete reduction of the calcium sulphate without deposition of carbon.—W. J. W.

Ammoniacal brine; Process for carbonation of —. R. Schad. G.P. 318,180, 28.1.19.

IN the ammonia-soda process the gases containing carbon dioxide are passed through a series of vessels containing ammoniacal brine in such order that the final escaping gases are discharged from a vessel in which the whole or nearly the whole of the ammonia in solution has combined with carbon dioxide.

—W. J. W.

Liquid air; Apparatus for continuous rectification of —. E. Barbet et Fils et Cie. E.P. 142,519, 30.5.18. Conv., 9.10.17. Addition to 131,321 (J., 1919, 766A).

FOR the process described in the original patent the heat exchangers consist of bundles of tubes of great length and very small cross-section, enclosed in a shell terminating at each end in a chamber of large cross-section in which the ends of the tubes are spread out. The tubes may have small bands soldered on them at intervals to ensure equidistant spacing.—W. J. W.

Slaking lime; Apparatus for —. Spoerri & Co. E.P. 135,172, 27.10.19. Conv., 11.11.18.

Calcining lime etc. G.P. 314,584 and 317,832. See IX.

Marine alga. E.P. 123,325. See XVIII.

VIII.—GLASS; CERAMICS.

Glass resembling mother of pearl; Production of —. O. Schwarzbach. *Sprechsaal*, 1920, 53, 251—252.

VARIOUS recipes are given for the production of glass resembling mother of pearl. In each case they relate to the ground or body glass, which is repeatedly heated and cooled. Finely ground mica may be sprinkled on the hot body-glass as it is withdrawn from the melting pot, after which it is covered with a semi-crystal or crystal glass. The best opacifying medium is bone ash or cryolite, with or without zinc oxide. The cryolite may be replaced by felspar, kaolin, or fluorspar. The colour of opal glasses containing fluorides usually fades on repeated heating. The lustre may be intensified by reheating the finished glass in a reducing atmosphere.

—A. B. S.

Silicates and quartz; Prolonged action of carbonic acid on —. C. Matignon and Marchal. *Comptes rend.*, 1920, 170, 1184—1186.

CARBON dioxide under a pressure of 10 atm. has been allowed to act for ten years and three months on aqueous suspensions of quartz, wollastonite, diopside, mica, talc, asbestos, and glass respectively. At the end of that time all of the silicates and the quartz itself were more or less corroded, and varying amounts of silica had passed into solution. Wollastonite showed the greatest signs of attack, and glass the least.—W. G.

Clays; Classification of — on a ceramic basis. A. S. Watts. *J. Amer. Ceram. Soc.*, 1920, 3, 247—251.

A CLASSIFICATION based on five properties: colour, shrinkage, bonding strength, vitrification, and fusion, each property (except the colour) being divided into five groups ranging from "low" to "high," is suggested, the following limits being recommended: Colour: White, cream, buff, tan.

	Low.	Medium low.	Medium	Medium high.	High.
Shrinkage on drying	0-3%	3-6%	6-9%	9-12%	above 12%
Shrinkage total, cone 10	0-4%	4-8%	8-12%	12-16%	above 16%
Bonding strength, modulus of rupture, 50% clay—50% standard sand (lb.)	0-100	100-200	200-400	400-800	above 800
Vitrification (expressed in Orton cones)	below 5	5-15	15-25	25-30	above 30
Fusion (expressed in Orton cones) ..	below 5	5-15	15-25	25-30	above 30

—A. B. S.

Magnesia refractories. J. S. McDowell and R. M. Howe. *J. Amer. Ceram. Soc.*, 1920, 3, 185—246.

A COMPILATION of the chief information previously published on the occurrence, properties, and uses of magnesia as a refractory material with a selected bibliography. Magnesite bricks of good quality should have true sp. gr. 3.44—3.60, apparent sp. gr. 2.60—2.75, porosity 24—30%, crushing strength 5000—8600 lb. per sq. in., mean specific heat between 0° and 1300° C. 0.391, thermal conductivity 0.0085 at 1000° C., electrical resistivity 6000 ohms per cm.² at 1300° C., and average linear expansion 1.94% at 1200° C. under a pressure of 25 lb. per sq. in. Microscopic investigation shows that in the production of dead-burned magnesia any iron oxide present enters into solid solution with the magnesia crystals and forms magnesioferrite, but its chief value is its catalytic effect in hastening the forma-

tion of periclase. A small amount of silica appears to form a useful bond, consisting chiefly of forsterite, 2MgO.SiO₂. When silica and lime are both present they form with the magnesia mixtures of lower melting point than when only one of these oxides is present with the magnesia.—A. B. S.

[*Refractory materials; Measurement of physical properties [of —] at high temperatures.* A. G. Tarrant. *Faraday Soc.*, Dec., 1919. [Advance proof.]

To determine thermal expansion a cylindrical test piece, 4 in. long by $\frac{3}{8}$ in. diam., is enclosed at the bottom of a long tube of pure electric furnace graphite, which is heated in a vertical nichrome-wound electric furnace. On top of the test piece rests a plunger, which at the top of the tube is connected with a lever magnifying arrangement. Temperatures up to 900° C. are measured with a nichrome-constantan thermo-couple, and above this temperature with a Holborn-Kurlbaum optical pyrometer. Results obtained with this apparatus are concordant, but the method gives only the relative expansion of the test substance and the graphite sheath. An expansion of 0.0003 in. can be detected, and when a test-piece of pure silica is used the apparatus becomes a more sensitive temperature indicator than the thermo-couple. For measuring tensile strength at high temperatures test bars 30 in. long by $\frac{3}{8}$ in. diam. are used, with a semi-circular groove, $\frac{1}{4}$ in. deep, in the middle to localise the point of fracture. The test-bar is heated in a carbon tube furnace with a heating element consisting of a spiral cut from a solid piece of Acheson graphite. All the refractories tested were stronger at the high temperatures employed (up to 1800° C.) than in the normal "soft-burnt" condition. For testing samples in the air-dried or soft-burnt condition, a cement-testing machine is satisfactory. Attempts to measure accurately thermal conductivity at high temperatures did not give satisfactory results. For rough works routine tests, pieces in the form of shallow trays, $\frac{3}{8}$ in. thick at the bottom by 2½ in. dia., are used. A standard weight of lead is cast in each tray, and, when cold, they are placed side by side on a hot cast-iron slab, and the time taken for the lead to melt is taken as a measure of the thermal resistivity.—E. H. R.

Glass for ampullae. Ewe. *See* XX.

PATENTS.

Crucible furnaces, particularly such as are used in the treatment of glass. A. E. White. From *Fourts et Proc. Mathy Soc. Anon.* E.P. 143,117, 10.9.19.

A CRUCIBLE or glass-pot furnace, in which a crucible rests on a dished slab of refractory material from which any spilt glass is led away from the furnace through a pipe. The furnace is heated by surface combustion in a mass of porous refractory material beneath the dished slab.—A. B. S.

Glass furnaces. H. Wilkinson. E.P. 143,125, 10.11.19.

A GLASS-POT furnace having a grate beneath its eye has means for supplying solid fuel to the grate and a recuperator for preheating the secondary air before it enters the eye. It is claimed that this furnace can use an inferior coal and that it requires less fuel and less attention than either the old English or the Hermansen furnaces.—A. B. S.

Kiln for burning clay products, glazed ware and pottery. H. Webster. E.P. 133,123, 25.9.18.

A KILN with furnaces on opposite sides consists of a series of chambers connected by underground flues with a main flue and chimney. Adjacent chambers

are separated by a solid and a perforated wall respectively, the space between which forms a flue for conducting the gases from the main underground flue to the next or any subsequent chamber. Additional fuel may be supplied through feed-holes in the crown of each chamber. The chambers may be provided with muffles if desired.—A. B. S.

Refractory material. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 142,721, 19.8.19.

A REFRACTORY material, particularly suitable for lining electric furnaces operated at very high temperatures, consists of a mixture of 75–85% of fused, crystallised magnesia (sp. gr. 3.5–3.6) which has been reduced to particles of $\frac{1}{4}$ –1 in. diam., 20–10% of magnesia which has been calcined at about 700° C., and has sp. gr. 3.0–3.2, and 5% of a binder composed of magnesium chloride or other compound which forms magnesia when heated. The proportions are adjusted so that the shrinkage of the lightly burned magnesia and the binder counterbalances the slight expansion of the crystalline magnesia when the material is in use.—A. B. S.

Firebricks for furnaces, coke ovens, and the like. T. W. Keillar. E.P. 142,955, 28.2.19.

HOLLOW silica or fireclay bricks are rendered less heat-conducting by filling the cavities with lightly packed kieselguhr. The filling may be introduced through a hole which is subsequently plugged with fireclay, and the plug vitrified by means of a blast-lamp; or the filling may be inserted into the partly fired hollow brick and the cover sealed by a continuation of the firing.—A. B. S.

Silicon carbide; Formation of shaped objects of—. Gebr. Siemens und Co. G.P. 319,465, 23.2.17.

SILICON carbide is intimately mixed with a carbonaceous material, such as tar, and sand, the mixture then being moulded and heated to promote combination of the free carbon with the silicon in the sand. The process is adaptable for the manufacture of furnace bricks for use at high temperatures.—W. J. W.

Enamel; Method of cooling—. S. Wiester, Assignor to R. Weimer. U.S.P. 1,340,353, 18.5.20. Appl., 2.7.17.

A STREAM of molten enamel is subjected to the action of a jet of air moving with sufficient rapidity to separate the enamel into small particles and carry them a distance sufficient to cool them so that they will not adhere to each other.—A. B. S.

Wrought iron (sheet iron); Production of transparent and opaque enamels on — by means of layers of fine gold or silver foil. Fix und Schmidt. G.P. 319,207, 12.3.19.

SHEET iron is first given a preliminary coat of enamel, which is burnt on. An intermediate coating of powdered silver and an easily flowing flux mixed with a volatile oil is then applied, and, after drying, burnt on and polished. This serves as a carrier for the final layer of ordinary enamel on which the gold or silver foil is laid. The process is applicable to articles of jewellery and to the panelling of furniture.—A. R. P.

Furnaces for melting glass or other fusible substances; Oil-fired —. A. A. Mohr. E.P. 143,094, 11.7.19.

Enamels and enamelling. C. Musiol. E.P. 142,688, 6.6.19.

See U.S.P. 1,332,058 of 1920; J., 1920, 297 A.

IX.—BUILDING MATERIALS.

Concrete aggregate; Testing clays for —. D. H. Fuller. J. Amer. Ceram. Soc., 1920, 3, 256–258.

THE most feasible method of burning clay for the production of a light aggregate for concrete consists in firing it rapidly in a rotary kiln under strongly reducing conditions. To test the suitability of a clay for use in the preparation of such an aggregate, it is ground, pugged so as to develop maximum plasticity, and moulded into 2-in. cubes, which are dried and heated to 1375° C. in 360 mins., specimens being withdrawn from the kiln every half hour. Upon the appearance of marked fusion the firing is stopped, the kiln allowed to cool, and the specimens examined as to structure, and the apparent density determined. Products which fail to float in a salt solution of sp. gr. 1.1 or which consist of a shell of dense, vitrified clay enclosing large cavities are unsuitable for use as concrete aggregate.—A. B. S.

PATENTS.

Cement. D. W. Adamson. U.S.P. 1,338,117, 27.4.20. Appl., 25.7.19.

A CEMENT made of calcined, comminuted gypsum and calcined alunite.—A. B. S.

Fireproofing wood. H. F. Weiss, Assr. to C. F. Burgess Laboratories. U.S.P. 1,339,488, 11.5.20. Appl., 23.1.19.

WOOD is impregnated with an aqueous solution of a borate containing excess of alkali and then with a solution capable of reacting with the first solution with precipitation of an insoluble borate.
—D. F. T.

Calcining furnace for lime, dolomite, etc.; Annular —. E. Skuballa. G.P. 314,584, 31.8.17.

IN a furnace with a central gas producer, a single opening is provided in each chamber for admission of gas and air and for discharge of exhaust gas. A rotary device enables one or other of the three conducting pipes to be brought into communication with the opening. Air or gas may be supplied to any chamber as required by an arrangement of rotating valves.—W. J. W.

Lime, dolomite, magnesite, cement, etc.; Process and plant for calcining —. A. Heimsoth. G.P. 317,832, 8.6.17.

IN a furnace with two calcining shafts, and either one or two cooling shafts below the calcining shafts, the heating gases are preheated by passing them down through the hot calcined material in one calcining shaft and are then passed upward through the material in the other calcining shaft, together with air which has been preheated by passing it upward through the cooling shaft or shafts.—W. J. W.

X.—METALS; MÉTALLURGY, INCLUDING ELECTRO-MÉTALLURGY.

Case hardening; Industrial control of depth of —. J. Galibourg and M. Ballay. Rev. Mét., 1920, 17, 216–221.

TO estimate the depth of the case, a specimen after fracture is etched, without polishing, with an alcoholic acid copper chloride reagent. The core and case are attacked differently. The ferrite of the core is coated with copper, whilst the martensite of the case remains almost unaffected, giving a sharp line of demarcation between core and case, which is visible to the eye, so that the depth of the case can be estimated accurately to 0.1 mm.

—J. W. D.

Chrome-nickel steel for aeroplane shafts; Production of —. E. Kothny. Stahl u. Eisen, 1920, 40, 677—684. (Cf. J., 1920, 299a.)

A SERIES of experiments on the production under working conditions of an electro chrome-nickel steel (C 0.20, Mn 0.4—0.6, Si 0.2—0.3, Cr 1.0—1.5, Ni 4.0—4.5%) gave the following general results. In order to obtain in the finished steel the necessary fibrous structure only the purest materials must be used, and the bath must be worked with two oxidising slags. A long period of solidification either by cooling in a preheated iron mould or by casting in a sand mould is disadvantageous, resulting in the production of a steel with a well developed transverse structure on fracture in its worked condition. A manganese content of over 0.8% develops a granular structure in the treated steel.—J. W. D.

Ferro-molybdenum; Production of — at Orillia, Ont., and notes on molybdenum. B. C. Lamble. Trans. Canad. Min. Inst., 1919, 22, 61—67.

A FURNACE for the production of ferromolybdenum from molybdenum oxide or sulphide, consisted of a wrought iron jacket fitted on to a cast iron base, connected with one terminal of the electric supply. A cubical crucible, with a taphole arranged at the bottom, was built up inside the iron jacket by means of carbon blocks, the space between the walls of the crucible and the jacket being filled with magnesia bricks. The second electrode consisted of a 10 in. graphite rod held by a water-cooled head over the centre of the furnace. The power used was 4000 amp. at 55 volts. By direct reduction of a mixture of 70% of molybdenite concentrates and 30% of iron pyrites by coke in the presence of lime and silica as fluxes, 100 tons of ferro alloy assaying 71% Mo, 3.66% C, 0.08% S, 0.03% P, 1.35% Si, and 23.8% Fe has been produced, 80% of the total molybdenum in the charge being recovered in the alloy with a power consumption of 6—7 kw.-hrs. per lb. of alloy. The remainder of the molybdenum was recovered as dust or by wet dressing the slag.—A. R. P.

Aluminium alloys [duralumin]; Quenching of certain —. L. Guillet, J. Durand, and J. Galibourg. Rev. Mét., 1920, 17, 202—215.

A DETAILED account of an investigation of which the results have been published previously (J., 1919, 776a).

Nickel plating of aluminium and its alloys. L. Guillet and M. Gasnier. Comptes rend., 1920, 170, 1253—1256.

THE surface of the aluminium is prepared by subjecting it to a sand blast applied under a pressure of 1500 g. per sq. cm.; the sand used should pass through a 0.2 mm. sieve. Using an ordinary nickel plating bath, if the deposit of nickel does not exceed 0.01 mm. it is quite satisfactory, when tested by stamping, folding, or burnishing. If coarser sand or a lower pressure is used the deposit is not so satisfactory. If a thicker deposit is required it is better first to plate with nickel to a depth of 0.006 mm., then apply to this a deposit of copper, 0.02 mm. thick, and then a further coating of nickel.—W. G.

Zinc; Theoretical principles underlying the metallurgy of —. E. Jänecke. Metall u. Erz, 1919, 16, 247—251.

IF solid zinc oxide and carbon be added to molten zinc in a space from which the air has been exhausted, then at temperatures above the melting point of zinc an equilibrium will be established between the two solid phases and one liquid phase mentioned and a gaseous phase. It is a univariant equilibrium, since the four phases may be formed

from three independent components, and hence the pressure and composition of the gas will be constant at a definite temperature and will vary with the temperature. In applying the results of a theoretical discussion of the process to the technical process regard must be paid to the slowness of the reaction between two solid substances which, even at high temperatures, have only a small vapour pressure. By increasing the velocity of the reaction improvements in the smelting process might be effected.

Mercury; Determination of —. C. M. Bouton and L. H. Duschak. U.S. Bureau of Mines, Tech. Paper 227, 1920, 44pp.

ONE grm. of the finely powdered ore is mixed with 0.5 g. of dry calcium oxide and introduced into a glass tube (22 cm. long and not exceeding 10 mm. external diameter) in which has previously been placed a short layer of coarse sand. The ore is covered with a 1 cm. layer of lime, and then with coarse sand, and the whole kept in position by a brass wire gauze plug. The tube is heated in a furnace at 500° C., with the open end projecting about 8 to 12 cm., for 15 mins., and then placed, open end downwards, in 5 c.c. of hot nitric acid. If much sulphur is present cupric oxide is added to the lime (2:1), whilst if organic matter is also present 0.1—0.3 g. of powdered potassium chlorate is mixed with the coarse sand used for filling the end of the tube. The mercury dissolved by the nitric acid is oxidised by potassium permanganate, the excess of which is destroyed by hydrogen peroxide, and the solution (not exceeding 30 c.c.) treated with 2 c.c. of 10% ferric sulphate or nitrate solution (free from halides) and titrated with potassium thiocyanate solution. Mercury in solution is best determined by precipitation with hydrogen sulphide and treatment of the precipitate as described above.—C. A. M.

Tin; Electrolytic estimation of — in tetrachloride. Revision of the atomic weight of tin. G. P. Baxter and H. W. Starkweather. J. Amer. Chem. Soc., 1920, 42, 905—917.

THE atomic weight of tin has been determined by the electrolysis of solutions of carefully purified stannic chloride in 0.3N hydrochloric acid in a cell provided with a mercury cathode, the tin being weighed as tin amalgam. The value 118.703 is found as a mean of 16 experiments.—J. F. S.

PATENTS.

Steel; Refining of —. R. A. Hadfield. E.P. 142,885, 14.11.18.

A PARTLY refined normally finished basic carbon steel or a partly refined normally finished basic nickel-chromium steel is subjected to a melting and refining treatment in an electric furnace in the presence of two successive slags, one comprising lime and iron oxide, and the second composed of lime, fluorspar, and ferrosilicon.—J. W. D.

Iron-ore smelting furnace; Electric —. J. D. Shipton. U.S.P. 1,338,103, 27.4.20. Appl., 22.9.19.

THE furnace comprises a series of electrical smelting chambers disposed around a central mixing chamber, with means for delivering ore separately to each smelting chamber. The molten metal is tapped from each smelting chamber, delivered to the mixing chamber, and tapped from the latter.—B. N.

Iron and steel; Manufacture of —. H. Lang. U.S.P. 1,340,548, 18.5.20. Appl., 7.7.19.

FINELY ground iron ore and a reducing agent are gradually heated in closed iron containers to a reducing temperature and the containers are then

transferred to a melting furnace without bringing them into contact with an oxidising agent.

—J. W. D.

Iron and steel objects; Process and apparatus for coating — with white metal. K. Haas. G.P. 302,440, 2.12.14.

THE metal to be coated is cleaned by treatment with a reducing flux which also dissolves oxides, and then, in the absence of air, immediately brought in contact with the coating metal at a temperature above its melting point and that of the flux. Suitable fluxes are boric or phosphoric acids or their salts, or, for lead and tin, a mixture of lead or stannous chloride respectively, with zinc and ammonium chlorides.—A. R. P.

Iron articles; Process for producing a rust-proof coating of phosphate on —. H. Arnold. G.P. 305,677, 17.9.16.

THE articles are treated with a highly concentrated solution of phosphoric acid or phosphate of a definite degree of acidity, which may be produced by the addition of sulphuric or other acid to the solution.—A. R. P.

High-speed steel; Manufacture of —. M. Bormann. G.P. 310,042, 7.11.15. Conv., 19.4.15.

THE steel contains 7—12% Cr in place of the usual tungsten content, and 1—2% of one or more of the following: boron, molybdenum, vanadium, titanium, cobalt, tungsten. In order to obtain a good cutting edge, the steel is allowed to cool and then reheated one or more times during the forging. The heat treatment consists in heating to 50°—100° C. above the hardening temperature (700°—800° C.), quenching in water at 18° C., then hardening at the correct temperature.

—A. R. P.

Ore; Apparatus for desulphurising and treating —. B. Q. P. Foss, Assr. to The Foss International Ore Reduction Co. U.S.P. 1,338,292, 27.4.20. Appl., 22.7.14.

THE ore is quickly heated to a temperature sufficient to dry it and remove volatile constituents, and is maintained in motion during the heating. Refrigerated fluid is applied to the ore immediately after heating to reduce it to a friable state.—B. N.

Platinum substitute in electrical terminals, etc.; Composition of matter [alloy] for —. F. A. Fahrenwald, Assr. to The Rhotanium Co. U.S.P. 1,339,505, 11.5.20. Appl., 18.2.18.

THE alloy consists largely of gold and palladium having a melting point above 1150° C. and hardness at least twice that of gold.—B. M. V.

Metal alloy. G. C. Deeter. U.S.P. 1,340,437, 18.5.20. Appl., 18.6.19.

AS alloy containing as its principal constituents Pb 90, and As 5%.—J. W. D.

Alloy. T. Koch, Assr. to American Platinum Works. U.S.P. 1,340,451, 18.5.20. Appl., 24.8.18.

AS alloy containing not less than 75% Au, 0.25—5.0% Mn, and 24.75—3.3% Ni.—J. W. D.

Open-hearth furnace. F. B. McKune. U.S.P. 1,339,855, 11.5.20. Appl., 27.10.19.

THE furnace has a gas port at each end, and air is supplied through passages leading both direct to the gas ports and to the top of the furnace at each end, the latter passages being controlled by dampers.—B. M. V.

Electrolytic process. [Depositing copper from impure solutions.] W. E. Greenawalt. U.S.P. 1,340,826, 18.5.20. Appl., 19.3.15.

IN the electrolysis of impure copper solutions,

copper sulphide is precipitated by hydrogen sulphide and the copper sulphide applied to the electrolyte as a reducing agent.—B. N.

Basic slag with high phosphorus content; Method of obtaining —. B. Quehing. G.P. 318,147, 25.2.16. Addn. to G.P. 301,839.

A basic slag, rich in citrate-soluble phosphate, may be obtained by adding lime, mixed with oxides of iron or manganese, in the form of briquettes, to the furnace charge. The melting point of the briquettes is about 1010°—1030° C.—W. J. W.

Tin alloys; Electrolytic refining of —. Siemens und Halske A.-G. G.P. 319,384, 27.3.17.

THE formation of gelatinous stannic acid may be avoided by adding colloids such as tannin, indigo, and alizarin to the electrolyte. The addition of phosphoric acid to the electrolyte, and agitation with air, are advantageous.—W. J. W.

Furnaces for heating metal articles. Aktiebolaget Svenska Kullagerfabriken. E.P. 124,427, 4.3.19. Conv., 4.3.18.

Furnaces for heat treatment of metal articles [in an atmosphere of coal gas]. H. J. Yates and S. N. and E. R. Brayshaw. E.P. 142,671, 21.5.19.

Foundry cupolas [; Movable floors of —]. G. Green and E. A. Roper. E.P. 143,030, 14.5.19.

Amalgamator; Centrifugal — and method of obtaining metal from ore. S. G. Musser. E.P. 143,383, 9.5.19.

Steel alloy. Stahlwerke R. Lindenberg A.-G. E.P. 103,809—10, 16.8.16. Conv., 26.1.16.

SEE G.P. 309,175 and 312,311 of 1916; J., 1919, 908 A; 1920, 160 A.

Solder for aluminium and other metals. R. Iversen. E.P. 141,280, 6.10.19.

SEE U.S.P. 1,332,899 of 1920; J., 1920, 372 A.

Furnace for annealing, hardening, quenching, or like heat treatment of metals and the like. A. Smallwood. U.S.P. 1,339,424, 11.5.20. Appl., 31.7.18.

SEE E.P. 120,266 of 1917; J., 1918, 771 A.

Metallurgical process. A. Foss and B. F. Halvorsen, Assrs. to Norsk Hydro-Elektrisk Kvaestof-aktieselskab. U.S.P. 1,340,480, 18.5.20. Appl., 28.4.19.

SEE E.P. 126,951 of 1919; J., 1920, 238 A.

Separating devices. U.S.P. 1,339,682—4. See I.

Blast-furnace gas. E.P. 142,938. See HA.

Enamelling. G.P. 319,207. See VIII.

Electrodes for soldering. U.S.P. 1,339,515. See XI.

XI.—ELECTRO-CHEMISTRY.

PATENTS.

Electrolysing liquids; Apparatus for —. J. S. Withers. From National Electro-Products, Ltd. E.P. 143,082, 26.6.19.

A BI-POLAR electrode comprises a plate of non-conducting material having a gas chamber, with open bottom, formed at the top of each side of the plate, and a series of electrode members formed of wire located at each side of the plate below the chambers, and spaced from the plate by hollow vertical ribs provided with holes for the passage of electrolyte. The upper ends of the spaces communicate with the gas chambers. A sheath of porous material is fitted over the electrode embracing the opposite faces, whereby gas and electrolyte spaces are formed communicating with the chambers.—B. N.

Electric insulation. Elektro-Material G. m. b. H. E.P. 143,280, 12.7.18. Conv., 11.4.18.

THE insulating fabric is impregnated with a solution of a cellulose derivative such as cellulose acetate, nitrocellulose, cellulose formate, and the like. When employed for insulation purposes, the impregnated fabric is moistened with a solvent while in position, in order to effect adherence to the material to be insulated.—J. S. G. T.

Electrical accumulators. C. H. Worsnop. E.P. 143,327, 24.2.19.

PLATES for use in accumulators of the "alkaline" or iron and nickel type are produced by electrolytically depositing an alloy of iron, cadmium, tin, and titanium upon an iron plate for the negative electrode, and an alloy of nickel, cadmium, tin, and titanium upon a nickel plate for the positive electrode. At intervals the plates are removed from the electrolytic bath and dusted with graphite, then subjected to further treatment in the electrolytic bath until of the desired thickness. The plates may be used as produced or the deposited layers of alloy with intervening graphite layers may be stripped from their cores, and either bound together in a metal frame or pressed between sheets of metal gauze.—J. S. G. T.

Resistance element; Electrical-heater —. W. E. Gardner, Assr. to H. C. Black and J. Jacob. U.S.P. 1,338,294, 27.4.20. Appl., 7.7.19.

AN electrical resistance element consists of an intimate mixture of finely-divided carbon and bone ash.—B. N.

Electrical process and apparatus for compounding liquids with gases or other components and the products thereof, including carbonating beverages. J. E. Bloom. U.S.P. 1,338,352, 27.4.20. Appl., 30.9.19.

THE liquid components are separately electrified, with opposite signs and different potentials, and then mixed. The process is repeated with the mixed product and a third gaseous component, all the actions being carried out under insulated conditions.—B. N.

Electric furnace. J. Herlenius, Assr. to Hamilton and Hansell, Inc. U.S.P. 1,338,631, 27.4.20. Appl., 8.2.18.

AN electrode projects through an aperture in the side wall into the furnace, and is fixed to a pivotal support on the outside of the furnace shell. The electrode is surrounded by a flat packing ring substantially in the plane of the pivotal axis of the support, thus preventing leakage of air into the furnace chamber through the opening in the side wall.—B. N.

Electric furnace. J. Hughes; E. W. Hughes, admtrix. U.S.P. 1,339,393, 11.5.20. Appl., 11.10.19.

THE metal shell of the furnace is provided with an inner refractory lining, the outer surface of which has an offset so that a gap is formed between the inner surface of the shell and the outer face of the refractory lining. A foraminous filler between the shell and refractory lining prevents warping and distortion of the lining and shell, whilst closing the gap without preventing circulation of a cooling medium through it.—B. N.

Electric furnace; Method of operating an —. M. R. Trembour, Assr. to Ludlum Electric Furnace Corp. U.S.P. 1,339,428, 11.5.20. Appl., 24.3.16.

AN electric current is passed through the charge

between an electrode adjusted to form an arc with the charge and an electrode of carbonaceous material adjusted to contact with the charge, the electric current being used both to supply heat at the arcing electrode as well as to prevent carbonisation of the charge and dissolution of the contacting electrode.—A. G.

Electric furnace; Process of controlling the thickness of walls in an —. C. H. Chapman, Assr. to General Abrasive Co., Inc. U.S.P. 1,340,155, 18.5.20. Appl., 20.2.17.

THE furnace walls are constructed by first melting and then cooling a portion of the charge, whereby the cooled portion forms a solid, continuous, homogeneous, crystalline, heat-resisting wall about the central portion of the furnace, providing a receptacle for the molten mass.—B. N.

Electrodes for soldering and depositing metals by the electric arc. E. H. Jones. U.S.P. 1,339,515, 11.5.20. Appl., 18.2.20.

AN electrode is formed by moistening a rod coated with silica, and applying a slag-forming powder to the moist silica.—B. N.

Electrodes for soldering and depositing metals by the electric arc. E. H. Jones, and Alloy Welding Processes, Ltd. E.P. 142,934, 14.2.19.

SEE U.S.P. 1,339,515 of 1920; preceding. Sodium silicate or other silicious compound is used for coating the rod.

Electric furnaces [; Raising and lowering electrodes of tilting —]. W. E. Moore. E.P. 143,176, 14.2.19.

Electric furnaces [; Mounting of electrode arms in tilting —]. W. E. Moore. E.P. 143,292, 14.2.19.

Furnace; Electric — [for heat treatment]. T. F. Baily and F. T. Cope, Assrs. to The Electric Furnace Co. U.S.P. 1,340,203, 18.5.20. Appl., 12.8.18.

Electric furnaces. E. F. Collins. E.P. 133,026, 6.8.19. Conv., 21.9.18.

SEE U.S.P. 1,310,060 of 1919; J., 1919, 644A.

Electrolysing liquids; Apparatus for —. J. S. Withers. From National Electro-Products, Ltd. E.P. 143,002, 23.4.19.

SEE U.S.P. 1,324,511 of 1919; J., 1920, 120A.

See also pages (A) 476, *Electrical precipitators* (U.S.P. 1,337,488-9, 1,339,471, and 1,339,480, and G.P. 315,931); *Electro-osmosis* (G.P. 316,443). 480, *Carbon electrodes* (G.P. 317,179). 484, *Electrical dyeing* (U.S.P. 1,338,353). 487, *Electrolysis* (E.P. 138,862). 488, *Calcium cyanamide* (G.P. 305,532). 493, *Platinum substitute* (U.S.P. 1,339,505).

XII.—FATS; OILS; WAXES.

Salmon oil. H. A. Gardner. Circ. 92, Paint Manufacturers' Assoc., U.S.A. May, 1920.

OIL from the king salmon had the following characters: Sp. gr. 0.9268; $n_D^{25} = 1.4788$; acid value, 9.8; saponif. value, 183; and iodine value, 159. The raw oil was tacky after 3 days' exposure, but with 10% drier gave a fairly firm film. Oil from the silver salmon had: Sp. gr. 0.9183; $n_D = 1.4753$; solidif. pt. -3° to -11° C.; saponif. value, 193.3; acetyl value, 17.83; insol. fatty acids, 92.59%; unsaponif. matter, 1.69%; iodine value, 150.6; solidif. pt. of fatty acids, $29-2^\circ$ C.; and hexabromide 45.03%. Mixed with a suitable drier it dries well in 18 hrs., especially when blown.

—C. A. M.

Channel cat fish oil. H. A. Gardner. Circ. 93, Paint Manufacturers' Assoc., U.S.A. May, 1920.

A SAMPLE of channel cat fish oil had: Sp. gr. 0.9231; $n_D^{20}=1.4741$; acid value, 10.9; saponif. value, 192; and iodine value, 123. It is unsuitable for paints or varnishes in the raw state, but dries better after heat treatment.—C. A. M.

Ceryl alcohol and cerotic acid of Chinese wax. A. Gascard. Comptes rend., 1920, 170, 1326—1328.

CHINESE wax consists for the most part of ceryl cerotate. Brodie's formulae for ceryl alcohol $C_{21}H_{43}OH$ and for cerotic acid $C_{26}H_{53}CO_2H$ are confirmed.—W. G.

Boiling oils. Gardner and Parks. See XIII.

PATENTS.

Catalyst [for hydrogenation of oils]; Production of —. W. P. Schuck. E.P. 142,576, 18.2.19.

A SOLUTION, containing 0.1716 g. of nickel nitrate and 0.25 g. of sugar per c.c., is passed in a state of suspension in an inert gas through a muffle, heated to 550°—650° C. The water is vaporised and the nickel nitrate is decomposed and forms a "black, flaky, non-oxidisable powder" which is removed from the muffle while still in a state of suspension in the gas. The product contains the catalytic metal but no free carbon (cf. U.S.P. 1,305,173; J., 1919, 589A).—W. H. C.

Fish oil; Process for removing the odour from —. U. Ioka. U.S.P. 1,340,050, 11.5.20. Appl., 10.2.19.

THE oil is mixed with cypress and cedar sawdusts and green cedar leaves, the mixture heated, cooled, and filtered, and the filtered oil exposed to sunlight.—C. A. M.

Bile; Purification of — [for use as detergent]. K. Horkenbach. G.P. 317,721, 14.9.17.

THE bile, after standing to separate mucilaginous substances, is subjected to osmosis either after treatment with acids and alkalis, or concomitantly therewith. Electro-osmosis may, if desired, be substituted for simple dialysis.—G. F. M.

XIII.—PAINTS; PIGMENTS; VARNISHES ; RESINS.

Paint: a plastic material and not a viscous liquid: the measurement of its mobility and yield value. E. C. Bingham and H. Green. Proc. Amer. Soc. for Testing Materials, 1919, 19, 640—661.

Viscous liquids will start to flow no matter how small a pressure be applied, whereas with plastic materials no flow takes place until the pressure has exceeded a certain definite value to which the name "yield value" is given. When pressures are plotted as abscissæ and volumes of flow per unit time as ordinates, the curve of a true liquid is linear and passes through the origin, whilst in plastic solids the "yield value" appears as the intercept of the linear curve on the pressure axis. For plastic solids, Poiseuille's equation requires modification to $\mu = KV/(P-f)$, where μ is the mobility (corresponding to the fluidity of liquids), V is the volume of flow in unit time, P is the pressure, and f is the yield value. Paint behaves as a plastic solid in that it will maintain a vertical surface provided the weight is not too great to exceed the yield value. A "plastometer" has been devised which permits of the determination of flow of a plastic solid through a capillary in unit time with varying pressure, whereby both its mobility and its yield value can be measured. The apparatus, for the mechanical details of which the

original paper must be consulted, comprises a pressure tank, a pressure regulator and stabiliser, a manometer, and a container for holding the paint under examination. This container is a form of viscometer of the efflux type wherein the whole unit together with the receiver may be kept at constant temperature. The volume of flow is determined by reference to the density of the paint and the weight of the drops, the latter being constant at a constant rate of falling from the end of the capillary. Readings obtained from the examination of three typical paints in the plastometer are as follows:—

Pigment base of paint.	μ .	f.
Zinc oxide	0.204	87
Leaded zinc	0.373	320
White lead	0.171	117

The "thinning down" of paints on storage is stated to be a function not of change of mobility but of fall in yield value.—A. do W.

Oils; Fume loss in boiling —. H. A. Gardner and H. Parks. Circ. 95, Paint Manufacturers' Assoc., U.S.A. May, 1920.

THE losses on heating linseed, boiled linseed, menhaden, soya bean, perilla, and sunflower oils for 2—4 hrs. at 500° F. (260° C.) and 550° F. (about 290° C.) in a small open metal pan are given in tables together with the corresponding sp. gr. Linseed oil lost 2% after 2 hrs. and 2.9% after 4 hrs. at 500° F., and 3.9% after 2 hrs. and 8.6% after 4 hrs. at 550° F.—C. A. M.

Salmon oil. Channel cat fish oil. Gardner. See XII.

PATENT.

Anti-fouling paint for ships' bottoms and process for the manufacture of same. E. Ariò. E.P. 131,601, 21.8.19. Conv., 23.5.14.

THE effectiveness of anti-fouling paints containing inorganic or organic poisons is increased by incorporating in the paint fatty acids (or the corresponding sodium salts) of the series $C_nH_{2n}O$, ranging from 4 to 14 atoms of carbon.—D. F. T.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Rubber compounds. N. D. Nielsen. E.P. 127,821, 2.6.19. Conv., 31.5.18.

RUBBER is mixed with a suitable quantity of tough, durable, flexible and resilient organic matter in the form of thin rough flakes, such as fish scales, whereby the rubber material is greatly improved in durability and toughness.—D. F. T.

Rubber; Utilisation of bark shavings from rubber trees, and extraction of — from such bark shavings. R. T. Smith. From A. H. Claessen. E.P. 142,946, 20.2.19.

THE bark shavings are ground and masticated between heated rollers, preferably at a temperature near 275° F. (135° C.), so as to convert the whole into a dough-like mass. This may then be mixed with a vulcanising agent and, after shaping or moulding, vulcanised. Alternatively the mass may be passed through a scrap-washer and the separated rubber crépé; a larger yield of rubber is then obtained than is possible by the customary procedure.—D. F. T.

Rubber waste; Apparatus for reclaiming —. F. L. Kryder and E. W. Snyder. U.S.P. 1,310,776, 18.5.20. Appl., 8.3.18.

The reclaiming chamber consists of a rotary steam-

jacketed drum with horizontal axis; the axles at each end are tubular, one permitting the introduction of steam into the jacket whilst through the other there passes an inlet pipe reaching to the interior of the chamber and serving to introduce a solution for treatment of the rubber; the solution is forced in under pressure by means of a pump, thus increasing the pressure within the reclaiming chamber without substantially raising its temperature.—D. F. T.

Rubber waste; Process of reclaiming and regenerating —. F. L. Kryder. U.S.P. 1,340,777, 18.5.20. Appl., 18.8.19.

RUBBER waste is disintegrated, heated with an aqueous solution containing 10–15% of sodium hydroxide for 6–20 hrs. at a pressure between 50 and 200 lb. per sq. in., and then subjected to the action of a solvent comprising phenylenediamine (1 pt.), paraffin oil (2 pts.), mineral rubber (2 pts.), rubber resin (1 pt.), and acetic acid (2 pts.). The resulting mass is washed and dried.—D. F. T.

Waterproofing fabrics. E.P. 129,630. See V.

Artificial leather. U.S.P. 1,337,802. See XV.

XV.—LEATHER; BONE; HORN; GLUE.

Tanning materials; The "shake" method and the "filter bell" method in the analysis of — with hide powder. G. Baldracco and S. Camilla. J. Soc. Leather Trades' Chem., 1919, 3, 110–123.

THREE sensitive tests for gallic acid and protocatechuic acid in the non-tan solution obtained in the analysis of tanning materials are given. On adding three drops of a 0.5% solution of ferric chloride to the non-tan solution, gallic acid gives a blue and protocatechuic acid a green coloration. On adding three drops of a 2% solution of ammoniacal nickel sulphate to the non-tan solution, shaking, boiling, and adding excess of ammonia, gallic acid gives a red coloration at first, turning to brown, and protocatechuic acid a reddish-brown. On adding three drops of 4% solution of formaldehyde to the non-tan solution and then a few drops of concentrated sulphuric acid, gallic acid gives a reddish colour, and a rose-coloured precipitate forms after standing for 1 hr.; protocatechuic acid gives a violet ring surmounted by a milky layer. On submitting the non-tans from the "filter bell" method of analysis to these tests negative results were always obtained, thus proving that gallic and protocatechuic acids, though non-tanning substances, have been completely absorbed by the hide powder. Positive results are always obtained on testing the non-tans from the "shake" method of analysis. Greater efficiency is claimed for a modified "shake" method (J., 1920, 123A).

—D. W.

Tannin; Titration and estimation of — with iodine. F. T. Lee. J. Soc. Leather Trades' Chem., 1919, 3, 8–10.

FIVE c.c. of filtered tannin solution, containing not more than 1 g. of pure tannin per l., is oxidised with 25 c.c. of N/50 iodine solution for 20 mins. in a stoppered bottle, and the excess of iodine titrated with N/50 thiosulphate, using starch as an internal indicator. Six c.c. of the non-tannin solution obtained in the official S.L.T.C. method of tannin analysis is similarly titrated to determine the iodine absorbed by the non-tannins. The iodine solution must be standardised against 5 c.c. of a 0.1% solution of pure gallic acid to obtain the gallic acid equivalent in terms of which the other titrations are all calculated and expressed as outlined by Procter ("Leather Chemists' Pocket

Book," p. 107). The method is designed for comparative control purposes.—D. W.

Mangrove [tannin]; Detection of — in mixed tannin extracts. E. Schell. J. Soc. Leather Trades' Chem., 1918, 2, 284–286.

A REVIEW of the several methods suggested for the detection of mangrove tannin in tanning extracts. The solubility of mangrove tannin in ethyl acetate is a very uncertain guide. The amount of chlorine or lime in the ash is also a doubtful indication of the presence of mangrove, since these constituents may have been introduced by using hard water. The Mulhouse dyeing test with mordanted cotton is unreliable. The precipitates obtained in the Schell test, in which a cobalt solution is added to a solution of the tanning extract, are not sufficiently well defined in colour to render it an efficient test. It can only be employed with normal extracts, and fails with solubilised extracts, and with those which have been subjected to a treatment with alkaline compounds and which give an alkaline reaction in solution.—D. W.

Tannins of galls, sumach, myrobalans, divi-divi, and quebracho; Relative affinity for cotton and mordanting value of the —. R. H. Wisdom. J. Amer. Leather Chem. Assoc., 1919, 14, 6–9.

THE affinity of tanning materials for cotton does not bear any simple relation to their tannin content. The percentage of total solid matter absorbed by the cotton was determined for each material, and is termed the "absorption value." The mordanted cotton was washed, passed through an iron solution, incinerated, and the amount of iron fixed was determined. The percentage of iron fixed by each part of absorbed matter was multiplied by the "absorption value" and yielded the following "mordant values": Galls 135, sumach 118, myrobalans 98, divi-divi 80, quebracho 66. The actual colour depths on cotton agreed with these figures. This method is suggested for comparing the relative mordanting values of these and other materials in dyeing leather, since galls and sumach are the best mordants for leather, and myrobalans is the next in order.—D. W.

Degree of tannage in different stages of process. O. Riethof. J. Amer. Leather Chem. Assoc., 1919, 14, 20–22.

SAMPLES of leather cut from the backbone of the hide about two feet from the tail during various stages of the sole leather and belting leather tanning processes were dried and analysed. The leather was found to be half tanned in 27 days, and three-fourths to four-fifths tanned in half the time necessary for completion of the tannage (100–108 days). The assumption that the stock in process is, on the average, only half tanned is thus proved to be a serious under-estimate.—D. W.

Chrome [tanning] liquors; Statement of basicity of —. J. R. Blockey. J. Soc. Leather Trades' Chem., 1919, 3, 11–13.

It is suggested that the basicity should be stated as the number of hydroxyl groups attached to two atoms of chromium. The variation would then be between 0 and 6. Such an expression of the basicity is determined by the formula:—

$$6 - \left(\frac{152}{\text{Amount of Cr}_2\text{O}_3} \times \frac{\text{c.c. N/1 NaOH}}{1000} \right)$$

—D. W.

Chrome leather problems for research laboratories. E. J. White. J. Amer. Leather Chem. Assoc., 1919, 14, 2–5.

IN the neutralisation of chrome-tanned leather the absorption of the alkali may be due to neutralisation of the acid introduced from the tanning baths,

to combination of alkali with the hide substance, and to adsorption. The amount of alkali used in neutralising acidity could be ascertained by determining the sodium salts (thiosulphate and sulphate) in the residual liquors. In the initial stage of neutralisation the proportion is about 75% thiosulphate and 25% sulphate, indicating a tannage by 3 pts. of basic chromium thiosulphate and 1 pt. of basic chromium sulphate. Other problems on tanning include a study of the boiling test before and after neutralisation, and the presence or absence of acid in the essential tanning agent. The dyeing and fat liquoring of chrome-tanned leathers can be studied by the analysis of the residual liquors for sulphate and thiosulphate. The absorption of chromic acid from various mixtures by the skin, and the preferential absorption by the skin of different acids, both require further study.

—D. W.

Animal hides; Coloration of — by means of o-vanillin and o-protocatechuic aldehyde and tanning with aldehyde. O. Gerngross. *Z. angew. Chem.*, 1920, 33, 136—138.

DECOMPOSITION products of proteins (glycylglycine ester and tyrosine ester) give the same colorations with Schiff's o-protocatechuic aldehyde base as those produced in hides by o-protocatechuic aldehyde and o-vanillin. Formaldehyde-tanned leather does not give the same colorations as ordinary leather with these reagents, but reacts like ordinary leather with a solution of sodium diazobenzene-sulphonate, giving a blood-red coloration. This indicates that the imino group of the iminazole ring of histidine in formaldehyde-tanned leather has not been replaced by formaldehyde, and that no envelopment of the fibres by polymeric compounds of formaldehyde has occurred. The tanning properties of o-protocatechuic aldehyde are due not to an aldehyde, but to a dihydroxy group.

—C. A. M.

Gelatin; Kjeldahl's method for the determination of nitrogen as applied to —. H. G. Bennett and N. L. Holmes. *J. Soc. Leather Trades' Chem.*, 1919, 3, 24—27.

The digestion of gelatin in the Kjeldahl method differs from that of leather in the small amount of carbonaceous matter to be oxidised. The clearing of the liquor is no criterion of complete hydrolysis. Experiments with and without accelerators and with various periods of digestion ranging from 1 to 6 hrs. showed that sulphuric acid alone does not give correct results. The most reliable figures are obtained by heating 0.25 g. of gelatin, cut into small pieces, in a 250 c.c. flask with 10 c.c. of concentrated sulphuric acid until it is completely dissolved. The liquid is cooled, and 10 g. of pure anhydrous potassium sulphate is added, and the boiling continued for 4—6 hours, after which the ammonia formed is distilled.—D. W.

Chromic chloride solutions. Baldwin. See VII.

PATENTS.

Artificial leather. A. A. Somerville, Assr. to New York Belting and Packing Co. U.S.P. 1,337,802, 20.4.20. Appl., 15.1.18.

A VULCANISED rubber layer and a sheet of paper are wound up into a roll, subjected to the action of a vulcanising medium, and the sheet of paper removed. The material has the appearance, texture, flexibility, and general characteristics of dull finished leather.—D. W.

Artificial leather and similar products; Manufacture of —. E. Reidel. G.P. 302,445, 8.5.15.

DRIED animal intestines, sinews, and the like are plumped, e.g., with acetic acid or sodium sulphide, washed, compressed between rollers, dried, and

saturated with a solution of albumin and a marine animal oil. The product, after compression to sheets, is dried and tanned by the combined mineral and vegetable process.—L. A. C.

Tanning [; Degreasing hides and skins preparatory to—]. Degreasing apparatus. C. C. Krouse. U.S.P. (A) 1,338,307 and (B) 1,338,308, 27.4.20. Appl., 20.6.19 and 27.1.20.

(A) Raw hides and skins containing a high percentage of fat are treated before tanning with sufficient grease solvent to dissolve the grease, which is carried away on removal of the solvent. (B) The apparatus comprises a closed tank having a closure at one side. In the tank are parallel, hollow, spaced elements to which heating fluid is supplied, guides between the heating elements, and reticulated frames to which the hides and skins are clamped. Means for the supply and withdrawal of liquid and for producing a vacuum are connected to the tank.

—D. W.

Shark-skins and the like; Treating — preparatory to tanning. A. Rogers, Assr. to the Ocean Leather Co. U.S.P. 1,338,531, 27.4.20. Appl., 8.5.19.

THE dermal armouring of shark-skins and the like is removed by subjecting them to the action of heated fluid. The epithelium layer from the flayed skins is removed with a mixture of 5 parts of lime and 1 part of sodium sulphide.—D. W.

Glue; Manufacture of water-resistant —. H. Grünwald. G.P. 317,963, 9.7.18.

ANIMAL glue is treated with a product obtained by the interaction of albumin and formaldehyde. When the glue is used the action of heat and pressure causes the liberation of formaldehyde, which acts on the glue and renders it insoluble.—W. J. W.

Mocha leather; Manufacture of —. L. Forse, Assr. to S. T. Thring and R. J. Luffman. U.S.P. 1,339,751, 11.5.20. Appl., 29.12.16.

SEE E.P. 102,986 of 1916; J., 1917, 298.

Chrome leather; Detanning —. M. C. Lamb. U.S.P. 1,340,721, 18.5.20. Appl., 20.6.19.

SEE E.P. 132,864 of 1918; J., 1919, 872 A.

XVI.—SOILS; FERTILISERS.

Calcium sulphate; Effect of — on the solubility of soils. M. M. McCool and C. E. Millar. *J. Agric. Res.*, 1920, 19, 47—54.

FROM field observations in Michigan it was considered that calcium sulphate was a soil stimulant. Laboratory experiments were made with sand and loam soils, which were allowed to stand in contact with excess of calcium sulphate solution for 24 hours, and then dried, transferred to containers, and made up to a low and a high water content. The samples were examined every few days for a month by the freezing-point method for the formation of soluble salts. The treatment was found to have increased the solubility of the soil considerably. When the soils were well washed after the calcium sulphate treatment the increase in solubility was even greater. Thus the calcium sulphate had effected some change in the soils, leading to the formation of soluble compounds. Calcium phosphate alone decreased the solubility of the soil, and in combination with calcium sulphate neutralised much of the effect of the latter. The carbon dioxide formed by the soils during the experiments was rather less than in untreated soil, so that the action of calcium sulphate was not due to any bacterial activity.—J. H. J.

Germination of seeds and growth of seedlings; Effect of reaction of solution on —. R. M. Salter and T. C. McIlvaine. *J. Agric. Res.*, 1920, 19, 73—95.

SEEDS of wheat, maize, soya bean, alfalfa (lucerne), and clover were germinated and grown in culture solutions of uniform composition, but with varying hydrogen-ion concentration. It was found best to adjust the reaction by adding a constant amount of phosphoric acid to all the solutions, followed by progressively increasing amounts of caustic soda. The optimum reaction for germination was found to be between $p_H=7.7$ and $p_H=3$, a slightly acid solution being most favourable. The maximum growth of the seedlings occurred between $p_H=5.16$ and $p_H=5.94$. The wheat seedlings tended to adjust the reaction during growth to a point just below neutrality.—J. H. J.

Calcium cyanamide; Physiological action of crude —. Siebner. *Chem.-Zeit.*, 1920, 44, 369—370, 382—383.

A DETAILED review of the literature on the physiological action of crude calcium cyanamide is given. Its injurious action, which is confined chiefly to the skin and to the respiratory organs, is not greater than that of other dusty substances, and can be prevented by the avoidance of dust. Crude calcium cyanamide cannot be called a poison in the ordinary sense of the word, since as much as 40—50 g. has to be taken to produce any poisonous effect. It does, however, produce certain cases of sudden illness, which have been found always to be associated with the consumption of alcohol. Workers with the substance, who were exposed to the inhalation of dust, have been accustomed to partake of alcoholic beverages to remove the irritation, and it is this combination which has brought about "cyanamide disease." Farmers who always work with the substance in the open air are not subject to illness. It is suggested that illness would be prevented in works by the use of drinks free from alcohol, and that even on the land it would be advisable that the substance should be applied with the wind and not against it.—J. H. J.

PATENTS.

Mineral-manure process. W. McA. Johnson. U.S.P. 1,338,734, 4.5.20. Appl., 21.6.18. Renewed 6.2.20.

A MOLTEN slag, containing a potassium compound in solution, is blown into a fine state of subdivision.—W. J. W.

Fertiliser, and manufacture of same. M. Fjellanger, Assr. to Norsk Hydro-Elektrisk Kvaelfstofaktieselskab. U.S.P. 1,340,708, 18.5.20. Appl., 6.2.20.

A FERTILISER consisting of urea nitrate and calcium carbonate.—W. J. W.

Crude calcium cyanamide; Process for decomposing —mixed with a liquid, by means of carbon dioxide. G. Zarniko. G.P. 300,632, 6.7.16.

THE process of incorporating and disintegrating crude calcium cyanamide with a liquid and its treatment with gases may be carried out in a mill with free-moving grinding bodies, or in a drum mill provided with teeth. The operation may be made continuous by introducing the calcium cyanamide and liquid at one end and removing the reaction product containing urea at the other.

—W. J. W.

Superphosphates; Process of manufacturing —. O. Rosenthal. G.P. 315,393, 11.8.14.

DRY phosphorus pentoxide is mixed with dry crude phosphate and sufficient water added to convert the pentoxide into phosphoric acid. The mixture may

be first treated with part of the water only, the remainder being added later. The phosphorus pentoxide may be used direct from the process of manufacture whilst still hot. The reaction is assisted by the heat liberated by the combination of the water with the pentoxide, and is initiated at 100° C.—W. J. W.

Furnace for the manufacture of a germ-free fertiliser from faeces and other organic waste products. Stettiner Chamotte-Fabr. A.-G. vorm. Didier. G.P. 316,148, 13.6.17.

A FURNACE grato is supported on a vertical partition wall between the drying and the sterilising chambers, the length of the grate being slightly less than the length of the chambers, thus leaving an intermediate space. An overhanging arch is erected above the grate, which causes the furnace gases to pass first over the surface and then underneath the grate.—W. J. W.

Boiling and drying organic materials, especially slaughterhouse waste; Process of —. K. Niessen. G.P. (A) 318,542, 12.5.15, (B) 319,335, 21.7.16, and (C) 319,336, 13.5.17. Addns. to G.P. 317,818 (J.), 1920, 379 A).

(A) IN vessels having a single steam-jacket an air-pipe passes through the jacket into the interior of the vessel, the inner opening of the pipe being provided with mechanically operated valves. By this means the steam in the jacket is condensed and the temperature of the vessel gradually lowered. (B) The vessel may be divided into two chambers by a grating, and the air-pipes caused to open into distributing boxes communicating with the lower compartment by means of pipes. In this way the fats and gelatin can be removed during the boiling process and then separated from each other outside the vessel. (C) The pipe for drawing off condensed water from the steam jacket is provided with a cock or similar contrivance so that during boiling or sterilising the water can be run off, whilst it is allowed to accumulate during the drying process. These objects may be also achieved by suitably regulating the size of the condensed water pipe to cope with the amount of water in one case, but not in the other.—W. J. W.

Calcium cyanamide. E.P. 142,354 and G.P. 301,321, 305,061, and 305,532. See VII.

Cyanamides and nitrides. G.P. 302,461. See VII.

Carbon dioxide. G.P. 315,019. See VII.

Basic slag. G.P. 318,147. See X.

Fungicides. G.P. 318,710. See XIXb.

XVIII.—FERMENTATION INDUSTRIES.

Hop bitter acids; Colloid-chemical studies on —. H. Lüers and A. Baumann. *Koll.-Zeits.*, 1920, 26, 202—212.

HUMULONE (a hop bitter acid) exists in aqueous solution and in wort in the form of hydrosol, with a negative charge (*Cf. J.*, 1920, 462a). It is strongly adsorbed by charcoal from aqueous solution, and its power of producing a stable foam is in keeping with its power of reducing the surface tension and its adsorption. (*Cf. J.C.S.*, July.)

—J. F. S.

Chloropicrin; Action of — on yeast and on the flora of wine. G. Bertrand and Rosenblatt. *Comptes rend.*, 1920, 170, 1350—1352.

CHLOROPICRIN at a concentration of 5—6 mg. per l. entirely arrested fermentation due to yeast (a bottom fermentation yeast from a Champagnewine), but did not kill the yeast cells. In order to kill

the yeast, by an exposure for 24 hrs. at 27° C., a concentration of 30—40 mg. per l. was necessary. A concentration of 1—2 mg. per l. was sufficient entirely to prevent the development of *Saccharomyces vini*.—W. G.

Pentose-destroying bacteria; Fermentation characteristics of —. E. B. Fred. W. H. Peterson, and A. Davenport. *J. Biol. Chem.*, 1920, 42, 175—189.

THE action of organisms on a large number of substances of a carbohydrate nature and on closely related compounds was investigated. Arabinose, xylose, dextrose, galactose, mannose, levulose, sucrose, maltose, lactose, mannitol, glycerol, salicin, xylan, pyruvic, lactic, and malic acids are all decomposed more or less completely. Rhamnose, raffinose, melizitose, esculin, starch, inulin, cellulose, and succinic, tartaric, and citric acids are not attacked.—J. C. D.

Vitamines from yeast. Myers and Voegtlin. See XIXA.

PATENTS.

Malting process. R. de Fazi and F. Peroni. E.P. 143,121, 17.9.19. Conv., 22.5.19.

BARLEY, wheat, or other grain is exposed to ultra-violet rays during steeping and germination, whereby these processes are assisted.—L. E.

Alcohol and yeast from seaweed; Production of —. E. C. Bayer and S. Orlajensen. E.P. 119,030, 14.5.19.

SEAWEED is treated with hot water or a solution of salts such as chloride of lime, the extract being hydrolysed, e.g., by heating with 0.5% of sulphuric acid for 6 hrs. under a pressure of 3 atm., neutralised with calcium carbonate, and fermented with yeast. The residual seaweed is utilised for the production of algin.—L. E.

Acids; Utilisation of marine algæ for manufacture of formic, acetic, and butyric — [by fermentation] and for extraction of salts of iodine and potash. Darrasse Frères, and L. Dupont. E.P. 123,325, 13.2.19. Conv., 13.2.18.

THE material is fermented at, say, 35° C. with the bacteria on the algæ themselves, or with pure cultures obtained from earth manured with sea-wrack, the fermentation medium being kept slightly alkaline or neutral by addition of oxide, hydroxide, or carbonate of an alkali or alkaline-earth metal, or of the zinc or iron group. The organic acids formed are recovered, e.g., by distillation with sulphuric acid, the potassium sulphate in the residue is obtained by fractional crystallisation, and the iodine is precipitated from the mother liquor as cuprous iodide.—L. E.

Carbohydrate contained in plants, vegetable offal, and in natural or industrial waste products; Utilisation of —. Darrasse Frères, and L. Dupont. E.P. 123,326, 13.2.19. Conv., 13.2.18.

THE material is fermented with aceto-butyric bacteria found in earth fertilised with dung or dead leaves. Fermentation is effected preferably at 40°—50° C. for 2—3 days, and in presence of a neutralising agent such as oxides, hydroxides, carbonates, or other basic salts, in order to increase the yield of organic acids (formic, acetic, and butyric). The acids are separated by distilling the fermented product with a mineral acid. Since the cellulose substances are very little affected by the fermentation, the process serves for the preparation of pure cellulose fibre, provided the raw material consists of plants capable of yielding such fibre.—L. E.

Carbonating beverages. U.S.P. 1,338,352. See XI.

XIXA.—FOODS.

Bread; Colloid chemistry of —. I. *Colloid-chemical problems in the preparation of bread.* W. Ostwald. *Koll.-Zeits.*, 1919, 25, 26—45.

THE chief differences between flour, dough, and new and stale breads are of a physical rather than of a chemical nature. Each of these materials is essentially composed of colloidal particles, and by application of viscometric methods light may be thrown on such phenomena as the variation in the strength of flours, the process whereby bread becomes stale, variations in porosity of bread etc.

—H. W. B.

Bread; Colloid chemistry of —. II. *Viscometry of flour.* H. Lüers and W. Ostwald. *Koll.-Zeits.*, 1919, 25, 82—90, 116—136.

THE viscosity of mixtures of flour and water containing as much as 20% of flour can readily be determined by using large viscometers with large capillaries. The measurements must be made quickly owing to the tendency of the mixtures, even after boiling, to undergo sedimentation. The viscosity of rye mixtures is always considerably higher than that of mixtures of wheat flour and water of the same concentration. On keeping rye mixtures become more viscous, whilst wheat mixtures become thinner. Wheat suspensions are extremely sensitive to traces of acids, which greatly increase the viscosity; sodium chloride appears to reduce it. A flour of poor baking quality was found to give viscosity figures considerably below those given by normal flours.—H. W. B.

Bread; Colloid chemistry of —. III. *Colloid chemistry of rye and wheat gliadins, with special attention to gluten and baking problems.* H. Lüers. *Koll.-Zeits.*, 1919, 25, 177—196, 230—240.

THE viscosity of gliadin solutions is increased by traces of acids and alkalis and diminished by neutral salts. Changes in the gluten and in the baking qualities of flour appear to be dependent largely on changes occurring in the gliadin constituent of the gluten. The viscosity of gliadin solutions is greatly affected by changes of temperature, and the temperature at which the viscosity reaches a maximum is also the temperature at which the best dough and bread are produced. The addition of a trace of acid, which increases the viscosity of the gliadin solution, likewise improves the quality of the bread.—H. W. B.

Bread; Colloid chemistry of —. IV. *Flour of inferior baking quality.* H. Lüers and W. Ostwald. *Koll.-Zeits.*, 1920, 26, 66—67.

TWO further samples of flour with bad baking properties have been examined and found to give flour-water mixtures of abnormally low viscosity.

—H. W. B.

Milk; Carbon dioxide content of — a basis for distinguishing heated from unheated milk. L. L. van Slyke and R. F. Keeler. *J. Biol. Chem.*, 1920, 42, 41—45.

NORMAL milk shows a minimum value of 3.5—3 volume percentage of CO₂. Pasteurisation lowers this value below 3—2.5. This is of value in distinguishing the two classes of milk.—J. C. D.

Moisture in foodstuffs; Estimation of —. G. A. Stutterheim and H. Lazet. *Pharm. Weekblad*, 1920, 57, 457—462.

THE time necessary for the estimation of moisture in foodstuffs may be greatly shortened by assuming that with the same quantity of material and the same apparatus and conditions of experiment the ratio of the total moisture to the amount of

moisture lost in a given time is constant. The ratio factor is determined by numerous experiments. The method gave satisfactory results when applied to bread, cocoa, and sausages.—W. S. M.

Vitamine; Extraction of fat-soluble — from carrots, alfalfa [lucerne], and yellow maize by fat solvents. H. Steenbock and P. W. Boutwell. *J. Biol. Chem.*, 1920, 42, 131–152.

ALCOHOL is the best solvent to remove the fat-soluble accessory factor from these sources. Ether and other fat-solvents seldom give good results. The factor appears to resist saponification in the cold and to be associated with the fraction containing the lipochrome caroteno.—J. C. D.

Vitamines [from yeast]; Chemical isolation of —. C. N. Myers and C. Voegtlin. *J. Biol. Chem.*, 1920, 42, 199–205.

THE separation of an active crystalline fraction from dried yeast is described. The fraction readily loses its activity. (*Cf. J.C.S.*, July.)—J. C. D.

Aluminium vessels. Grossfeld. *See I.*

PATENT.

Foodstuffs; Manufacture of —. H. Bollmann. E.P. 142,764, 27.8.17.

SEE U.S.P. 1,260,656 of 1918; *J.*, 1918, 347 A.

XIXB.—WATER PURIFICATION; SANITATION.

Calcium cyanamide. Siehner. *See XVI.*

Chloropicrin. Bertrand and Rosenblatt. *See XVIII.*

PATENTS.

Water; Process for softening and purifying —. A. Gawalowski and J. Overhoff. G.P. 319,436, 17.7.14

THE water is treated with a ferrate or perferrate of an alkali, alkaline-earth, or heavy metal, preferably by passing it through a filter or contact mass impregnated with the purifying agent so as to remove the insoluble reaction products at the same time as the impurities.—A. R. P.

Air; Continuous production of pure, germ-free —. A. Wolff. G.P. 316,516, 13.9.17.

AIR is washed with ozonised water (G.P. 307,433–4; *J.*, 1918, 747 A), to which may be added a substance capable of increasing the ozone-fixing power of the water. (*Cf. U.S.P.* 1,152,066; *J.*, 1915, 1010.)—W. J. W.

Fungicides containing lime-copper; Method of increasing the adhesion of —. H. Cailler and C. Arragon. G.P. 318,710, 29.8.18. *Conv.*, 14.6.18.

CASEIN, caustic alkalis, or alkaline earths, or calcium carbonate may be added to lime-copper mixtures to increase their adhesive properties.—W. J. W.

Boilers for distilling water. C. Turnbull. E.P. 142,669, 16.5.19.

Antifouling paint. E.P. 131,601. *See XIII.*

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Ipecacuanha preparations; Determination of ipecacuanhic acid in —. R. Heuerre. *J. Pharm. Chim.*, 1920, 21, 425–433.

AN aqueous extract of the sample is filtered and saturated with ammonium sulphate; the precipitate formed is collected, washed with ammonium

sulphate solution, and, while still wet, dissolved in 90% alcohol. The alcoholic solution is evaporated and the residue dissolved in water; the weight of total solids of this solution gives the amount of ipecacuanhic acid after allowance has been made for the small quantity of ammonium sulphate still present. The acid is insoluble in ethyl acetate, chloroform, amyl alcohol, and acetone, but soluble in water and in alcohol. It has a distinct cupric-reducing power, and is precipitated by many saturated salt solutions. Powdered ipecacuanha contains 3–4% of the acid.—W. P. S.

Croton resin. R. Boehm. *Arch. Exp. Path. Pharm.*, 1915, 79, 138–154.

THE optical activity of croton oil is due to a resin which is dissolved in it. On removing the resin from the oil by extraction with methyl alcohol, the oil becomes inactive. The optical rotation of croton oil may therefore serve as an indication of its resin content. The extraction, purification, properties, and pharmacological character of the resin are described. (*Cf. J.C.S.*, July.)—S. S. Z.

Sulphurised derivatives of proteins having therapeutic value; Production of —. A. Pellizza. Sealed note 1446, 22.1.14. *Bull. Soc. Ind. Mulhouse*, 1919, 85, 295–296. Report by C. Meininger, *ibid.*, 296–297.

SULPHURISED derivatives of proteins having therapeutic value may be produced by condensing egg albumin, casein, etc., in the cold with carbon bisulphide in the presence of sodium carbonate. The products are precipitated by dilute acids, and are purified by re-solution in alkalis and re-precipitation by acids. The sulphur content of egg albumin was by this method increased from 1.44% to 3.68–3.75%, the respective proportions of assimilable sulphur being 27.7% and 62%.—A. J. H.

Local anæsthetics; Relationship between chemical constitution and physiological action in —. 1. *Homologues of procaine (novocaine).* O. Kamm. *J. Amer. Chem. Soc.*, 1920, 42, 1030–1033.

THE structure of *p*-diethylaminopropyl *p*-aminobenzoate, m.p. 69° C. (hydrochloride, m.p. 164° C.), bears considerable similarity to that of cocaine. Correspondingly, in physiological action it is intermediate between cocaine and procaine, being slightly more toxic than the latter, but more effective for the production of surface anæsthesia. *p*-Diethylaminopropyl *m*-aminobenzoate is an oil (hydrochloride, m.p. 151° C.). (*Cf. J.C.S.*, July.)—J. K.

Dimethylaminoantipyrine (pyramidone); Analytical characters of —. J. A. Sánchez. *Anal. Soc. Quim. Argentina*, 1918, 6, 422–428, 513–521.

THE methods given include: the colorimetric determination of pyramidone by iodic acid; detection of pyramidone in presence of large quantities of antipyrine by silver nitrate; gravimetric separation of antipyrine and pyramidone with formaldehyde; detection of a small quantity of antipyrine in pyramidone by means of nitrous fumes; and the alkalimetric determination of pyramidone. (*Cf. J.C.S.*, July.)—W. R. S.

Glass for the manufacture of ampullæ; Selection of —. G. E. Ewe. *J. Franklin Inst.*, 1920, 189, 649–653.

GLASS tubing intended for the manufacture of ampullæ for sterilised pharmaceutical preparations must not yield alkali or produce spicules of glass in the process. A 1% solution of morphine sulphate or a 0.2% solution of strychnine sulphate in normal saline solution, must not become turbid or opalescent when sterilised in the ampulla previously washed with dilute hydrochloric acid and water. No glass spicules must be formed when a 0.6% sodium

chloride solution is sterilised in the vessel. Tests for alkalinity and results obtained with typical samples of glass tubing are given.—C. A. M.

Oxalic acid and oxalates; Action of heat on —. G. Calcegni. Gazz. Chim. Ital., 1920, 50, i., 245—251.

HYDRATED oxalic acid melts at 98° C. and yields carbon monoxide and dioxide in unequal volumes at 110°—120° C. The anhydrous compound, obtained by protracted storage of the hydrated acid in a vacuum over sulphuric acid, begins to turn pasty and to decompose at 135° C., complete fusion and vigorous decomposition and distillation occurring at 160°. Among the products of the thermal decomposition of ammonium oxalate are carbon monoxide and dioxide, ammonium carbonate, and apparently oxaminic acid. Sodium (or potassium) hydrogen oxalate yields more carbon monoxide than corresponds with the equations: $2\text{NaHC}_2\text{O}_4 = \text{Na}_2\text{C}_2\text{O}_4 + \text{CO}_2 + \text{CO} + \text{H}_2\text{O}$ and $\text{Na}_2\text{C}_2\text{O}_4 = \text{Na}_2\text{CO}_3 + \text{CO}$, while formic acid is also formed and the residue contains carbon. Normal potassium oxalate yields at first water and carbon monoxide and dioxide, possibly owing to contamination with the acid salt; later only carbon monoxide is evolved, leaving the carbonate and carbon. Calcium oxalate gives carbon monoxide as sole gaseous product, while the residue contains calcium carbonate and carbon and, as it reduces silver nitrate solution, probably the formate as well. Barium and strontium oxalates behave similarly to the calcium salt, whilst magnesium oxalate decomposes directly into carbon monoxide and dioxide and magnesium.—T. H. P.

Acids; Identification of [organic] —. V. *p*-Halogeen phenacyl esters. W. L. Judefind and E. E. Reid. J. Amer. Chem. Soc., 1920, 42, 1043—1055.

A NUMBER of the phenacyl esters previously described (J., 1919, 199A) are oils, or solids of low melting-point. The *p*-bromoacetophenone previously used has therefore been replaced by its *p*-chloro-, bromo-, and iodo-derivatives, with the result that the esters obtained are found to have a range of melting-points very convenient for their identification. The esters of maleic, racemic, tartaric, and asparaginic acids decomposed without melting, whilst esters could not be obtained from gallic, linolic, oleic, oxalic, monochloroacetic, trichloroacetic, and formic acids. A liquid ester was obtained from α -hydroxybutyric acid. (Cf. J.C.S., July.)—J. K.

Formaldehyde; Preparation of —. M. D. Thomas. J. Amer. Chem. Soc., 1920, 42, 867—882.

FORMALDEHYDE is prepared with an absolute yield of 95% and a process yield of 55·6% by passing air at 125—150 l. per hr. through methyl alcohol and then over a coil of silver gauze heated to 400° C. The coil is only heated at the commencement of the process and afterwards is kept hot by the heat of the reaction. The yields with gold and copper spirals are rather less (50·5 and 40% respectively). With silver the best yield is obtained if the gas contains 0·4—0·5 g. of oxygen per 1 g. of alcohol. The presence of 1·7% of acetone or 10% of water does not influence the yield appreciably. (Cf. J.C.S., July.)—J. F. S.

Water; Detection of — in alcohol and other organic solvents. F. Henle. Ber., 1920, 53, 719—722.

A XYLENE solution of partially decomposed aluminium ethoxide is prepared by allowing aluminium turnings (27 g.), absolute alcohol (276 g.) and mercuric chloride (0·2 g.) to react until the evolution of hydrogen subsides and heating the product on the water bath until it appears dry. The alcohol of crystallisation is then distilled from an oil bath at 210°—220° C., and the crude aluminium ethoxide is subsequently cautiously heated to about

310° C. (thermometer immersed in the molten mass); after about 1 hr. the temperature falls to about 330° C. and heating is then discontinued. The product, whilst still hot, is dissolved in $\frac{1}{2}$ l. of boiling xylene and the solution is filtered through a dry paper; the clear, pale yellowish-brown filtrate can be preserved indefinitely if air and moisture are excluded. If a few drops of the reagent be added to a few c.c. of the liquid under examination, then, according to the quantity of water present, a voluminous, gelatinous precipitate of aluminium hydroxide is formed immediately or in the course of a few seconds. The percentage of water which can thus be detected in various solvents is as follows: ethyl alcohol, 0·05; methyl alcohol, 0·1; ether, 0·005; ethyl acetate, 0·1; acetaldehyde, 0·1; acetone, 1. The two substances last named also yield white turbidities due to the precipitation of aluminium ethoxide from the reagent, but these can be readily distinguished from aluminium hydroxide, since they immediately disappear on addition of a larger excess of reagent or of xylene. (Cf. J.C.S., July.)—H. W.

Removing sulphur from organic compounds. Herzog. See IV.

Bromine [in organic matter]. Wünsche. See XXIII.

PATENTS.

Cymene; Manufacture of para —. British and Foreign Chemical Producers, Ltd. From Rheinischo Kampferfabrik Ges. E.P. 142,738, 6.10.19.

THE terpenes of b.p. 175° C. obtained by the elimination of hydrogen chloride from the liquid additive compounds of pinene and hydrogen chloride, or the fractions of the same b.p. from Swedish and Polish turpentine oils, are chlorinated and then distilled with steam. *p*-Cymene, in yield approximating to 60% of that theoretically obtainable, is thus produced and is purified by treatment with suitable oxidising agents, such as potassium permanganate, or polymerising agents, such as zinc chloride.—D. F. T.

Organic substances; Purification of — by sublimation. The Selden Co., and J. McC. Selden. E.P. 142,902, 3.2.19.

THE constituents of a mixture of sublimable solid organic substances are separated by vaporising at least a part of the mixture into a current of substantially inert gas and fractionally crystallising by cooling the vapour mixture progressively in a chamber comprising a series of compartments of graded temperatures. Anthracene may thus be separated from crude anthracene press cake and phthalic anhydride from naphthalene, while crude anthraquinone, naphthalene, benzoic acid, and salicylic acid may be separated from their common impurities. (Cf. U.S.P. 1,321,716—7 of 1919; J., 1920, 100A.)—D. F. T.

Acids and acid anhydrides; Manufacture of organic —. M. Moest and E. Schirm, Assrs. to The Chemical Foundation, Inc. U.S.P. 1,337,253, 20.4.20. Appl., 27.5.16. Renewed 26.6.19.

ORGANIC acids and acid anhydrides are prepared by the action of oxides of nitrogen, intermediate in composition between nitric oxide and nitrogen pentoxide, on salts of organic acids.—G. F. M.

Ethyl sulphates; Process of making —. H. R. Curme, Assr. to Union Carbide Co. U.S.P. 1,339,947, 11.5.20. Appl., 7.7.19.

EXCESS of ethylene is treated with sulphuric acid with formation of ethyl hydrogen sulphate and diethyl sulphate; the latter is entrained with the excess of ethylene and separated subsequently.

—D. F. T.

Mercury sludge from electrolytic processes; Conversion of — into metallic mercury. Konsortium für Elektrochemische Ind. G.P. 319,476, 14.5.18.

THE sludge is stirred with acid liquids, to which small quantities of metals, such as zinc or iron, are added to liberate hydrogen. In certain cases, such as the production of acetaldehyde from acetylene, the formation of sludge may be avoided by adding these metals during the process.—W. J. W.

Photochemical apparatus. U.S.P. 1,339,675. See I. *Marine algæ.* E.P. 123,325. See XVIII.

Carbohydrate in plants etc. E.P. 123,326. See XVII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENTS.

Photographic sensitising dyes of the isocyanin type; Process of making —. L. E. Wise and E. Q. Adams. U.S.P. 1,338,346, 27.4.20. Appl., 10.2.19.

PHOTOGRAPHIC sensitising dyes are prepared by the interaction of sodium methoxide in absolute methyl alcoholic solution with mixtures of the quaternary halide addition products of an α -methylated quinoline derivative and of a γ -unsubstituted quinoline derivative.—G. F. M.

Photo-sensitising-dyes effective for infra-red radiation; Process of making —. E. Q. Adams and L. E. Wise. U.S.P. 1,338,349, 27.4.20. Appl., 4.4.19.

INFRA-RED photographic sensitising dyes are prepared by the interaction of air and sodium methoxide in absolute methyl alcoholic solution with the quaternary halide addition products of α -dimethylated quinoline derivatives.—G. F. M.

XXII.—EXPLOSIVES; MATCHES.

Military explosives; Theoretical maximum pressure developed by — in their own volume. J. E. Crawshaw. J. Franklin Inst., 1920, 189, 607—625.

A METHOD is described for calculating from experimental results the maximum pressure which an explosive would exert if exploded or detonated within a space which it fills completely, and tables are given showing the values obtained for 13 different explosives at varying densities.—C. A. M.

Picric acid; Preparation of —. Influence of the degree of sulphonation on the yields of trinitrophenol. M. M. Doll. Bull. Soc. Chim., 1920, 27, 370—374.

OTHER things being equal, the yield of trinitrophenol increases with the degree of sulphonation of the phenol.—W. G.

PATENTS.

Explosives; Manufacture of —. G. A. Hedberg. E.P. 142,763, 30.12.19. Addition to 140,967 (see U.S.P. 1,335,785; J., 1920, 429 A).

PINE-BARK meal to be used as an ingredient for explosive mixtures is ground to an extreme fineness, so that it gives a weight when loosely filled of 24 lb. per cub. ft., as compared with 15 lb. per cub. ft. obtained hitherto.—W. J. W.

Explosive. J. A. Hardel. U.S.P. 1,337,451, 20.4.20. Appl., 4.2.19.

AN explosive mixture contains a liquid nitro-hydrocarbon derivative saturated with a solid nitro-hydrocarbon derivative.—W. J. W.

XXIII.—ANALYSIS.

Turbidimeter; Photometric —. W. G. Bowers and J. Moyer. J. Biol. Chem., 1920, 42, 191—198.

AN attempt to devise a more accurate instrument for comparison of turbidities. The photometric oil spot is applied to the turbidimeter to balance intensities of illumination.—J. C. D.

Measuring and circulating gases soluble in water; Apparatus for —. B. Neumann and H. Schneider. Z. angew. Chem., 1920, 33, 128.

AN apparatus for measuring and circulating small quantities of gases soluble in water comprises two pipette-shaped glass vessels, terminating at their upper ends in T-pieces with two taps, and provided with a single tap below, the two vessels being connected at both ends by tubes, and communicating with a levelling tube and a gas collector, the gas in the latter being controlled by a pressure vessel. A lateral branch from the top connecting tube leads to a gas-reacting vessel or any desired apparatus. The glass vessels are half filled with water, previously saturated with the gas, which acts as a seal for the gas during its passage, and by suitable manipulation of the various taps, the gas may be introduced into either vessel and measured. (Cf. J.C.S., July.)—W. J. W.

Qualitative analysis of cations of the third and fourth groups. P. de Pauw. Chem. Weekblad, 1920, 17, 191—192.

AFTER treatment with hydrogen sulphide the acid filtrate is treated with sodium sulphate solution, which precipitates barium, strontium, and, in part, calcium. The filtrate contains iron, cobalt, nickel, manganese, chromium, aluminium, zinc, calcium, magnesium. Solid sodium carbonate and sodium hydroxide are added and, after boiling, sodium sulphide. The precipitate contains all the metals except aluminium. Dilute hydrochloric acid dissolves the precipitate except a residue of nickel and cobalt sulphides. To this solution solid carbonate is added and then sodium hypochlorite. Zinc and chromium remain in solution as zincate and chromate respectively and may be separated by means of barium chloride. The residue consists of ferric hydroxide, hydrated manganese dioxide, calcium and magnesium carbonates. This is treated with 5% acetic acid containing a little phosphate, which dissolves calcium and magnesium as acetates. The residue is dissolved in sodium peroxide and nitric acid and examined for iron and manganese in the usual way.—W. S. M.

Gravimetric analysis; Theory of — with special reference to sources of error. L. Moser. Z. anal. Chem., 1920, 59, 1—10.

THE author discusses the various sources of error in gravimetric analysis, classified as avoidable and unavoidable.—W. P. S.

Colorimetric estimations with solutions containing two coloured substances. K. G. Falk and H. M. Noyes. J. Biol. Chem., 1920, 42, 109—130.

UNLESS the standard and unknown solutions are very nearly alike in composition and concentration such methods are open to grave errors.—J. C. D.

Chromium and manganese; Separation of —. M. Herschkowitsch. Z. anal. Chem., 1920, 59, 11—12.

CHROMIUM and manganese, in solution, are oxidised to chromate and permanganate respectively, and the solution is heated for some hours with the addition of ammonium sulphate and an excess of ammonia. The manganese is precipitated completely and may be collected and weighed as Mn_2O_3 , whilst the chromate remains in solution.—W. P. S.

Ferric hydroxide; Carrying down of calcium and magnesium hydroxides by precipitates of —. Toporescu. *Comptes rend.*, 1920, 170, 1251—1253.

Ferric hydroxide when precipitated tends to carry down with it both calcium and magnesium hydroxides if salts of these metals are present in the solution, and this is not entirely prevented by the presence of 2% of ammonium chloride. Any calcium hydroxide thus carried down may be removed either by washing the precipitate with a 5% boiling solution of ammonium nitrate or by dissolving the precipitate in acid and re-precipitating. Neither of these processes is adequate for the complete removal of any magnesium hydroxide carried down.

—W. G.

Nitrosoindole test. [Detection of nitrates.] J. Groenewege. *Med. Geneesk. Lab. Weltevreden*, 1919, 177—193.

WHEN certain organisms (*e.g.*, cholera vibrios) are grown on peptone medium the latter gives the nitrosoindole reaction (red coloration) when treated with a drop of mineral acid, the reaction being obtained without the addition of nitrite. This is due to the presence of nitrite formed by reduction of traces of nitrate present in the peptone. The reaction is not given if the peptone medium is inoculated previously or simultaneously with a denitrifying organism which reduces the nitrate to ammonia or nitrogen. Traces of nitrate may be detected by adding the substance to be tested to a culture of cholera vibrios *etc.*, in peptone medium freed from nitrate as described, and then applying the nitrosoindole test.—W. P. S.

Gas analysis; Exact methods of —. E. Ott. *J. Gasbeleucht.*, 1920, 63, 198—205, 213—220, 246—252, 267—271.

A GAS-ANALYSIS apparatus is described of the constant pressure type, with control tube, the gases being stored over mercury. Carbon monoxide and hydrogen are estimated separately or together by burning in contact with platinum wire at 300° C., the wire or wires being enclosed in a silica capillary tube, which is preferable to a platinum tube, being non-porous and less expensive. Methane does not combine with oxygen at 300° C., but commences to do so at 400° C., burning completely at bright red heat. Measured quantities of the pure gases were mixed together and check analyses carried out by completely burning all the gas and measuring the oxygen used up and the carbon dioxide produced; by absorbing the carbon monoxide first with ammoniacal cuprous chloride followed by fractional combustion of the methane and hydrogen; and by fractional combustion, first of the hydrogen and carbon monoxide together, followed by that of the methane. The apparatus and method of manipulation are described in detail, together with the results of the check analyses. The platinum wire must be re-vivified or replaced from time to time.—W. P.

Bromine [in organic matter]; Estimation of —. F. Wütsche. *Arch. Exp. Path. Pharm.*, 1919, 84, 328—339.

THE substance is heated with sodium peroxide in a nickel basin first on the water bath, then over a free flame, and eventually incinerated. The ash is washed out with distilled water, the solution filtered, and the filtrate neutralised with concentrated sulphuric acid and treated with potassium permanganate and sulphuric acid. The free bromine is blown over by means of a current of air into a vessel containing a known volume of fuchsine and sulphurous acid, and estimated colorimetrically by comparing the violet coloration which is produced with a set of standards. 2 mg. of neuronal was estimated in 30 c.c. of blood with an error of 12%. Neuronal, adaline, and bromural were estimated in frog's tissue by this method with an error of about 7%.—S. S. Z.

See also pages (x) 477, *Combustion gases from coal* (Custek). 481, *Sulphite cooking process* (Baker). 482, *Lignin* (Becker). 485, *Nitrous acid* (Matignon and Gire); *Arsenic acid* (Fleury); *Sulphites* (Waterman); *Nitrites* (Hermans). 486, *Graphitic carbon* (Selvig and Ratliff). 490, *Refractory materials* (Tarrant). 492, *Mercury* (Bouton and Duschak); *Tin* (Baxter and Starkweather). 495, *Paint* (Bingham and Green). 496, *Tanning materials* (Baldracco and Camilla); *Tanning extracts* (Schell); *Tannin* (Lee). 497, *Gelatin* (Bennett and Holmes). 499, *Milk* (Van Slyke and Keeler); *Moisture in foods* (Stutterheim and Lazet). 500, *Ipecacuanha preparations* (Heurre); *Pyrambiones* (Sánchez). 501, *Organic acids* (Judekind and Reid); *Water in organic solvents* (Henle).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

- Adams. Purifying liquids. 16,125. June 15.
 Allgem. Ges. f. Chem. Ind. Carrying out operations at high temperature in an air-free space. 17,056. June 23. (Ger., 13.3.19.)
 Ayres. Process of refining liquids. 16,412. June 17. (U.S., 3.10.17.)
 Brownlee and Ganahl. Heat treatment of liquids. 16,776. June 21.
 Elektro-Osmose A.-G. Leaching vegetable, animal, or mineral substances. 16,113. June 14. (Ger., 9.1.15.)
 Feldenheimer and Plowman. Catalysts and catalytic reactions. 16,917. June 22.
 Fuller-Lehigh Co. Pulveriser mills. 16,760. June 21. (U.S., 1.6.18.)
 Heisch. Apparatus for precipitating solids by action of gas on liquids. 16,529. June 18.
 Hewitt. Removal of liquids from pastes, wet precipitates, and crystalline magmas. 16,803. June 22.
 Hunt. Apparatus for maintaining finely-divided solids in suspension in liquids *etc.* 16,612. June 19.
 Lilienfeld and others. 16,726. See XI.
 Krause. Evaporation of solutions and effecting chemical reactions. 16,320. June 16. (Ger., 29.1.17.)
 Metallbank u. Metallurgische Ges. Production of solid bodies in fine powder from liquids, pulpy masses, *etc.* 16,172. June 15. (Ger., 16.4.18.)
 Skinninggrove Iron Co., and Smith. 16,701. See XI.

COMPLETE SPECIFICATIONS ACCEPTED.

- 20,169 (1918). Frey and Henz. Suppressing froth or scum when boiling liquids. (121,293.) June 23.
 5730 (1919). Gay. Absorption, reaction, distillation, rectification, and like columns. (144,374.) June 23.
 6330 (1919). Chemical Equipment Co. Corrosive fluid valves. (124,445.) June 30.
 7052 (1919). Summers and Whittard. Rotary furnaces, retorts, *etc.* (141,815.) June 30.
 9819 (1919). Smith. Furnace retorts. (126,614.) June 30.
 30,112 (1919). Watt. Treating filtration membrane. (144,961.) June 30.
 3664 (1920). Elmore. Centrifugal dryers. (144,979.) June 30.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

- Auburtin and others. Coke-gas generator. 17,050. June 23. (Fr., 23.6.19.)
 Bateman. Heating vertical retorts for gasifying coal etc. 16,144. June 15.
 Chadbourn and Remington. Production of hydrocarbons from oil-bearing minerals. 16,088. June 14.
 Coe. Treatment of peat. 16,181. June 15.
 Farbenfabr. vorm. F. Bayer u. Co. 17,178 and 17,198. *See VII.*
 Farb. vorm. Meister, Lucius, u. Brüning. Manufacture of methane. 16,757 and 16,905. June 21 and 22. (Ger., 28.6. and 1.7.19.)
 Fischer. Distillation of coal. 17,174. June 24. (Ger., 13.11.16.)
 Fyelman and Sprott. Purification of acetylene. 16,528. June 18.
 Robin. Treatment of fabrics for gas mantles. 16,898. June 22.

COMPLETE SPECIFICATIONS ACCEPTED.

- 6445 (1919). Hood. Recovery of cyanogen from coal gas. (144,398.) June 23.
 9819 (1919). Smith. *See I.*
 13,606 (1919). Campbell. Process of making gas. (144,868.) June 30.
 21,478 (1919). Wells. Gas-producers. (144,520.) June 23.
 10,496 (1920). Griffin. Enrichment of producer-gas. (144,989.) June 30.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

- British Dyestuffs Corp., and others. 16,179 and 17,037. *See XX.*
 Brock and others. 17,238. *See XIII.*
 Irinyi. Distillation of tar etc. 17,347. June 25. (Ger., 25.11.18.)

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

- Act.-Ges. f. Anilinfabr. Manufacture of azo dyestuffs. 16,198—16,202. June 15. (Ger., 4.6, 29.6, 28.7, 31.7.15, and 18.1.18.)
 Act.-Ges. f. Anilinfabr. Manufacture of sulphurised dyestuffs. 16,906—7. June 22. (Ger., 21.1.14.)
 British Dyestuffs Corp., Perkin, and Clemo. Manufacture of benzaldehyde derivatives and triphenylmethane colouring matters. 17,038. June 23.
 Johnson (Badische Anilin u. Soda-Fabr.). Manufacture of intermediate products and colouring matters, and their application in dyeing and printing. 16,299. June 16.
 Soc. Chim. des Usines du Rhône. Dyestuffs of the indigo series. 16,523. June 18. (Fr., 14.10.19.)

COMPLETE SPECIFICATION ACCEPTED.

- 20,799 (1918). Hart. Production of water-soluble dyes. (144,336.) June 23.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

- Act.-Ges. f. Anilinfabr. Manufacture of cellulose esters. 16,903—9. June 22. (Ger., 11.7.14 and 30.5.18.)
 Badische Anilin u. Soda-Fabr. Production of solutions of cellulose acetates. 16,861. June 22. (Ger., 9.2.14.)
 Bennert. Treating and scouring wool etc. 16,546. June 18. (Ger., 8.2.15.)

British Cellulose and Chemical Manufacturing Co. Cellulose-ester compositions. 17,435. June 26. (U.S., 30.6.19.)

- Burgess, Ledward and Co., and Harrison. Treatment of cellulose fibres or substances. 16,649. June 21.
 Charbonneau. Treatment of flax etc. 17,091. June 23. (Fr., 20.2.18.)
 Folien u. Flitterfabr. A.-G. Obviating high inflammability of celluloid. 16,458. June 17. (Ger., 13.9.17.)
 Glanzfäden A.-G. Preparing durable cupric-ammonia cellulose solutions for spinning artificial threads etc. 16,075. June 14. (Ger., 12.10.17.)
 Hartridge and Newcombe. Manufacture of waterproof paper. 16,916. June 22.
 Kaye. Use of new materials in paper making. 17,252. June 25.
 Robin. 16,898. *See II.*
 Simons (Sager). De-inking paper for re-pulping. 16,437. June 17.

COMPLETE SPECIFICATIONS ACCEPTED.

- 8086 (1919). Dorr Co. *See VII.*
 31,192 (1919). Gillet et Fils. Treatment of vegetable fibres. (144,563.) June 23.
 1597 (1920). Bonwitt. Manufacture of dissolved or gelatinous cellulose esters. (138,078.) June 23.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

- Bennert. Preparation of dye-baths. 16,892. June 22. (Ger., 24.12.15.)
 Hitchen. Printing warps or yarns. 16,831. June 22.
 Johnson (B.A.S.F.). 16,299. *See IV.*
 Mitchell. Waterproofing fabric. 16,783. June 21.
 Petty. Dyeing textiles. 16,607. June 19.
 Wioland. Dyeing and bleaching apparatus. 16,409. June 17. (Fr., 7.7.19.)

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

- Audianne. Apparatus for manufacture of sulphuric anhydride. 16,731. June 21.
 Badische Anilin u. Soda-Fabr. Manufacture of nitrogen-hydrogen mixtures for synthetic production of ammonia. 16,209. June 15. (Ger., 2.7.15.)
 Badische Anilin u. Soda-Fabr. Production of oxides of nitrogen from ammonia by catalytic oxidation. 16,210. June 15. (Ger., 1.4.16.)
 Badische Anilin u. Soda-Fabr. 16,086. *See XVI.*
 Bergve. Manufacture of alkaline reducing agents. 16,936. June 22.
 Codrington. 16,146. *See X.*
 Crossley. Mica, mica compounds, etc. 17,066. June 23.
 Farbenfabr. vorm. F. Bayer u. Co. Purifying gases containing hydrogen sulphide and obtaining sulphur. 17,178 and 17,198. June 24. (Ger., 24.5.17 and 3.7.18.)
 Levinson and Martin. Utilising waste lime sludge. 16,534. June 18.
 Maucelaire and Nouvelle. Apparatus for producing hydrogen. 16,578. June 18.
 Metallbank u. Metallurg. Ges. Treating sal ammoniac skimmings. 16,392. June 17. (Ger., 28.2.18.)
 Naef. 16,586. *See X.*
 Röhm. Production of solid non-hygroscopic iron salts. 17,437 and 17,453. June 26. (Ger., 27.12.18 and 11.4.19.)
 Soc. Electro Metallurgique Française. Preparation of aluminate of lime for manufacture of pure alumina. 17,062. June 23. (Fr., 7.3.16.)

COMPLETE SPECIFICATIONS ACCEPTED.

3621 (1919). British Oxygen Co., Bray, and Balfour. Manufacture of hydrogen. (144,751.) June 30.

6445 (1919). Hood. *See* II.

7131 (1919). Jackson (Sperry). Producing lead salts. (144,819.) June 30.

8086 (1919). Dorr Co. Manufacture of caustic soda, particularly from pulp digestion residues. (131,272.) June 30.

11,654 (1919). Bergve. Decomposition of alkali aluminosilicates. (127,566.) June 23.

13,660 (1919). Clark. Removal of arsenic from sulphuric acid. (144,869.) June 30.

24,040 (1919). Clayton. Apparatus for making oxygen compounds of sulphur. (141,661.) June 30.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Adair. Drying china clay. 16,017. June 14.

Corning Glass Works. Glass compositions. 16,452. June 17. (U.S., 26.2.17.)

Giddens, Hoxby, Rudd, and R. G. H. Furnace Co. Glass furnaces. 16,303. June 16.

Gimingham. Glass manufacture. 16,436. June 17.

Porzellanfabr. Kahla. Sweigniederlassung Freiberg. Cement for joining porcelain bodies after burning. 16,047. June 14. (Ger., 6.10.17.)

Schiff. Manufacture of dental cements. 16,196 and 16,297. June 15 and 16.

Truel. Glass-melting tanks or furnaces. 16,191. June 15.

COMPLETE SPECIFICATIONS ACCEPTED.

21,443 (1918). Shaw, Shaw, and Shaw. Furnace for melting glass etc. (144,744.) June 30.

5441 (1919). Denis. Manufacture of porous refractory material. (144,359.) June 23.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Larson. Manufacture of porous mortar products. 17,312. June 25. (Norway, 3.7.19.)

Schiff. 16,196 and 16,297. *See* VIII.

Schol. 16,067. *See* X.

COMPLETE SPECIFICATION ACCEPTED.

2775 (1919). Thompson (Arentox Co.). Protecting and fireproofing wood etc. (144,342.) June 23.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Aoyagi. Electrometallurgical manufacture of ductile bodies of high-fusing metals etc. 16,389. June 17.

Bayer. Transforming mineral stuffs. 16,640. June 19.

Bingham. Manufacture of rustless steel. 16,013. June 14.

Bowen. Furnaces for melting metals etc. 16,242. June 16.

Caink, and Radiant Products, Ltd. Annealing etc. metals. 16,881. June 22.

Codrington. Manufacture of lead and lead compounds. 16,146. June 15.

Gillespie. Manufacture of finely-divided metals and alloys. 16,742. June 21.

Jones. Coating metal. 16,325. June 16.

Krupp. Production of low-carbon ferrochromium. 17,388, 17,392—3. June 26. (Ger., 27.7.16, 3.10.16, 11.1.18.)

McKenna. Treating ferrotitanium to eliminate impurities. 16,559. June 18. (U.S., 11.8.16.)

Metallind. Schiele u. Bruchsalser. Welding aluminium to iron. 16,107. June 14. (Ger., 27.6.18.)

Minerals Separation, Ltd. (Wilkinson). Ore concentration. 17,092. June 23.

Naef. Manufacture of nickel and nickel salts. 16,586. June 19.

Naef. Manufacture of copper. 16,587. June 19.

Schol. Obtaining porous slag in dry state. 16,067. June 11. (Ger., 15.9.13.)

Thal. Apparatus for producing steel. 16,020—1. June 11. (Fr., 25.11.15, 20.2.18.)

COMPLETE SPECIFICATIONS ACCEPTED.

4668 (1917). Stahlwerke Lindenberg A.-G. Steel alloy. (144,326.) June 23.

16,270 (1919). Gush. Furnace for hardening high-speed steel etc. (144,486.) June 23.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Allgem. Ges. f. Chem. Ind. Heating device in electric resistance furnaces. 17,055. June 23. (Ger., 13.3.19.)

Aoyagi. 16,389. *See* X.

Forshee. Electrodes. 16,576. June 18. (U.S., 18.3.20.)

Lilienfeld, and Metallbank u. Metallurg. Ges. Electrical gas purification. 16,726. June 21. (Ger., 8.8.18.)

Pechkrantz. Electrolysers. 17,348. June 25. (Switz., 27.6.19.)

Skinningrove Iron Co., and Smith. Electrostatic plant for depositing dust etc. from gases. 16,701. June 21.

COMPLETE SPECIFICATIONS ACCEPTED.

6654 (1919). Morgan Crucible Co., and Speirs. Electrically-heated furnaces. (144,802.) June 30.

10,357 (1919). Campbell and others. Control of power absorbed in an electric furnace. (144,438.) June 23.

17,902 (1919). British Thomson-Houston Co., and Hastings. Electric arc furnaces. (144,503.) June 23.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Ayres. Recovering neutral oils from their "foots" or soap stock. 16,411. June 17. (U.S., 21.5.17.)

De Bruyn, Ltd., and Revis. Treatment of edible oils etc. 16,711. June 21.

Dunham. Emulsifiable oil material in dry form. 16,893. June 22.

Dunham. Casein-oil product. 16,894. June 22.

Durden. Apparatus for extracting oil from nuts, seeds, etc. 17,206. June 24.

Elektro-Osmose A.-G. Decolorising liquids containing glycerin. 16,114. June 14. (Ger., 1.4.19.)

Fujimura and others. Apparatus for hardening fatty oils. 16,090. June 14. (Japan, 12.1.20.)

Johnston. 17,231. *See* XIX.

Persapol Ges. Manufacture of soaps. 16,786. June 21. (Ger., 10.10.16.)

Reinfurth. Manufacture of detergents. 17,475—6. June 26. (Ger., 31.7.16, 5.3.18.)

Rhodes. Soap. 16,289. June 16.

Warburton. Refining oils. 16,567. June 18.

COMPLETE SPECIFICATIONS ACCEPTED.

15,419 and 15,422 (1919). Blair, Campbell, and McLean, Blair, and Ferguson. Apparatus for hydrogenating oils and fats. (144,478—9.) June 23.

1841 and 1975 (1920). Verein. Chem. Werke. *See* XVIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Brock, Redman, and Weith. Manufacture of phenolic condensation products. 17,238. June 24. (U.S., 6.6.18.)

Ivinson and Roberts. Paint etc. impervious to oils, spirits, etc. 16,708. June 21.

Walpole. Production of resin from solvent naphtha. 16,280. June 16. (Ger., 1.2.16.)

COMPLETE SPECIFICATION ACCEPTED.

13,048 (1919). Grant. Drying-oil, giving a flat surface paint. (144,862.) June 30.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATION.

Pratt. Vulcanised articles and process for producing same. 17,366. June 25.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Bevis. Treatment of leather. 16,305. June 16.
Bloxam (Gerb- u. Farbstoffwerke Renner u. Co.). Manufacture of tanning agents. 17,203 and 17,340. June 24 and 25.

Fairweather (Luftfahrzeugbau Schütte-Lanz). Producing glue from blood. 17,308. June 25.

Gerb- u. Farbstoffwerke Renner u. Co. Manufacture of tanning agents. 17,293—5, 17,341—3, 17,461. June 25 and 26. (Ger., 10, 16, and 28.2.14, 7.7.15, 12 and 21.11.18, 31.7.19.)

McQuitty. Manufacture of sheets of edible gelatin. 16,743. June 21. (U.S., 20.6.19.)

Strain. Manufacture of sheet gelatin. 16,686. June 21.

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Badische Anilin u. Soda-Fabr. Manufacture of fertilisers. 16,084—5. June 14. (Ger., 29.7.16 and 26.3.17.)

Badische Anilin u. Soda-Fabr. Transforming ammonia into a salt for use as a fertiliser. 16,086. June 14. (Ger., 23.3.18.)

Bean. Soil sterilising. 16,998. June 23.

Holmes. Preparation of peat etc. for application as fertiliser. 16,301. June 14.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

Lowe and Patterson. Manufacture of sugar. 16,322. June 16.

Miller. Filtration of sugar cane juices etc. 16,816. June 22.

COMPLETE SPECIFICATIONS ACCEPTED.

1841 and 1975 (1920). Verein. Chem. Werke. See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Aktie. Dansk Gaerings-Industrie. Production of air-yeast etc. 16,454. June 17. (Denmark, 5.7.19.)

Deutsch Koloniale Gerb- u. Farbstoff Ges., and Römer. Fermentation-process. 16,395. June 17.

Soc. F. Huherty et Cie. Manufacture of milk vinegar. 17,445. June 26. (Fr., 3.10.13.)

Wohl. Production of pure pressed yeast. 17,079. June 23.

COMPLETE SPECIFICATIONS ACCEPTED.

1841 and 1945 (1920). Verein. Chem. Werke. Manufacture of glycerol from sugar. (138,099 and 138,329.) June 23.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Adams. 16,125. See I.

Beard, Menzies, and M.B. Laboratories. Disinfectants and antiseptics. 16,799. June 22.

De Bruyn, Ltd., and Revis. 16,711. See XII.

Dunham. 16,893—4. See XII.

Dunham. Dry shortening material. 16,895. June 22.

Johnston. Manufacture of butter fats. 17,231. June 24. (U.S., 26.6.19.)

Merrell-Soule Co. Production of food products. 16,570. June 18. (U.S., 2.10.19.)

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Aktie. North Western Cyanamide Co., and Lie. Producing urea from cyanamide. 16,309. June 16.
Badische Anilin u. Soda-Fabr. Manufacture of urea. 16,211. June 15. (Ger., 9.8.16.)

Blom. Manufacture of nitrophenol ethyl ethers. 16,187. June 15.

British Dyestuffs Corp., Green, and Herbert. Manufacture of chlorinated derivatives of toluene. 16,179. June 15.

British Dyestuffs Corp., Green, and Clibbens. Manufacture of chlorinated derivatives of toluene. 17,037. June 23.

Farbenfabr. vorm. F. Bayer u. Co. Manufacture of cyanamide derivatives of α -halogenated acids. 17,177. June 24. (Ger., 22.11.15.)

Pratt. Sulphur-terpene compounds. 17,367. June 25.

Schmidt. Manufacture of protocatechuic aldehyde and acid from piperonal. 16,376. June 17. (Ger., 15.9.13.)

Smith. Treatment of organic compounds. 16,729. June 21.

Wohl. Manufacture of phthalic anhydride. 16,298. June 16. (Ger., 28.6.16.)

COMPLETE SPECIFICATIONS ACCEPTED.

4017 (1919). Soc. des Aciéries et Forges de Firminy. Catalyst for making acetaldehyde from acetylene. (124,194.) June 30.

6639 (1919). Blom. Manufacture of asymmetric ureas. (125,584.) June 30.

6780 (1919). Job and Guinot. Preparation of arsine derivatives. (144,806.) June 30.

17,418 (1919). Baumann and Grossfeld. Recovery of lecithin from animal and vegetable substances. (144,895.) June 30.

17,633 (1919). Marsh. Elimination of carboxyl groups. (144,897.) June 30.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Bassani. Photochromographic processes. 17,190. June 24. (Fr., 24.6.19.)

Seidl. Colouring pigment, especially carbon, prints. 17,068. June 23. (Austria, 31.1.19.)

XXIII.—ANALYSIS.

APPLICATION.

Soc. des Hauts-Fourneaux de Rouen. Calorimeters. 16,503. June 18. (Fr., 10.5.19.)

COMPLETE SPECIFICATION ACCEPTED.

16,651 (1919). Foster. Radiation pyrometers. (144,489.) June 23.

I.—GENERAL; PLANT; MACHINERY.**PATENTS.**

Acid-proof and oil-proof containers. T. W. Jones. E.P. 143,651, 5.3.19.

THE interior surface of the container is coated with a layer of fireproof cement of the kind known as "hard finish plaster" so as to give a surface having a fine and even porosity. When dry and set, the surface is washed with petrol and then varnished with an alcoholic solution of shellac.

—W. H. C.

Drying machinery. A. A. Short and W. G. Watkins. E.P. 143,707, 16.5.19.

A HEATED gas passes through the central cylinder of a rotary dryer and then returns through a conduit which is provided with a valve, so that the whole of the gas may be made to return in contact with the material or part in contact with the material and part through an exterior jacket.

—W. H. C.

Drying machinery. A. A. Short and W. G. Watkins. E.P. 143,708, 16.5.19.

THE inner of a pair of co-axial cylinders is supported by tangential H-shaped stays, which are attached at one end to the outer cylinder and at the other to a collar which surrounds the inner cylinder and allows slight peripheral movement. The heating agent passes through the inner cylinder and/or around the outer cylinder, and the material to be dried passes through the space between the cylinders.—W. H. C.

Drying-kiln. C. A. Cutler. U.S.P. 1,341,884, 1.6.20. Appl., 26.8.18.

IN a drying-kiln constructed with a false ceiling and a number of supports above the floor, air openings are provided in the ceiling and false ceiling, air blasts in the space between the floor and the supports above it, and an air exhaust duct opening near the bottom of the kiln.—B. M. V.

Muffle furnace. W. O. Amsler. U.S.P. 1,341,031, 25.5.20. Appl., 3.4.16.

IN a single open-ended muffle furnace the gas is admitted above the muffle and, mixed with preheated air, it burns in a series of transverse combustion chambers adjacent to the top of the muffle; the products of combustion pass downwards around the muffle to a recuperator below in which the air supply is preheated.—J. H. L.

Furnace and [iron alloy] lining therefor. J. P. Copland, Assr. to The Elyria Iron and Steel Co. U.S.P. 1,341,299, 25.5.20. Appl., 10.8.18.

THE furnace lining is of cast metal consisting of Fe 70—75, Cr 15—25%, the remainder being a metal of the iron group.—B. M. V.

[Heat] recuperator. C. L. Trinks, Assr. to Tate-Jones and Co., Inc. U.S.P. 1,342,073, 1.6.20. Appl., 28.8.19.

THE conduit for conveying the air to be heated is made of metal, the low temperature end being of low, and the high-temperature end of high, heat-resisting quality.—B. M. V.

Heating furnace. C. L. W. Trinks, Assr. to Tate-Jones and Co., Inc. U.S.P. 1,342,074, 1.6.20. Appl., 28.8.19.

A FURNACE is constructed with thin refractory walls, roof, etc., enclosed in a metallic casing, having on its outer surface spacing ribs adapted to direct the flow of air along the casing. A second set of walls and roof of heat-insulating material

rest against the ribs and are enclosed in an outer metallic casing.—B. M. V.

Filling material for gas-absorption towers. Kunstler und Co. G.P. 319,558, 2.10.17.

THE filling material consists of strips of sheet metal, wider at one end than at the other, wound into spirals so that the narrower part is outside; the distance between two consecutive coils is smaller than the width of the outermost layer of metal.—A. R. P.

Ovens for baking, drying, and other purposes. C. Bucci. E.P. 143,809, 2.1.20.

Tunnel kilns. G. H. Benjamin. E.P. 143,652, 6.3.19.

SEE U.S.P. 1,295,955 of 1919; J., 1919, 347A.

Intermixing liquids, gases, and the like. O. F. Bruman. E.P. 143,775, 10.9.19.

SEE G.P. 301,042 of 1915; J., 1918, 453A.

[Electro-osmotic] process of treating colloidal and finely sub-divided substances. B. Schwerin, Assr. to Ges. für Elektro-Osmose. U.S.P. 1,326,106, 23.12.19. Appl., 19.6.12. Renewed 28.10.19.

SEE E.P. 14,369 of 1912; J., 1913, 241.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Nitrogen in coal, coke and tar. Determination of —. A. Parker. Gas J., 1920, 150, 624—625.

THE unreliability of the Kjeldahl method as applied to fuels is confirmed (*cf.* J., 1919, 399A). A modified Dumas method is described which gave more satisfactory results. Pure carbon dioxide, washed by passage through a solution of sodium carbonate, was passed through a strongly heated silica tube lined with asbestos paper and containing a platinum boat charged with 15 g. of potassium chlorate for subsequent generation of oxygen; 3 g. of ignited copper oxide powder; 10 g. of ignited copper oxide powder mixed with 1 g. of sample; 30 g. of ignited copper oxide; and 15 cm. of reduced copper gauze spiral. In the case of tars the sample was placed between layers of copper oxide in a boat. When the combustion with copper oxide was ended, the chlorate, previously cool, was heated to develop a small quantity of oxygen, which completed the combustion of the sample. The gases were collected in a nitrometer and the nitrogen measured after absorption of carbon dioxide, oxygen, and carbon monoxide in the usual way.

—H. J. H.

Carbonisation of coal in vertical retorts with steaming. Fourth Report of Research Sub-Committee of Gas Investigation Committee of Inst. Gas Eng., June, 1920. Gas J., 1920, 150, 604—631.

TESTS were made to study the effect of steaming the charge during carbonisation in a Glover-West vertical retort plant at Uddington Gas Works near Glasgow, using a Lanarkshire coal, a typical analysis of which was:—moisture 9.8%, volatile matter 32.7%, fixed carbon 50.5%, ash 7.0%. Five tests are reported with varying proportions of steam and the detailed results are tabulated. Thermal balance sheets on a dry coal basis are also given. The results showed that there was a rise in the thermal efficiency of gas production (ratio of the heating power of the gas to this quantity plus the heat expended in its production) as the

proportion of steam was increased until a maximum was reached at 32.7% added steam. More drastic steaming caused a decrease in efficiency. The composition of the gases produced showed that a considerable quantity of methane as well as water-gas was produced by steaming. The results are analysed in numerous tables showing how the heat was utilised in the process, also giving the weight balances of the carbon, nitrogen, and sulphur and their distribution in the products. Analyses of tar, liquor, and benzol obtained by oil washing are also given. Two tests are reported in which much more extensive steaming was practised with a view to effecting complete gasification. This was practically achieved, the residue drawn containing 94.9% of ash, but the fuel consumption for heating the retort and for the production of the steam exceeded the heating power of the coal treated. Practical details of the tests are given in an appendix and also of the analytical methods employed (*cf.* Parker, *ante*).—H. J. H.

Gas production; Oxygen in —. H. J. Hodsman and J. W. Cobb. *Inst. Gas Eng.*, June, 1920. *Gas J.*, 1920, 150, 640–647.

THE carbonisation of coal and the gasification of the coke residue by a suitable air-steam blast are almost thermally neutral processes. Theoretically then the production of gaseous fuel should be attainable at very little cost of fuel. The considerable expenditure of heat in modern carbonisation practice is occasioned by the difficulty inherent in external heating at a high temperature. When internal heating is applied as in producer practice, dilution with atmospheric nitrogen involves certain heat losses and also difficulties in distribution and utilisation. The elimination of inert constituents as in the water-gas process is only achieved at the cost of further heat losses and an intermittent operation of the process. The maximum thermal efficiency of carbonisation and gasification would be attainable in a process of internal heating by partial combustion of the fuel under the influence of a regulated mixture of oxygen and steam. The minimum heat losses corresponding, for processes of carbonisation and complete gasification under conditions postulated, are estimated at 2.33% and 5.09% respectively of the heating power of the coal treated. These heat losses correspond with a thermal efficiency of gas production exceeding 90%, even when the fuel for steam raising is deducted. The quantities of oxygen required are small relative to the volumes of gas produced, and while financial conditions are at present prohibitive, the claims of Claude and Jefferies and Norton (*cf.* J., 1919, 122r) indicate that in the near future the cost of oxygen may no longer prove an obstacle to its application to gas manufacture. Its application would apparently promote economy both in manufacture and utilisation of gaseous fuel, especially for high temperature work.—H. J. H.

Coal; Gasification of — in rotary furnaces. E. Roser. *Stahl u. Eisen*, 1920, 40, 741–747.

MANY of the lighter hydrocarbons are destroyed in the high-temperature zone of a gas producer and in order to recover bituminous constituents, a rotary retort was constructed, similar to a rotary cement kiln, in which a semi-coke was produced which could be subsequently gasified. The tube of the kiln was 24 m. long and 2.5 m. diameter. From 1 ton (metric) of raw coal the average yield of water-free tar was 10%, with 3.0% of crude light oils, and 150 cub. m. of gas containing CO, 6.3%, unsaturated hydrocarbons 10.6, O₂ 2.0, CO 4.6, CH₄ 64.6, N₂ 11.9%, H₂ nil. The amount of semi-coke, after deducting that required for heating the retort, was 650 kg. The thermal efficiency of the process is given as about 90%. The coke can be briquetted very easily, only a small quantity of pitch being required. The total heating value of

the gas from 1 ton of gas coal by the new method is nearly one-sixth less than that of the gas obtained from the same coal in ordinary gas producers, but there are obtained as by-products 30 kg. of light oils, 30 kg. of motor oils, 35 kg. of pitch, and 20 kg. of heavy oils and paraffin.—A. G.

[Gas] mains, services and meters; Internal corrosion of —. J. G. Taplay. Report of Life of Gas-meters Joint Committee, *Inst. Gas Eng.*, June, 1920. *Gas J.*, 1920, 150, 583–587.

THE previous conclusions as to the nature of the corrosive processes (J., 1919, 524a) were confirmed by the results of the examination of 40 more samples of liquid and solid deposits taken from distribution systems. Oxides of iron found are corrosion products and do not originate from purifier boxes. Moisture is essential to all corrosion processes, carbonyl formation excepting, and hence a preliminary compression of the gas is recommended, with separation of liquid, followed by expansion to reduce moisture content below the dew point. Protective paints tried prevented corrosion, but were not permanent in presence of benzene. Galvanising was ineffective, but tinning would be satisfactory if commercially feasible. Iron coated with lead, or "Armco" metal (iron coated with an alloy of Pb 80:Sn 20%), or aluminium showed considerable resistance to the usual corrosive agents. The desirability of careful purification of gas especially from hydrocyanic acid is emphasised. The corrosive agents may be classified under six headings in order of destructive importance, *viz.*, hydrocyanic acid; carbon bisulphide with ammonia; oxygen; carbon dioxide; sulphur dioxide; carbon monoxide, forming carbonyls.

—H. J. H.

Gas; Relative efficiency in use of different grades of —. Third Report of Research Sub-Committee of Gas Investigation Committee of *Inst. Gas Eng.*, June, 1920. *Gas J.*, 1920, 150, 592–603.

THE report deals with tests of the variation of efficiency with quality of gas when used in the normal upright incandescence mantle burner and in the gas fire. The qualities were varied by the separate addition of nitrogen, carbon dioxide, air, and water-gas to a high-grade coal gas, so that the influence of a single diluent on a combustible mixture of invariant composition could be determined.

Lighting tests.—An arbitrary minimum standard of satisfactory behaviour was defined by the dual requirement of a maximum candle-power of 70 c.p. for the low-pressure upright mantle burner with a thermal efficiency of not less than 30 c.p. per 1000 B.Th.U. of gas burnt. With straight coal gas the air requirement was so large that only by careful regulation of well designed appliances was it possible to secure the aeration necessary to attain the best possible efficiency. As a result of the reduction of air requirement the addition of a limited quantity of inert constituents has not the deleterious effect that might be anticipated. When nitrogen alone was added to straight coal gas the fall in efficiency was negligible at comparable rates of combustion until 25% was present and until 35% of nitrogen was present the above standard was maintained. The addition of 10.5% of carbon dioxide sufficed to depress the efficiency below the standard. The effects of nitrogen and carbon dioxide were apparently additive and thus it is only for small percentages that an equivalence as inerts can be assumed. Each 1% of nitrogen depressed the illuminating power by 0.38–0.52 c.p., whereas 1% CO₂ caused a fall of 1 c.p. The dilution of coal gas with air up to 20–30% had no influence on the illuminating power and thermal efficiency of the mantle, and even up to 40% the standard was maintained. Thus in reckoning inert constituents, for this purpose no account need be taken

of oxygen and its atmospheric complement of nitrogen. In addition to flame temperature, size and shape of flame relative to the mantle are important. The effect of inerts at high dilution affects both factors and the benefits of large additions of water-gas due to a rise in flame temperature are nullified by the alteration to the shape of the flame. The proposed standard was, however, attained by all mixtures containing up to 50% "blue" water-gas.

Gas fire tests.—The alteration, if any, of the percentage of heat radiated (radiant efficiency) when gas mixtures, of quality varied as above, were supplied to a gas fire fuel was examined. The fire used though not of the most efficient type was chosen as being capable of regulation over the whole range of gas mixtures from 600 to 140 B.Th.U. per cub. ft. The radiant efficiency did not suffer by dilution of straight coal gas with air until the calorific value of the mixture fell below 350 B.Th.U. per cub. ft. gross. No measurable effect was produced by the addition of nitrogen up to 20% (C.V. 500 B.Th.U. gross). No measurable effect was produced by the 300 B.Th.U. The actual fall in radiant efficiency, however, was only from 44 to 40%. At 400 B.Th.U. the efficiency was still 42%, and any depression observed in these experiments was not greater than that resulting from the imperfect aeration of high-grade gas. Carbon dioxide as diluent exerted a greater effect than nitrogen, but need not be considered separately unless more than 5% is present. "Blue" water-gas when added to coal gas actually raised the radiant efficiency slightly.—H. J. H.

Flue-gases; Control of — in mixed firing. W. Ostwald. Stahl u. Eisen, 1920, 40, 516—547.

THE main factors in the interpretation of flue-gas analyses are the quantity of excess oxygen over that theoretically required and the composition of the fuel. "Free" oxygen may be high owing to incomplete combustion. If the ultimate analysis of the fuel is known, the above two factors, in conjunction with the determination of the carbon dioxide and excess oxygen, will suffice for control purposes; but if the composition of the fuel is not known, then a determination of the carbon monoxide content of the gas must be made. If the carbon dioxide content be "a"%, carbon monoxide "b," oxygen "c," and nitrogen "d"%, then the carbon in the flue-gases is $a+b$ (in the gaseous state), the total oxygen is $a+b/2+c$, whilst the excess oxygen is $c-b/2$. With deficiency of air the latter expression is negative, and the two main factors are calculated as excess air = $100(c-b/2)/(a+b)$, and percentage of half-burnt carbon = $100b/(a+b)$. Four examples are given to show the application of these formulæ to mixed firing.—A. G.

Flue-gas analyses; Graphical evaluation of —. H. Meyer. Stahl u. Eisen, 1920, 40, 605—610.

THE author elaborates his arguments on the basis of simple carbon-hydrogen mixtures, and interprets the analyses by means of a Gibbs triangular diagram, plotting percentage oxygen, percentage carbon monoxide, and percentage carbon dioxide, excess or deficiency of air being indicated. Three diagrams are given for solid, liquid, and gaseous fuels, the solid fuel being a coal containing C 75%, H 6%, and O 8%, the liquid fuel benzol, and the gaseous fuel blast-furnace gas of the composition CO, 5.4%; CO₂, 30.8%; H₂, 2.3%; CH₄, 1.2%, and N₂, 60.3%. (Cf. J., 1919, 492A.)—A. G.

Acetylene; Volumetric estimation of —. R. Willstätter and E. Maschmann. Ber., 1920, 53, 939—941.

METHODS of estimating acetylene, based on its reaction with silver nitrate and titration of the acid liberated, are unreliable, since it has proved

impossible to find conditions under which 1 mol. of the gas liberates 2 mols. of nitric acid, the amount of the latter actually set free varying from about 0.5 mol. in alcoholic to about 1 mol. in aqueous solution. The following method gives reliable results: Acetylene, in gaseous form or in solution, is agitated for a few minutes with Hoesvay's reagent (J., 1899, 1158), freshly prepared from copper nitrate, ammonia, and hydroxylamine hydrochloride, and the precipitated copper acetylide is collected on an asbestos filter and washed until the filtrate no longer decolorises N/10 permanganate, care being taken not to allow the precipitate to become dry. The copper acetylide is washed from the filter with about 25 c.c. of a solution of 100 g. of ferric sulphate and 200 g. of concentrated sulphuric acid per litre. The ferrous sulphate formed in accordance with the equation, $C_2Cu_2 + Fe_2(SO_4)_3 + H_2SO_4 = 2FeSO_4 + 2CuSO_4 + C_2H_2$, is estimated by titration with N/10 permanganate. The liberated acetylene does not introduce any complications.—H. W.

Acetylene; Quantitative estimation of — in coal gas and air by means of Hoesvay's reagent. H. Arnold, E. Möllney, and F. Zimmermann. Ber., 1920, 53, 1034—1039.

THE gas is shaken in a separating funnel of about 3 l. capacity with about 20 c.c. of the reagent (J., 1899, 1158) at intervals during 1 hr. The precipitate is collected, thoroughly washed with water containing hydroxylamine, and dissolved from the filter by a little concentrated nitric acid. The filtrate is evaporated to dryness and the residuum ignited and weighed as copper oxide. Test analyses performed with mixtures of acetylene and air show the method to be very accurate, and to be available even when not more than 0.04% C₂H₂ is present. At such dilutions, however, it is necessary to add about 5% of carbon dioxide to the mixture, since otherwise the results are low in consequence of atmospheric oxidation. The process can also be used for the estimation of acetylene in coal gas, from which hydrogen sulphide, if present, must be removed, e.g., by dry copper sulphate-pumice.—H. W.

Petroleum oils; Rapid determination of aromatic hydrocarbons in light —. W. Hess. Z. angew. Chem., 1920, 33, 147—148.

SIXTY c.c. of the mineral oil distillate and about 200 c.c. of 95% sulphuric acid are placed in a 500 c.c. flask having a wide, graduated neck, and 50 g. of a mixture of 70% nitric acid, 1 pt., and 95% sulphuric acid, 2 pts. by weight, are added, drop by drop, from a tapped funnel which passes through a ground-in stopper. The latter also carries a small side tube. During the addition of the nitrating acid the contents of the flask are kept below 10° C. The flask is then filled up to the zero mark with 95% sulphuric acid, a plain stopper is inserted, the contents of the flask mixed and allowed to separate. The unattacked mineral oil separates and the volume is noted; the nitrated aromatic hydrocarbons remain dissolved in the acid mixture. If the sample contains more than 33% of aromatic hydrocarbons, more than 50 g. of nitrating acid will be required, but a large excess should not be used.

—W. P. S.

See also pages (A) 511, *Benzene and toluene* (Dufton and Cobb). 515, *Ferrocyanide* (Grube and Dulk). 518, *Gas retorts*. 521, *Castor oil and petrol* (Atkins); *Fatty acids from paraffin* (Fischer and Schneider, also Grün).

PATENTS.

Briquette-carbonising furnaces. C. H. Smith. E.P. 125,381, 3.4.19. Conv., 3.1.18.

THE briquettes are carbonised in sloping retorts arranged vertically in tiers, which are separated laterally by heating flues or chambers, the air

supply to which is preheated by means of a recuperator. This is constructed so as to provide alternately arranged series of air ducts and waste gas conduits. Means are provided for collecting the products of carbonisation.—A. G.

Carbonisation and gasification plant or the like [and apparatus for synthetic manufacture of ammonia]. W. E. Davies. E.P. 143,602, 21.2.19.

For the withdrawal of gas or vapour from a retort a withdrawal structure is provided consisting of two perforated walls, the space between which is subdivided by partitions into parallel flues, communicating with an exhaustor which withdraws the gas under suction relative to the pressure in the retort. A rapid fall in pressure is obtained, thus greatly favouring conservation of the products by rapid cooling and withdrawal. The structure may be placed centrally in the retort, and the outside wall of the structure may also form a wall of the retort. The invention may be applied to plant for the production of synthetic ammonia.—A. G.

Coke oven. H. W. Buhler. U.S.P. (A) 1,341,257 and (B) 1,341,258, 25.5.20. Appl., 24.1. and 23.2.17.

(A) A SERIES of vertical combustion flues are arranged alternately with a series of vertical air flues, each member of one series being connected with a member of the other series, thereby forming a series of pairs of combustion and air flues. Connections are made between the upper ends of the members of each pair, and a recuperator is located directly under the apparatus, connections being established between the air passages and the lower ends of the air flues and between the heating passages and the lower ends of the combustion flues. (B) A central vertical burner tube, open at the top, is connected at the bottom with a source of combustible gas, an outer concentric burner having its upper end projecting beyond the inner tube, and being connected at the base with a source of practically inert diluent gas.—A. G.

Gas; Means to generate — from coal and/or the like. J. H. Cortesby and S. T. S. Castelli. E.P. 143,361, 12.4.19.

In apparatus for generating gas from powdered coal by the impingement of a Bunsen flame, and wherein a portion of the gas generated is used for feeding the flame, a common controlling handle is provided whereby the supplies of gas and air to the flame are adjusted simultaneously. If steam is used for the production of producer gas, branch passages connect the outlet pipe for the generated producer gas with the fuel inlet pipe at points between the fuel supply and the generating flame, the rich gas distilled from the falling coal dust being led off separately.—A. G.

Gas producers. W. B. Chapman, Assr. to Chapman Engineering Co. U.S.P. 1,341,044, 25.5.20. Appl., 5.8.15.

THE grate of the producer has a relatively large ash opening, through which the ash is discharged by a rotating mechanism. The grate is constructed of radiating overlapping plates with their overlapping parts in contact with each other, and with recesses between the overlapping parts to provide orifices for the admission of air. A beam sweeps over the grate to remove the ash.—A. G.

Gas producer. E. Dolensky. G.P. 310,174, 22.2.17.

THE lower portion of a gas producer is divided into two chambers by means of partition walls, a revolving grate being provided in each chamber. This arrangement facilitates the removal of ash and clinker, and, further, obviates the necessity of adjusting the cross-sectional area of the producer to meet the requirements for ash removal or of retaining a circular shape to enable the grates to be revolved.—W. J. W.

Coal gas; Purification of —. [Removal of sulphur compounds.] F. W. Berk and Co., Ltd., and J. J. Hood. E.P. 143,641, 23.2.19.

THE crude gas is brought into contact with a bed of high-grade bauxite or ignited magnesite between 200° and 600° C., whereby the sulphur compounds are dissociated, producing hydrogen sulphide, which can be removed in the usual way.—A. G.

Hydrocarbon oils; Apparatus for treating —. R. H. Brownlee. U.S.P. 1,325,927, 23.12.19. Appl., 28.1.18.

THE apparatus comprises two vertical series of drums arranged at opposite ends of a furnace, with inclined treating tubes connecting drums of opposite series, and means for introducing oil into one of the drums and a gas into the lowermost of the treating tubes.

Gasoline or light vapours; Art of and apparatus for cooling and condensing —. A. A. Wehr. U.S.P. 1,340,427, 18.5.20. Appl., 2.7.19.

THE vapour is compressed and part of it is suddenly expanded. The low temperature thus produced is made use of in condensing the remainder of the vapour.—A. E. D.

Distillation of oil; Apparatus for —. S. D. Faust, Assr. to International Gasolene Process Corp. U.S.P. 1,340,532, 18.5.20. Appl., 10.3.19.

OIL is conducted through a continuous coil built in two sections. The lower section contains an inner steam-heating coil, and the upper section contains a coil through which superheated steam is passed to superheat the oil and its distillates.—A. E. D.

Oil-cracking apparatus. W. B. Poole, Assr. to Poole Oil Cracking and Refining Co. U.S.P. 1,340,793, 18.5.20. Appl., 23.12.18.

A RETORT is heated in the lowest of a series of superposed heating chambers. Oil enters the upper end of the retort by a pipe extending through the series of chambers, and the products escape from the still by a pipe which also extends through the series of heating chambers.—A. E. D.

Distillation [of liquid hydrocarbons]; Method of —. E. E. Pershall. U.S.P. 1,341,437, 25.5.20. Appl., 20.2.18.

A LIQUID hydrocarbon is heated and is agitated by currents of live steam until coke is formed.—A. G.

Lubricants; Method of making —. C. H. Bierbaum. U.S.P. 1,340,316, 18.5.20. Appl., 6.10.14.

IMPURE flocculated graphite is mixed with water free from deflocculating impurities. Hydrocarbon oil is added, the mixture is agitated, and separated impurities are removed.—A. E. D.

Pulverulent fuel; Supplying — to furnaces. J. E. Muhlfield. E.P. 143,817, 24.2.19. *Pulverulent fuel; Feeding — to furnaces.* V. Z. Caracristi. E.P. 143,819, 24.2.19. *Pulverulent fuel; Burner for supplying — to furnaces. Feeding fuel or other materials in pulverised form.* V. Z. Caracristi and J. E. Muhlfield. E.P. 143,818 and 143,820, 24.2.19.

Coke ovens; Apparatus for the quenching and transporting of coke from —. R. W. Cuthbertson and T. Greensmith. E.P. 143,965, 3.3.19.

SEE U.S.P. 1,301,662, 1,303,779, and 1,301,663 of 1919; J., 1919, 471 A, 545 A.

Dye from petroleum. U.S.P. 1,323,263. See IV.

Coke-oven gases. E.P. 136,833. See VII.

Hydrogen. E.P. 142,882 and 143,064. See VII.

Ferrous alloy. U.S.P. 1,341,992. See X.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Wood charcoal; Properties of —. A. Pickles. Chem. News, 1920, 120, 301—302.

PROLONGED heating at 900° C. increases the absorptive efficiency of charcoal, an increased absorption of ammonia gas, for example, from 17 to 70 vols., being observed after heating for 9 and 21 hrs. respectively. Treatment of the product with dilute hydrochloric acid removes sulphur and ash and tends to increase the efficiency. Cooling the hot charcoal in air is not advantageous. As a decoloriser, properly prepared wood charcoal (birch charcoal) in fine powder is superior to animal charcoal. The more complex the dissolved compound the more readily is it removed from solution by charcoal, and, conversely, compounds simple in structure are negatively adsorbed, i.e., the solvent is removed by the charcoal. Compounds negatively adsorbed are highly ionised, the metallic element is strongly electropositive and univalent, and the constituent ions have a high mobility. Negative adsorption is particularly marked with the alkali halides, notably at temperatures above 40° C.—G. F. M.

Efficiency of different grades of gas. See IIa.

PATENTS.

Distilling carbonaceous material; Apparatus for —. W. P. Perry. U.S.P. 1,341,517, 25.5.20. Appl., 23.7.18.

CARBONACEOUS material is distilled within a vertical chamber, the inner walls of which are provided with abutments at different heights, which, in conjunction with the walls of the chamber, form spaces for the collection of gas. Openings are formed in the walls of the chamber above the grate for the insertion of rakers. Means are provided for removing accumulated gases from the gas-collecting spaces, and gas and steam may be introduced through openings below the grate. A vertical tube with outlets extending downwards is provided within the chamber for introducing gases generated in the material near the grate into the material under treatment at various levels.

—J. S. G. T.

Electrode for searchlights and method of making the same. E. A. Sperry. U.S.P. 1,342,398, 1.6.20. Appl., 15.7.16.

A CARBONACEOUS electrode comprises a shell provided with an axial channel with an undulatory surface. The channel is filled with a core united to the shell.—J. S. G. T.

Mercury-vapour lamp. M. J. Cornu. U.S.P. 1,343,037, 8.6.20. Appl., 5.1.18.

SEE E.P. 129,022 of 1917; J., 1919, 622 A.

III.—TAR AND TAR PRODUCTS.

Benzene and toluene; High temperature reactions of —. S. F. Dufton and J. W. Cobb. Inst. Gas Eng., June, 1920. Gas J., 1920, 150, 588—591.

BENZENE and toluene at varying partial pressures in atmospheres of nitrogen, hydrogen, or methane were passed over a column of hot coke in a silica tube so as to approximate to conditions obtaining in the carbonisation of coal. The time of contact was 10—15 secs. The gases leaving the tube were cooled to condense liquid and solid products, while the gas was collected for analysis. The principal reaction of benzene was condensation to diphenyl, and it was appreciable at 500° C. in an inert atmosphere. At 750° it was more extensive, and further

condensation to diphenylbenzene commenced. At 920° free carbon was produced. The diphenyl condensation was shown to be reversible $2C_6H_6 \rightleftharpoons C_{12}H_{10} + H_2$, thus accounting for the relative stability of benzene at 800° in an atmosphere of hydrogen or coal gas. In an inert atmosphere toluene exhibited a molecular condensation at 550° giving stilbene and an oil, possibly ditolyl. At 750° the reaction was more extensive and complex, naphthalene, diphenyl, and anthracene being identified in the product. These condensations were inhibited by the presence of an atmosphere of hydrogen which, however, caused decomposition of the toluene by the elimination of the side chain: $C_9H_7.CH_3 + H_2 \rightleftharpoons C_9H_8 + CH_4$. This reaction was shown to be reversible by experiments in an atmosphere of methane. Xylene and cresol in hydrogen yielded toluene and benzene, and thence the decomposition products of these.—H. J. H.

Naphthalene; Thermal analysis of the products of nitration of —. Pascal. Bull. Soc. Chim., 1920, 27, 388—408.

A STUDY of the melting-point curves of the various binary and ternary mixtures of nitronaphthalene, 1,5- and 1,8-dinitronaphthalenes, and 1,3,5-, 1,3,8-, 1,2,5-, and 1,4,5-trinitronaphthalenes. The results are applied to the analysis of the industrial products of nitration of naphthalene. In products containing a percentage of nitrogen corresponding to dinitronaphthalene, points of arrest in the cooling curve noted at 90° C. and 50° C. indicate the simultaneous presence of nitro- and trinitronaphthalene.—W. G.

See also pages (A) 507, *Nitrogen in tar* (Parker). 526, *Chlorobenzenes* (Meunier). 528, *Nitration of nitrotoluene and xylene* (Marqueyrol and others).

PATENTS.

Methyl derivatives of arylamino compounds; Production of —. A. Mailhe. E.P. 124,219, 11.3.19. Conv., 21.4.17.

Mono- and di-methylated arylamines are obtained by passing a mixture of the vapours of an arylamine and methyl alcohol over a suitable catalyst, preferably precipitated alumina, at 350°—400° C., condensing the product, and separating the methylated bases from the condensed liquid. The catalyst may be spread on trays arranged in a series of tubes built into a suitable furnace, and when exhausted it can be regenerated by calcination in air. The mixture of mono- and di-methylamines produced by this process may, if desired, be converted entirely into the dimethylated base by passing the vapour, mixed with a further quantity of methyl alcohol, a second time over the catalyst. Examples are given of the preparation of the mono- and di-methyl derivatives of aniline, toluidines, xylydines, and naphthylamines.—G. F. M.

IV.—COLOURING MATTERS AND DYES.

Photographic sensitizers. Barbier. See XXI.

PATENTS.

Dyestuff [; Production of a — from petroleum]. H. A. Frasch. U.S.P. 1,323,263, 2.12.19. Appl., 23.6.17.

THE product obtained by treating petroleum or its distillates with inorganic acids, such as sulphuric acid, e.g., sludge or sludge coke, after suitable treatment to remove acid, is exposed to the action of a halogen in the presence of an oxidising agent, e.g., to sodium hypochlorite solution or *aqua regia*. On neutralising, a precipitate ("petracin B") is formed, and is separated from the solution, which contains a soluble product ("petracin A"). The

oxy-celluloses behave as mixtures of celluloses and their degradation products. In the cases of hydro-celluloses from cotton or from wood, the degradation products are present in amounts varying from about 24 to 36%. Oxycellulose prepared by the action of bleaching powder contains about 60% of pure cellulose and 40% of apparently oxidised hydration products. Sulphite-celluloses contain cellulose-dextrins, either from the original material or formed during the process of boiling; these can be removed by boiling with lime. A variety of cellulose which is practically without reducing power is obtained by the action of boiling milk of lime on sulphite-celluloses or on hydro- or oxy-celluloses. Sulphite wood celluloses lose up to 50% of their pentosan content when boiled with milk of lime, and the methyl content is also diminished, sometimes to a considerable extent; the residue, however, still contains pentosans in very stable combination. Treatment with lime dissolves but little from soda ligno-celluloses, since any cellulose-dextrins which may be present are dissolved during the boiling with alkali. Products which are closely similar to cotton cellulose are readily obtained by the treatment of wood celluloses with boiling milk of lime, and this is particularly the case with bleached sulphite-celluloses. The material is woolly in appearance and touch, and, in the form of paper, has a greater absorptive capacity than the untreated material.—H. W.

Wood; Distribution of certain chemical constants of — over its proximate constituents. W. H. Dore. *J. Ind. Eng. Chem.*, 1920, 12, 472–476.

ABOUT one-half of the furfural-yielding groups in redwood (*Sequoia sempervirens*) are associated with the cellulose, but only a small amount with the lignin; the portion present as true pentosan is hydrolysed and removed during chlorination. The groups yielding acetic acid are associated partly with the cellulose, much less so with the lignin, whilst a small amount appears to be detached from either. The methoxy groups are associated wholly with the lignin.—W. P. S.

Woods; Proximate analysis of coniferous —. W. H. Dore. *J. Ind. Eng. Chem.*, 1920, 12, 476–479.

A SCHEME of analysis described previously (*J.*, 1919, 496 A) is modified in order that the sum of the various constituents shall account for practically all the material of coniferous woods. The determinations made are: loss on drying at 100° C., benzene extract (6 hrs.' extraction), alcohol extract (6 hrs.), cellulose, lignin, soluble pentosans, mannan, and galactan. The alcohol extract is determined on the material already extracted with benzene; cellulose is determined in the extracted material by Sieber and Walter's method (*J.*, 1918, 132 T), and lignin by digestion with 72% sulphuric acid. Soluble pentosans are determined in the chlorination liquor from the cellulose determination, and mannan and galactan on separate portions of the raw material, the mannan being determined by Schorger's method (*J.*, 1917, 1003). In the case of redwood, yellow pine, and sugar pine, the sum of the constituents determined by the above methods amounts to slightly over 100%.—W. P. S.

Nitrocelluloses. Duclaux and Wollman. *See* XXII.

PATENTS.

Vegetable fibres; Treatment of —. Gillett et Fils, Assees. of C. Schwartz. E.P. 136,568, 12.12.19. Conv., 13.12.18.

Raw, bleached, or mercerised vegetable fibres are treated at 0°–25° C. with a solution of cellulose in nitric acid of not less than 65% for not more than

4 hr. without tension, and then washed. The fibres thus coated with nitrated cellulose resemble wool, and may be directly dyed with basic dyes. Denitration is unnecessary. For low-quality fabrics suitable cellulose solutions are prepared by dissolving 30 g. of disintegrated and bleached wood pulp or cotton waste, or 100 g. of hydrocellulose in 1000 g. of 81% nitric acid at 15°–20° C. and adding 112 g. of water, or by treating 70 g. of mercerised cellulose for 2 hrs. with 750 g. of 75% nitric acid, then expressing 460 g. of the acid, adding 500 g. of 92% nitric acid, and diluting with 145 g. of water. For high-quality fabrics a solution of 50 g. of mercerised cellulose in 1000 g. of 83% nitric acid, subsequently diluted with 250 g. of water, is used. The treatment does not impair the strength of the fabric and print effects may be obtained.—A. J. H.

Textile fabrics; Waterproofing —. L. Meyer. G.P. 309,131, 24.5.18.

In waterproofing fabrics with soap, and subsequent treatment with fats and solutions of metal salts, soap is economised by using it in the form of a lather.—C. A. M.

Balloon fabrics; Increasing the impermeability of — to gas. W. Deutsch. G.P. 309,201, 14.11.16. Conv., 11.11.16.

BALLOON fabrics are impregnated with aqueous solutions or suspensions of soaps, fats, fatty acids, or fatty acid salts, and then, with or without subsequent treatment with aluminium acetate, dried in the air at the ordinary or higher temperatures.—C. A. M.

Fibrous material for surgical dressings. S. von Kapff. G.P. 318,517, 26.7.17.

THE material consists entirely or chiefly of wool which has been treated with chlorine. The wool may, for example, be treated for a few minutes in a cold bath of sulphuric acid of 1°–3° B. (sp. gr. 1.007–1.021), and then repeatedly for periods of 15 min. in a clear solution of bleaching powder (1 l. of solution of sp. gr. 1.043 per kg. of wool), and subsequently rinsed in water. The chlorinated wool can absorb much more moisture than cotton without feeling wet, and even when thoroughly saturated with liquid remains open, loose, and permeable to air.

Acidity of sulphite liquor; Apparatus for regulating the — and other like purposes. J. B. Crandon, Assr. to Crandon Manufacturing Co. U.S.P. 1,311,361, 25.5.20. Appl., 12.8.18.

AN electric current is passed through the solution and through an electric heating device, thereby operating a device for controlling the addition of a neutralising agent to the solution.—A. J. H.

Paper, cardboard, and the like; Production of a paste for the manufacture of —. C. O. Lottero. U.S.P. 1,311,991, 1.6.20. Appl., 31.8.18.

PAPER pulp is obtained from the roots of the lucerne (*Medicago sativa*) or similar plants by drying, sifting, washing, macerating for about 8 days in the presence of lime, grinding, and mixing.—A. J. H.

Paper, fabrics, etc.; Sizing and impregnating — with glue, casein, etc. Exportingenieur für Papier- u. Zellstofftechnik. G.P. (A) 291,228 and (n) 291,229, 3.7.15.

(A) AN addition of sodium hydroxide (2%) or of lactic acid (30% on the weight of the dry glue) is made to glue solution to increase its penetrative power. (n) Methylformamide (about 2% on the dry glue) is used to harden the glue or casein.—C. A. M.

Paper; Sizing of — by means of mixtures of colloidal substances with water-glass. M. Müller. G.P. 317,948, 20.7.17.

THE sizing agents are precipitated partly or completely by the addition of acids or acid salts, especially aluminium compounds, prior to the addition of the mixture to the paper stock. Substances which increase the stability and have filling or sizing properties, or filling materials or colouring matters in the form of emulsions, may also be introduced.—C. A. M.

Paper; Manufacture of dense, strong —. C. G. Schwalbe. G.P. 319,826, 27.4.18. Addn. to 303,498.

PASTY materials, obtained by treating waste paper or cellulose with chlorine and water, followed by grinding with water, are incorporated with paper pulp.—W. J. W.

See also pages (A) 517, *Alkali silicate* (G.P. 318,516); *Aluminium compounds* (G.P. 319,420 and 319,521). 519, *Acetylcellulose films* (G.P. 297,955). 524, *Grain* (G.P. 318,701). 529, *Pyroxylin solvent* (U.S.P. 1,341,710).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Tussar silk; Black dyeing of —. J. P. Srivastava. J. Sec. Dyers and Col., 1920, 36, 173—177.

IN the degumming of genuine Tussar silk the loss of weight with boiling water is 15—18% and by treatment with alkalis and acids about 20%. Silk degummed with boiling water alone gave the best dyeing results with all classes of dyestuffs. Direct, acid-chrome, and logwood colours gave satisfactory results. With acid dyes the lustre was impaired, whilst sulphur blacks gave very poor dyeings having no lustre.—A. J. H.

Dyeing "polished" cotton thread. A. Winter. Färber-Zeit., 1920, 31, 121—123.

THE yarn is treated before dyeing by the usual methods. Yarn dyed with natural dyes has a pleasing bloom and "handle" and soft tone. For black, logwood used with fustic on an iron or copper mordant is especially suitable. Direct black dyes can also be used with logwood, tannin, and iron acetate. The dyed cotton is finished by treatment with a paste made thus: 300 g. of "diastafor" is allowed to react with 20 kg. of potato starch and the action stopped at a suitable point by heating; 500 g. of wax, 250 g. of paraffin, 250 g. of tallow, and 300 g. of borax are added, the mixture heated, diluted to 250 l., and suitably coloured.—A. J. H.

PATENTS.

Dye jigs. J. Gaston and H. Rushton. E.P. 139,971, 10.6.19.

IN an ordinary dye jig a cloth-stretching roller, such as is used in calendering machines, is provided at each side of the trough and rotated in the desired direction against the cloth by means of a chain drive or equivalent gearing from the large roller at the opposite side.—L. L. L.

Scouring, washing, crabbing, and otherwise treating textile fabrics; Machine for open —. J. Yates and H. Wilkinsen. E.P. 143,431, 21.7.19.

THE fabric passes through the machine by way of overhead stretching and guide rollers, down an inclined bed, through a front trough, then through a rear trough, and afterwards between a constantly driven drum and an adjustable pressure roller situated over the rear trough, which is provided

with perforated steam and water pipes and contains the liquid with which the fabrics are treated.

—A. J. H.

Textiles, skins, and other materials; Process and apparatus for treatment [impregnation] of — [with liquids under vacuum]. P. M. Quas-Cohen, M. N. Drucquer, and L. A. Levy. E.P. 143,443, 5.8.19.

THE materials are passed through a seal consisting of the liquid with which they are to be treated, upwards through a column of the same liquid (the height depends on atmospheric pressure), into a vacuum chamber, and then downwards through the same column and seal.—A. J. H.

Silk; Process of dyeing raw —. E. L. Maupai. U.S.P. 1,332,675, 2.3.20. Appl., 23.5.19.

UNTWISTED silk threads are treated to harden the natural gum or ceraceous matter, then dyed, and the gum or ceraceous matter subsequently removed.—L. L. L.

Yarn-treating mechanism. W. J. Goings. U.S.P. 1,333,481, 9.3.20. Appl., 8.11.17.

LIQUID is forced through a tubular yarn rack with yarn supports, which conduct the liquid from the rack. Detachable means are provided to prevent the yarn from floating off the supports.—L. L. L.

Dyeing of fibres and fibre products; Process and apparatus for electrical treatment in the — and the dyestuffs and the product resulting therefrom. J. E. Bloom. U.S.P. 1,333,700, 16.3.20. Appl., 3.12.17.

FIBRES and fibrous material are treated in solutions or suspensions of dyestuffs in suitable liquid media, under the influence of an alternating current.—L. L. L.

Yarn-treating mechanism. W. J. Goings. E.P. 142,771, 8.3.20.

SEE U.S.P. 1,333,481; preceding.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Arsenic in sulphuric acid; Rapid method for determination of —. A. A. Kohr. J. Ind. Eng. Chem., 1920, 12, 580—581.

TO determine arsenious acid, 20 g. of acid is diluted with water, and methyl orange added. To the solution is added a saturated solution of sodium carbonate until only a very faint pink colour persists, after which 2 g. of powdered sodium bicarbonate is added. The solution is diluted to 250 c.c. with water and titrated with N/10 iodine and starch, a blank titration being made. For the estimation of quinquivalent arsenic 20 g. of acid is heated at 105°—110° C. for 1 hr., diluted with a small amount of water, and saturated sodium carbonate added in slight excess, phenolphthalein being used as indicator. The solution is boiled and filtered. To the filtrate are added 3 g. of powdered sodium bicarbonate, 150 c.c. of concentrated hydrochloric acid slowly with agitation, and 1 g. of potassium iodide, air being excluded. After agitation and standing for 5 mins. the liberated iodine is titrated with N/10 thiosulphate and starch. Copper salts interfere with the determination, but are usually negligible. If present in quantity, the amount of potassium iodide should be increased. (Cf. J.C.S., Aug.)—W. J. W.

Nitrogen oxides; Absorption of — by dilute nitric acid. E. K. Rideal. J. Ind. Eng. Chem., 1920, 12, 531—538. (Cf. Partington and Parker, J., 1919, 75 t.)

THE absorption of nitrogen peroxide by dilute nitric acid in presence of air follows a uni-mole-

cular law. In aqueous solutions, as in the weak acid towers, the process is one of physical solution and depends on the partial pressure of the nitrogen peroxide; it proceeds most rapidly at high temperatures. In strong acid solutions, on the other hand, the reaction is a chemical one, and the rate of absorption depends on the concentration of the gas, absorption proceeding most rapidly at low temperatures. The point of inversion, i.e., where absorption is independent of temperature, is at about 10% nitric acid concentration. The limitation of nitric acid strength to 61% in towers is due to reduction by nitrous anhydride, the equilibrium constant K at this stage being 0.815. By limiting the amount of water vapour, and agitation with oxygen, concentrations above 61% are obtainable, the condensed acid separating into two layers of different strengths. Strong nitric acid may be produced by suitable refrigeration of a mixture of nitrogen peroxide, water vapour, and oxygen.

—W. J. W.

Hydrochloric acid; Production of — from chlorine and water. H. D. Gibbs. *J. Ind. Eng. Chem.*, 1920, 12, 538—541.

Is the formation of hydrogen chloride from chlorine and water, with charcoal as a catalyst, at low temperatures, influential factors are temperature, relative concentrations of water and chlorine, and the nature of the charcoal. Curves are plotted for temperatures of 0°, 12°, 25°, and 37.5° C., and for ratios of water to chlorine of 0.1:1; 3.1:1; 6.2:1; and 12.4:1. With two varieties of charcoal, a maximum hydrogen chloride production of 31%, falling to 18%, in 800 mins. was obtained in one case, as compared with 26%, falling to 7%, in the other. (*Cf.* J.C.S., Aug.)—W. J. W.

Sodium sulphate; Manufacture of — without sulphuric acid. II. G. Pollitz. *Chem.-Zeit.*, 1920, 44, 413—414.

THE Hargreaves-Robinson process (J., 1919, 814A) is principally used in works which also manufacture bleaching powder. There is complete utilisation of the by-products from all the chemicals used, only calcium carbonate, which can again be brought into the circuit, being left. The extension of the process depends upon discovery of new methods of utilising hydrochloric acid in place of sulphuric acid. In a recent process phosphates are decomposed by hydrochloric acid, the hygroscopic properties (due to calcium chloride) of superphosphates thus manufactured being eliminated by the addition of a small amount of a sulphate.—C. A. M.

Ammonium sulphate; Absorption of moisture by, and caking of —. J. T. Sheard. *Gas World*, 1920, 72, 512—513.

AMMONIUM sulphate will absorb moisture from a damp atmosphere to an unlimited extent, whether it contains free acid or not. By exposure to dry air a sample containing 0.5% free acid and 3.5% moisture was dried to 0.3%, while the moisture in material containing 0.16% free acid was reduced from 0.4% to 0.1%. Both neutral and acid samples, which have been allowed to absorb moisture, cake when again dried. Results obtained after treatment with ammonia to remove pyridine (J., 1920, 332A) showed a difference as regards moisture absorption of degree only from that of untreated samples, and caking occurred after drying the treated samples. Neutralisation of ammonium sulphate with alkalis may precipitate traces of iron in a form unassimilable by soils.—W. J. W.

Ammonium phosphates; Solubility of mono- and di- —. G. H. Buchanan and G. B. Winner. *J. Ind. Eng. Chem.*, 1920, 12, 448—451.

MONOAMMONIUM phosphate: 100 grms. of a saturated solution between 3° and 90° C. contains

18.0+0.455t g. of the salt. Diammonium phosphate: 100 g. of a saturated solution between 10° and 70° C. contains 36.5+0.213t g. of the salt.

—W. P. S.

Sodium hypochlorite; Formation and decomposition of —. F. Giordani. *Rend. Accad. Sci. Fis. Mat. Napoli*, 1919, [iii.], 25, 138—153.

THE formation of hypochlorite from chlorine and sodium hydroxide is accompanied by dilatation, which must be allowed for in calculating the amount of sodium hydroxide required to give a hypochlorite solution of definite titre. Perfectly neutral sodium hypochlorite solution rapidly decomposes, and in practice it is most convenient to stop the action of the chlorine when the concentration of the free sodium hydroxide reaches 1.5%. (*Cf.* J.C.S., Aug.)—T. H. P.

Ferrocyanides; Recovery of — from spent oxide. G. Grube and B. Dulk. *Z. angew. Chem.*, 1920, 33, 141—144.

THE material investigated by the authors gave on analysis: 7.8% $K_4Fe(CN)_6 \cdot 3H_2O$; 0.25% NH_4CNS ; 0.71% NH_3 (free); 1.7% NH_3 (combined); 34.34% S; and 26.3% H_2O . By treatment with slaked lime and subsequent washing with water calcium ferrocyanide is obtained. The yield increases with the quantity of lime up to a maximum with 10%, beyond which it diminishes owing to the formation of an almost insoluble double salt $2CaFe_2O_4 \cdot Ca_4Fe(CN)_6 \cdot xH_2O$. The most favourable temperature for the reaction is 50° C. Below this the reaction velocity is too slight, while at higher temperatures thiocyanate is produced and the yield of ferrocyanide rapidly diminishes between 70° and 80° C. The lixiviated mass may retain 1—2% of insoluble ferrocyanide produced by interaction of ferric hydroxide, calcium hydroxide, and calcium ferrocyanide and the formation of the double salt referred to. This salt may be decomposed by means of hydrogen sulphide and the yield of ferrocyanide correspondingly increased. A similar advantageous effect is obtained by air blowing.

—W. J. W.

Chlorides; Titration of certain — with silver nitrate, using potassium chromate as indicator. H. W. Bolam. *Chem. News*, 1920, 120, 292.

BARIUM and other elements which yield chromates less soluble than silver chromate interfere with the titration of a chloride unless the quantity of potassium chromate added is more than sufficient to precipitate the barium etc. which may be present.

—W. P. S.

Arsenites of lithium and potassium. F. A. H. Schreinemakers and W. C. de Baat. *Rec. Trav. Chim.*, 1920, 39, 423—428.

LITHIUM forms one arsenite, $LiAsO_2$, and potassium forms two arsenites, K_2AsO_3 and $K_4As_2O_6 \cdot 12H_2O$, all of which are soluble in water without decomposition.—W. G.

Carbon monoxide; Electromotive activity of —. K. A. Hofmann. *Ber.*, 1920, 53, 914—921.

IN an open circuit carbon monoxide reacts with alkali hydroxide in the presence of copper, to yield carbonate, very little formate, and mere traces of oxalate and hydrogen; in a closed circuit the hydrogen is oxidised to water by the oxygen liberated at the opposite pole. (*Cf.* J.C.S., Aug.)

—H. W.

Catalysis of oryhydrogen gas by palladium mixed crystals. G. Tammann. *Z. anorg. Chem.*, 1920, 111, 90—96.

EXPERIMENTS were made with palladium-silver and palladium-gold alloys, containing from 0 to 100% Ag and Au respectively, to determine the influence of the composition of the alloy on the temperature

at which catalysis of a hydrogen-oxygen mixture (prepared by the electrolysis of caustic soda solution) commences and, on cooling, ceases. The catalyst in each case was in the form of thin wire, wound in a close spiral. With pure palladium catalysis started at 145° – 163° C., and ceased at 35° – 50° C. With increasing silver content, in the Pd-Ag series, both temperatures rose slowly up to 80% Ag, at which point there was a sudden rise to 370° – 380° C. (commencement of catalysis) and 270° – 276° C. (cessation of catalysis). In the Pd-Au series there was little change from 0 to 70% Au, then a rapid rise in the limiting temperatures from 70 to 100% Au. The temperature at which catalysis starts depends on the rate of distribution of heat through the catalyst and hence, to a marked degree, on the form of the catalyst. With a wire spiral catalysis begins at a lower temperature than with a compact mass of wire. (Cf. J.C.S., ii., 422.)
—E. H. R.

Bromine chloride; its combination with ethylene. M. Delépine and L. Ville. Comptes rend., 1920, 170, 1390–1392.

THE behaviour of a solution of chlorine in bromine towards ethylene, the main product being chlorobromethane, is given as strong evidence in favour of the existence of a bromine chloride.—W. G.

Sulphur; Detection of arsenic in —. H. S. and M. D. Davis. J. Ind. Eng. Chem., 1920, 12, 479–480.

A METHOD proposed by Schaeppi (extraction of arsenic sulphide from the sulphur by means of hot dilute ammonia and determining the arsenic indirectly by weighing the silver sulphide produced when the neutral solution is treated with silver nitrate) is untrustworthy, owing to the fact that sulphur is appreciably soluble in ammonia and yields a precipitate of silver sulphide. Reliable results are obtained when the sulphur is oxidised with bromine and nitric acid and the residue tested by the Gutzeit method.—W. P. S.

See also pages (A) 524, *Dextrose and hydrocyanic acid* (Bougault); *Phosphate-carbonate mixtures* (Windisch and Dietrich). 528, *Nitrates* (Faber and Stoddard). 529, *Conductimetric titrations* (Koltzoff). 530, *Nitrates* (Harvey).

PATENTS.

Sulphuric acid; Method of producing and purifying —. P. Haeseler, Assr. to The Liberty Laboratories, Inc. U.S.P. 1,341,462, 25.5.20. Appl., 25.9.18.

A SUBSTANCE containing sulphur dioxide is allowed to react with a reducible compound of selenium, containing oxygen, access of free oxygen being prevented.—W. J. W.

Sulphuric acid; Process of making —. E. L. Larison. U.S.P. 1,342,024, 1.6.20. Appl., 25.8.19.

GLOVER tower gases are passed through a series of packed towers in which the most favourable temperature is maintained by circulation of weak acid, whereby the sulphur dioxide is completely oxidised.
—W. J. W.

Hydrogen and nitrogen [for synthesis of ammonia]; Manufacture of mixtures of —. J. Harger. E.P. 142,874, 23.11.17.

COAL of high nitrogen content and containing little sulphur is mixed with a paste of iron oxide and calcium carbonate and coked in the upper portion of a double producer. Below this is a gas producer proper, fed with hot coke from the preliminary producer, into which steam, air, and oxygen are blown, the temperature being kept at 650° – 850° C. The gaseous mixture of hydrogen, nitrogen, carbon monoxide, and carbon dioxide is passed over limestone or magnesia to remove sul-

phur, mixed with steam, and passed over a catalyst, such as iron oxide, whereby the carbon monoxide is converted into dioxide, and carbon bisulphide into hydrogen sulphide, which is later removed by passing over heated iron oxide, traces of carbon monoxide being eliminated by addition of air and employment of a catalyst (J., 1919, 577 A). After compression of the mixed gases to condense steam they are blown into towers, in which they are treated with ammonia and water, the carbon dioxide being converted into ammonium bicarbonate. Ammonia and calcium sulphate may be used as absorption mediums. The gases are freed from oxygen in a tower, compressed to 100–150 atm., and the water separated utilised to remove traces of ammonia from the circulating gases in the synthetic ammonia plant. The mixed gases are washed, passed through caustic alkali solution, and then solid caustic alkali, heated, and conducted to the plant for production of synthetic ammonia.
—W. J. W.

Ammonia; Synthetic production of — [and estimation of ammonia in the gases]. E. K. Rideal and A. G. Tarrant. E.P. 143,341, 10.3.19.

THE gases are passed into an absorption apparatus, through which distilled water circulates at a fixed rate, by means of a control valve and a flow meter; the latter may consist of a constricted tube the ends of which are in communication with a liquid such as aniline, the rate of flow being indicated by an index scale. The ammonia solution from the absorber flows through an electrolytic cell with suitable electrodes in which its electrical conductivity is measured. The rates of flow of the gases and of the absorbent must be adjusted as necessary.
—W. J. W.

Sodium monochromate; Transforming — into bichromate or chromic acid. Soc. Ind. de Prod. Chim. E.P. 131,289, 6.8.19. Conv., 13.8.18. (Cf. J., 1919, 253 A.)

CARBON dioxide is passed into sodium monochromate solution at 30° C., the precipitated sodium bicarbonate is separated, and the filtrate is boiled and treated with slaked lime to convert the sodium bichromate into calcium chromate, which is separated, washed, centrifuged, and agitated with sodium bisulphate solution at 70° C. The sodium bichromate formed may be separated from the calcium sulphate and converted into chromic acid by means of sodium bisulphate or sulphuric acid.—W. J. W.

Ammonium and sodium sulphates; Recovering — from coke-oven and like gases. Soc. Ind. de Prod. Chim. E.P. 136,833, 27.9.18. Conv., 27.5.18.

AMMONIA is absorbed from the gases in a solution of sodium bisulphate to obtain a double sodium-ammonium sulphate, $\text{Na}_2\text{SO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 4\text{H}_2\text{O}$ (or a salt of about the same composition). The solution is heated to boiling to obtain anhydrous sodium sulphate, which is separated from the ammonium sulphate by hot filtration.—A. G.

Potassium sulphate; Manufacture of —. Fabr. de Prod. Chim. de Thann et de Mulhouse. E.P. 137,296, 29.12.19. Conv., 28.12.18.

POTASSIUM bisulphate and potassium chloride, ground so as to pass through a sieve of 50–80 meshes per linear inch, are intimately mixed and heated to 300° C., at which temperature the hydrogen chloride may be condensed free from sulphuric acid. Water may be added to the mixture before introducing it into the furnace. The product contains very little chloride. By using 3–5% excess of potassium bisulphate and heating the mass after the first calcining to a temperature of 700° C., with condensation of the sulphuric acid evolved, potassium sulphate entirely free from chloride is obtained.—W. J. W.

Sulphur dichloride; Production and utilisation of —. W. J. Pope and C. T. Heycock. E.P. 142,879, 17.5.18.

THE employment of 1% by weight of finely powdered absorbent charcoal as a catalyst greatly facilitates the reaction between chlorine and sulphur monochloride, and similarly, when sulphur dichloride is subjected to conditions which lead to its decomposition into chlorine and the monochloride, re-combination is facilitated by similar means.—G. F. M.

Sulphur chloride; Process for making —. R. E. Gegenheimer and M. Mauran, Assrs. to The Mathieson Alkali Works (Inc.). U.S.P. 1,341,423, 25.5.20. Appl., 28.12.18.

A DEEP bath of sulphur chloride in contact with molten sulphur is treated with free chlorine, and the temperature of the mixture and of the reaction products is regulated so that the sulphur-bearing constituent of the products consists almost solely of sulphur monochloride.—W. J. W.

Thorium; Recovering — from monazite sands. O. N. Berndt, Assr. to Lindsay Light Co. U.S.P. 1,323,735, 2.12.19. Appl., 27.6.17.

MONAZITE sand is heated with concentrated sulphuric acid first until decomposed, and then for a long time at 250°–300° C. until the thorium is converted into a crystalline compound, $\text{Th}(\text{PO}_4)_2 \cdot \text{SO}_4$, insoluble in dilute sulphuric acid. The other rare earths form soluble compounds.

Magnesia; Manufacture of —. E. E. Dutt, Assr. to P. C. Dutt. U.S.P. 1,325,932, 23.12.19. Appl., 18.10.18.

A MAGNESIUM compound containing calcium and iron is dissolved in hydrochloric acid, the solution treated with an alkaline-earth oxide or hydroxide to precipitate iron and other similar impurities, then evaporated to dryness, and the residual mixture of chlorides treated with steam at 450°–700° C., after which the calcium chloride is dissolved in water and separated from the magnesia.

Magnesium from dolomite; Process of separating —. A. M. Mitchell, Assr. to Mitchell and Grenelle, Inc. U.S.P. 1,340,905, 25.5.20. Appl. 20.1.19.

PULVERISED dolomite is mixed with an alkaline hydroxide which does not react to form a compound with the calcium.—W. E. F. P.

Potash reclaiming. H. W. Charlton. U.S.P. 1,341,110, 25.5.20. Appl., 18.8.19.

GREENSAND is heated with ferrous chloride.
—W. E. F. P.

Sulphur dioxide from gases; Method of and apparatus for recovering —. A. H. Eustis. U.S.P. 1,341,114 and 1,341,115, 25.5.20. Appl., 14.4. and 16.7.19.

MATERIAL containing sulphur dioxide is heated in an extractor in which a partial vacuum is maintained by means of a pump. The sulphur dioxide extracted is passed through the pump, the temperature of the latter being so regulated that the temperature of the gas in immediate contact with it is raised above the dew point with respect to the contained liquid.—W. E. F. P.

Sulphur dioxide solution; Method of obtaining a concentrated —. N. C. Christensen, Assr. to J. E. Barlow, and Big Indian Copper Co. U.S.P. 1,341,734, 1.6.20. Appl., 3.7.17.

IN processes in which a concentrated sulphur dioxide solution is deprived of part of the sulphur dioxide and caused to absorb it later, losses of sulphur dioxide are made good by causing the weakened solution to absorb the weak replacement

sulphur dioxide, the strong solution so obtained being caused to absorb the relatively strong sulphur dioxide gas previously taken from the solution.

—W. J. W.

Sodium silicate; Process of dissolving —. E. A. Taylor, Assr. to Grasselli Chemical Co. U.S.P. 1,341,288, 25.5.20. Appl., 10.10.17.

THE solvent is passed over and through an excess of the silicate and heated by means of an inert gas.

—W. E. F. P.

Alkali silicate solutions; Increasing the adhesive power of —. W. Dahse. G.P. 318,516, 23.8.18.

SOLUTIONS of alkali silicates become very viscous when treated with solutions of zinc and cadmium salts, and the product may be used as an adhesive for paper, cardboard, wood, leather, etc.—A. J. H.

Titanium-oxygen compounds; Manufacture of —. P. Farup, Assr. to Titan Co. A/S. U.S.P. 1,341,307, 25.5.20. Appl., 25.10.16. Renewed 29.9.19.

TITANIUM hydroxide containing iron and sulphuric acid is treated with sulphuric acid, heated to 300°–500° C. in presence of sodium chloride, and the sintered product lixiviated.—W. J. W.

Chlorine cell [; Electrolytic —]. T. A. Patterson, Assr. to Niagara Alkali Co. U.S.P. 1,341,516, 25.5.20. Appl., 17.2.20.

A CLOSED electrolytic cell is provided with a gas space, with all joints thereof liquid-sealed.

—J. S. G. T.

Electrolytic [chlorine] cell. P. McDorman, Assr. to Electro-Chemical Co. U.S.P. 1,342,336, 1.6.20. Appl., 19.6.19.

AN electrolytic cell comprises a hollow cathode mounted on a base, an anode within the cathode, and a porous diaphragm separating the two. The cell is provided with a head, and a gasket arranged between the head and the anode and cathode prevents the escape of chlorine and the admission of air to the cell.—J. S. G. T.

Electrolytic [hypochlorite] cell. J. Gerstle, Assr. to Electro-Chemical Co. U.S.P. 1,342,378, 1.6.20. Appl., 9.6.19.

AN electrolytic cell comprises separate chambers for the production of chlorine and caustic soda, and an additional chamber wherein chlorine and caustic soda are mixed at the rate of their production, out of contact with the electrodes.—J. S. G. T.

Composition of matter [zirconium compound] and method for producing the same. L. E. Barton, Assr. to Titanium Alloy Manufacturing Co. U.S.P. 1,342,084, 1.6.20. Appl., 4.9.19.

A MATERIAL consisting mainly of zirconium, but also containing nitrogen and (to a smaller extent) carbon, is prepared by heating zirconia-bearing ore with a carbonaceous reducing agent until the zirconium oxide is decomposed, and treating the melt with dilute sulphuric acid.—W. E. F. P.

Aluminium compounds for sizing papers and other purposes; Preparation of —. G. Muth. G.P. (a) 319,420, 14.5.19, and (n) 319,541, 26.11.18.

(A) ALUMINIUM compounds, such as bauxite, clay, etc., in an unburned state, are treated with sulphuric acid or alkali bisulphate solution, in presence of hydrofluoric acid or other fluorine compound. (B) The material may be treated with sulphuric acid or sodium bisulphate, after which the free acid is neutralised by a magnesium compound which may be derived from the precipitation of magnesium sulphate with slaked lime. The product may be used at once, or may be first brought to a solid state by evaporation.—W. J. W.

Hydrogen generators. A. R. Griggs. E.P. 142,882, 9.11.18.

In hydrogen generators comprising an ore chamber and an inner heating space a combustion chamber is constructed at the lower end of this space below the bottom level of the ore, air and reducing gas being led into the combustion chamber through inclined passages. The chamber may be of decreasing cross-sectional area towards the upper end. Efficient mixing of air and reducing gas to give the required degree of combustion at the desired position outside the inner heating space is thus ensured, and the inner metal lining of the ore chamber is protected from overheating and burning.

—W. J. W.

Hydrogen gas manufacture; Process and apparatus for preparing powerful oxidising gases for removing carbon and sulphur deposits in —

Blair, Campbell and McLean, Ltd., D. A. Blair, and J. L. Ferguson. E.P. 143,064, 11.6.19.

THE removal of carbon and sulphur which are deposited in hydrogen retorts and contaminate the gas is effected by periodically passing atmospheric air enriched with oxygen through the retorts. An oxygen-evolving mixture, such as potassium chlorate mixed with manganese dioxide, or with precipitated ferric oxide and kieselguhr, or bleaching powder mixed with cobalt oxide, cobalt sesquioxide, or nickel oxide, is placed in a series of retorts, built in a furnace and heated to 300°–350° C. in the former case and to 80°–85° C. in the latter case. Air is admitted to the bottom of the retorts. The oxidising gas and air are drawn from the top of the retorts into a main by means of an ejector and passed through an alkaline solution to an atmospheric condenser and holder.—W. J. W.

Bromine from brine; Process of extracting —

H. H. Dow, Assr. to The Dow Chemical Co. U.S.P. 1,323,173, 25.11.19. Appl., 24.4.16. Renewed 17.4.19.

BRINE containing bromide is over-oxidised (cf. U.S.P. 1,188,838, J., 1916, 890), and the products of oxidation containing bromine and chlorine are reduced, for example, by treatment with a bromide solution, so that the net result of the oxidation is equivalent to the bromine content.

—W. E. F. P.

Zinc solutions; Purification of — H. L. Sulman and S. Field, Assrs. to Metals Extraction Corp.

U.S.P. 1,341,601, 25.5.20. Appl., 12.5.19.

SEE E.P. 138,947 of 1918; J., 1920, 372 A.

Alkali-package. J. Armstrong, Assr. to Patent Corp., Ltd. U.S.P. 1,342,148, 1.6.20. Appl., 18.4.18.

SEE E.P. 114,896 of 1917; J., 1918, 302 A.

Acid-proof containers. E.P. 143,651. See I.

Synthetic ammonia. E.P. 143,602. See IIa.

Base-exchanging compound. E.P. 142,974. See XIXb.

VIII.—GLASS; CERAMICS.

Gas retorts; Casting of — Report of the Refractory Materials Research Committee. Inst. Gas Eng., June, 1920. Gas J., 1920, 150, 576–577.

THE casting slip was prepared from Stourbridge fireclay and Stourbridge greg with sodium carbonate or silicate (or a mixture of these) added in sufficient quantity to produce a smoothly flowing mixture without reducing the consistency to such

an extent as to allow the greg to settle. The amount of alkali mixture varies with the nature of the clay and size and proportion of greg, but on an average 0.3%, calculated on the dry weight of the clay used, is sufficient, and this has virtually no influence on the refractoriness of the clay. Solid cylinders, 20 in. in height, were cast from mixtures containing various amounts of greg (up to 175 parts of greg to 100 parts of clay). Vertical sections appeared uniform throughout, showing that the suspended greg did not tend to sink in the slip. Similar uniformity was found in castings made in small saggar moulds with a straight central core if cast under a suitable head. The shrinkage from mould to finished saggar (fired at cone 7) was 4.375%. The surface cracks formed on drying increased with the proportion and coarseness of the greg. They are due to the separation in the mould of an extremely thin layer of fireclay on the surface of the ware, which on drying contracts more than the gregged interior. They do not extend inwards on firing. Retorts of 5 ft. length and of cross-section with outside dimensions 2 ft. 4 in. by 2 ft. were also cast. Determinations of porosities and after-contractions showed the difference in texture at the opposite ends of the cast retort to be negligibly small. The plaster of Paris moulds are estimated to make 50 to 60 casts.—H. S. H.

Silica bricks; Comparative tests of machine-made and hand-made — W. Emery and L. Bradshaw. Gas J., 1920, 153, 577–579.

THE bricks were prepared together from the same materials ((crushed Dinas silica rock bonded with lime), one batch being moulded by hand and the other in a power press. Practically no difference was found in the refractoriness, the refractoriness under load, the reversible expansion at 1200° C., the after-expansion on heating to 1350° C., and the porosity. The apparent specific gravity of the machine-made brick was 1.85 compared with 1.8 for the hand-made brick. The resistance to crushing of the hand-made brick was 28% less than that of the machine-made; the spalling of the hand-moulded brick was greater than that of the machine-made whether determined by the loss in weight after alternately heating and quenching in water or from the decrease in mechanical strength after heating to 600° C. and allowing to cool. The abrasive action of a sand blast on the hand-made bricks was greater than on the machine-made bricks, while the former were less accurate in shape than the latter. Properly prepared machine-made silica bricks are not inferior in any of the laboratory tests tried to hand-made bricks, while in several important respects they are superior.—H. S. H.

Refractory materials; Specific heats of — at high temperatures. I. L. Bradshaw and W. Emery. Gas J., 1920, 150, 579–583.

THE mean specific heats of the materials were measured between 25° and 1000°, 1200°, and 1400° C. by dropping a weighed cylindrical test-piece and its supports, heated to the required temperature in an electric furnace of the Hirsch type into a calorimeter containing water and measuring the rise of temperature with a Beckmann thermometer. The heat communicated by the supports (made from a mixture of china clay and calcined alumina) was about 25% of that conveyed by the whole charge. The values obtained were as follows:—

Temperature. ° C.	Silica brick (96.6% SiO ₂).	Silica brick (84.65% SiO ₂).	Firebrick (59.2% SiO ₂ , 35.3% Al ₂ O ₃).	Zirconia (99% ZrO ₂).
600	0.226	0.228	0.228	0.137
1000	0.263	0.262	0.265	0.157
1200	0.282	0.283	0.284	0.167
1400	0.293	0.295	0.297	0.175

The specific heats of all the materials examined are much greater at high temperatures than at ordinary temperatures.—H. S. H.

PATENTS.

Glass-annealing furnace. M. A. Smith, Assr. to McKee Glass Co. U.S.P. 1,312,233, 1.6.20. Appl., 15.5.19.

A GLASS-ANNEALING furnace is provided with flues for air and gas in the sides and top, communicating with a series of checker-work chambers at the bottom of the furnace and with a chimney.

—A. B. S.

Reinforced glass and process of making same. Manufacture of reinforced glass. L. F. Mascart, Assr. to Soc. du Verre Triplex. U.S.P. (A) 1,312,267 and (B) 1,312,268, 1.6.20. Appl., (A) 21.3.14, (B) 12.9.18.

REINFORCED glass is made by applying to a sheet of glass successively a solution of gelatin and one of cellulosic material (cellulose ester), one or both of these solutions containing a solvent of both gelatin and cellulosic material.—A. B. S.

Acetylcellulose films on glass. Deut. Gasglublicht A.-G. (Auerger). G.P. 297,955, 4.2.16.

GLASS coated with acetylcellulose, by a short immersion in feebly alkaline or better in aqueous methyl alcohol solutions, is not obscured by condensed moisture.—A. J. H.

Glass; Composition for articles for moulding — and method of producing such composition. A. M. Glass. From B. C. Gilligan and W. B. Duck. E.P. 143,792, 5.11.19.

SEE U.S.P. 1,319,151 of 1919; J., 1919, 917 A.

Tunnel kilns [for enamelling]. G. H. Benjamin. E.P. 143,974, 6.3.19.

SEE U.S.P. 1,311,487 of 1919; J., 1919, 723 A.

Kiln. F. Fidler and J. G. Maxwell. U.S.P. 1,341,550, 25.5.20. Appl., 27.12.19.

SEE E.P. 111,124 of 1919; J., 1920, 408 A.

Porcelain. C. Dantszen, Assr. to General Electric Co. U.S.P. 1,313,040, 8.6.20. Appl., 3.12.18.

SEE E.P. 139,315 of 1919; J., 1920, 297 A.

IX.—BUILDING MATERIALS.

PATENTS.

Wood waste and kindred substances; Treatment of —. H. Gallinowsky, Assr. to A. Vizard. U.S.P. 1,326,891, 30.12.19. Appl., 2.6.19.

A BUILDING material is produced from sawdust or the like by treating it with hydrochloric acid, squeezing out the excess of acid, and then mixing with calcined magnesia.

Refractory cement. S. A. McMinn. U.S.P. 1,341,510, 25.5.20. Appl., 9.3.20.

SEE E.P. 137,419 of 1919; J., 1920, 192 A.

Artificial stones or slabs of marble-like appearance; Production of —. H. Grönroos. U.S.P. 1,341,979, 1.6.20. Appl., 20.6.19.

SEE E.P. 138,799 of 1919; J., 1920, 298 A.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Nickel steels; Action of metallurgical additions on the anomaly of the dilatation of —. C. E. Guillaume. Comptes rend., 1920, 170, 1433—1435.

THE effect of additions of manganese (up to about

8%), chromium (up to 10%), carbon (up to 0.6%), and copper (5%) on the dilatation of nickel steels was studied. The additions studied diminish, in every case, the intensity of the anomaly of dilatation in the region of its minimum value. In the case of manganese, carbon, and copper, within a limited region of nickel content, ternary alloys are obtained which have a slightly lower coefficient of dilatation than the binary alloys with the same nickel content. In the case of carbon, if the other constituents are known, measurement of the dilatation of the steel gives a means of fixing the carbon content to within about one ten-thousandth.

—W. G.

Brass; Corrosion of — in dilute electrolytes. J. H. Reedy and B. Feuer. J. Ind. Eng. Chem., 1920, 12, 511—517.

SELECTIVE or electrolytic corrosion is marked by pitting or perforation. The effect of various ions has been investigated experimentally by current potential curves. Marked corrosion is indicated with hydrogen chloride solution; metallic chlorides are less active. Ammonium and calcium chloride solutions are more corrosive than potassium or sodium chlorides. High concentration and elevated temperatures stimulate corrosion, while dissolved oxygen has no effect. In electrolytic mixtures the influence of the various ions is additive. The ions play no specific part in promoting corrosion except in cases where they form complexes of low ionisation, and in the case of the hydrogen ion. Complete corrosion of brass, i.e., of all its components, only takes place in presence of oxygen and high concentrations of solutions and elevated temperatures, which reduce the solubility of oxygen, therefore retard or inhibit corrosion. The influence of the ions is of a specific nature, and may be related to their effect on the solubility of oxygen. Solutions containing chlorides cause marked corrosion, and the ammonium group has a very positive effect. The speed of corrosion is affected by stirring and by the nature of the surface.—W. J. W.

Mercury ores; Assaying —. R. G. Place. Eng. and Min. J., 1920, 109, 1313.

THREE methods are described. In the first, 0.05—0.5 g. of ore, according to richness, is intimately mixed with 0.5 g. of lime in a small mortar, and the mixture transferred to a hard glass tube, bent so that the lower 3 in., in which the charge is placed, is parallel to the upper 6 in. The tube is held in the flame with the mouth slightly downwards, and the lower end is heated to redness for 10 min. The tube is fused off at the bend, a few c.c. of 1% cyanide solution and 2—3 gold balls each weighing 50—80 mg. are put in and the tube gently shaken. The balls are then replaced by fresh ones, this operation being repeated until all the mercury has amalgamated. The balls are washed, dried on blotting paper, then in a desiccator for 10 min., weighed, ignited to volatilise the mercury, and weighed again. The second method is a modification of Eshka's, in which the gold plate is cupped to fit the crucible, the cup being filled with water, which is kept cool by a casserole of cold water placed over it. The third method is a variation of Krickhaus' volumetric method, in which the ore is dissolved in *aqua regia*, the mercury precipitated by stannous chloride, washed by decantation, and dissolved in 4 c.c. of nitric acid. The solution is treated with permanganate till a permanent pink tinge is obtained; this is just discharged with 3% hydrogen peroxide, 5 c.c. of ferric alum indicator added, and the liquid titrated with thiocyanate.—A. R. P.

Film colours of metals. G. Tammann. Z. anorg. Chem., 1920, 111, 78—89.

WHEN a metallic surface of silver, copper, lead, or thallium is exposed to the action of iodine vapour a

thin film of iodide is formed. The rate of increase of thickness of the film was measured by observing the change of colour during exposure of a silver surface to air charged with iodine vapour, and applying well-known laws of optics. The increase in thickness of the film at constant temperature follows a parabolic law, and appears to depend on the rate of diffusion of iodine through the layer of iodide formed. The thinnest film of silver iodide which could be detected corresponded with a thickness of 63×10^{-6} mm., or approximately 90 atomic layers, the corresponding colour being bright yellow. The development of film colours on steel when heated in air appears, from a limited number of observations, to follow the same parabolic law, $y^2 = 2pt$, where y is the film thickness, t the time, and p a constant.—E. H. R.

Corrosion of gas mains etc. Taplay. See IIa.

Aluminium. Faber and Stoddard. See XXII.

PATENTS.

Iron and molybdenum; Production of alloys of —. Deutsche Molybdaen-Werke G.m.b.H. E.P. 131,896, 27.8.19. Conv., 25.11.16.

FERRO-MOLYBDENUM or molybdenum steel is produced by the interaction of alkaline-earth salts of molybdic acid with molten iron in the presence of carbon or with steel at about 1700°C .—T. H. Bu.

Iron and steel; Cementation of —. M. L. M. Luttenschlager. E.P. 143,746, 2.7.19.

IRON or steel is heated to 700° — 800°C . in a closed muffle containing a mixture of 1 pt. of potassium ferriocyanide, 1 pt. of potassium chromate or bichromate, 2 pts. of potassium ferrocyanide, and 20—25 pts. of wood charcoal, and then hardened in a solution of sodium chloride and caustic soda, with or without ammonium chloride.—T. H. Bu.

Acid-resisting alloy. A. W. Clement, Assr. to The Cleveland Brass Manufacturing Co. U.S.P. 1,333,706, 16.3.20. Appl., 26.7.17.

THE alloy contains Cr 60, Fe 39.2—39.7, and C 0.3—0.8%, the carbon being present as a carbide of chromium and iron.—L. L. L.

Ferrous alloy; Corrosion-resisting —. S. McClure. U.S.P. 1,341,992, 1.6.20. Appl., 31.1.19.

AN iron-copper alloy containing more than 0.5% Cu, but not sufficient to prevent the alloy from being rolled into sheets, is specially suitable for parts of plant exposed to products of combustion of fuel.

—A. J. H.

Rust-proofing compound. W. H. Allen. U.S.P. 1,341,100, 25.5.20. Appl., 18.11.18.

A RUST-proofing compound consists of ferric oxide (1 pt.), ferrous oxide (3 pts.), and phosphoric acid (equivalent of 60 pts. of the commercially pure acid).—A. de W.

Sherardising; Processes and apparatus for —. S. O. Cowper-Coles. E.P. 143,674, 26.3.19.

A ROTARY drum, which is maintained at a constant temperature in a suitable heating apparatus, is divided diametrically into two communicating chambers. When one chamber is being charged with the articles to be coated, the other chamber containing the hot zinc dust is cut off from access of air. After charging, the drum is closed, given a half turn, and the chambers put into communication with each other, so that the hot dust falls on to the articles.—T. H. Bu.

Flotation method. G. D. Van Arsdale and C. G. Maier. U.S.P. 1,325,817, 23.12.19. Appl., 25.9.17.

THE ore pulp is treated with an emulsifying agent (oil), and a true emulsion produced, for example,

by agitation after addition of caustic soda. The mixture is then de-emulsified, e.g., by addition of sodium sulphide, and subjected to flotation. A much better recovery of copper is obtained in this way from certain clayey ores such as Arizona porphyries.

Tin and other ores; Smelting —. J. H. Gray. U.S.P. 1,340,951, 25.5.20. Appl., 18.3.19.

ORES of low volatile metals, e.g., tin, are smelted on the hearth of an electrically-heated furnace in which non-oxidising conditions are maintained, the outlet flues containing material adapted to trap the metal in the fumes.—T. H. Bu.

Solder for aluminium. R. A. Jacobsen. U.S.P. 1,341,508, 25.5.20. Appl., 2.1.19.

AN aluminium solder is composed of Sn 67.60, Zn 16.90, Bi 2.82, Al 1.41, and phosphorus tin 11.27%. —J. S. G. T.

Aluminium; Process for the autogenous welding of —. Chem. Fabr. Griesheim-Elektron. G.P. 319,684, 2.5.17.

SOLUBLE fluorides (e.g., sodium or potassium-sodium fluoride) are added to the alkali halide used as a flux; the slag produced is then readily removed by washing with water. (Cf. G.P. 315,231; J., 1920, 162 A.)—A. R. P.

Platinum and similar metals; Process of extracting from their sands and ores. R. Thayer. U.S.P. 1,341,686, 1.6.20. Appl., 26.6.19.

A CHARGE of ore is mixed with a halogen compound of an alkali metal and heated, and the volatilised metal is collected.—T. H. Bu.

Sintering; Method of preparing fine ores for — and the product thereof. F. A. Eustis. U.S.P. 1,341,890, 1.6.20. Appl., 21.5.19.

THE ore is made into a filter cake, and finely divided fuel is applied to the surface of the cake.—B. M. V.

Light metals; Process and apparatus for the recovery of — from turnings, residues, and ashes. K. Hess. G.P. 318,304, 4.4.18.

THE material is added gradually to a bath of a molten salt contained on the hearth of a furnace, through a feeding device which just touches the surface of the bath, the bath being well stirred by means of a rake after each addition. Openings are provided in the furnace through which the impurities which accumulate on the surface of the bath may be removed. When the salt is saturated with impurities, the charge is poured by tilting the furnace.—A. R. P.

Magnetic separators. F. Uhlig. E.P. 144,221, 28.1.20. Conv., 10.10.19.

Steel; Process for producing —. A. Vögler, Assr. to The Chemical Foundation, Inc. U.S.P. 1,342,701, 8.6.20. Appl., 24.1.14.

SEE G.P. 280,044 of 1913; J., 1915, 363.

Electric welding of iron or steel or alloys thereof. A. C. Hyde. U.S.P. 1,341,558, 25.5.20. Appl., 25.11.19.

SEE E.P. 128,463 of 1918; J., 1919, 640 A.

Solder for aluminium and process for making same.. A. M. Ayala. E.P. 123,993, 4.3.19. Conv., 4.1.19.

SEE U.S.P. 1,333,237 of 1920; J., 1920, 372 A.

Malleableising cast metal; Method of —. W. G. Webster. E.P. 143,989, 12.3.19.

SEE U.S.P. 1,298,607 of 1919; J., 1919, 423 A.

Alloys. A. Cohn. E.P. 144,119, 4.7.19.

SEE U.S.P. 1,323,833-4 of 1919; J., 1920, 118 A.

Furnace lining. U.S.P. 1,341,299. See I.

XI.—ELECTRO-CHEMISTRY.

Carbon monoxide. Hofmann. See VII.

PATENTS.

Electric furnaces and the like; [Temperature] control for —. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 143,737, 19.6.19.

Two temperature controlling devices are used to regulate the power supply to the heating element of the furnace in such a manner that one of two predetermined temperatures may be maintained. The heating element is quickly raised to and maintained at the higher of these temperatures until the furnace charge reaches a predetermined temperature. The temperature control is then automatically switched over from one device to the other and the temperature of the heating element reduced to and maintained at the lower predetermined temperature.—J. S. G. T.

See also pages (A) 513, *Sulphite liquor* (U.S.P. 1,311,361). 514, *Dyeing* (U.S.P. 1,333,700). 517, *Chlorine cells* (U.S.P. 1,341,516 and 1,342,336). *Electrolytic cell* (U.S.P. 1,342,378). 525, *Meat* (E.P. 143,669). 527, *Acetic acid* (E.P. 121,195). *Ethyl acetate* (E.P. 131,600). 529, *Loose compounds* (U.S. 1,333,701).

XII.—FATS; OILS; WAXES.

Cottonseed oil; Chemical composition of —. G. S. Jamieson and W. F. Baughman. J. Amer. Chem. Soc., 1920, 42, 1197—1204.

REFINED oil from Sea Island cottonseed consists of the glycerides of myristic (0.3%), palmitic (20%), stearic (2%), arachidic (0.6%), oleic (35.2%), and linolic acids (41.7%). By treatment of the lead salts with ether, a fairly complete separation of the saturated from the unsaturated acids was achieved. By fractionation of the methyl esters of the saturated acids under reduced pressure, and crystallisation of the products of hydrolysis of each fraction, the saturated acids were each isolated and identified.—J. K.

Castor oil and petrol from various sources; Solubility relations of —. W. R. G. Atkins. J. Inst. Petrol. Techn., 1920, 6, 223—228.

SAMPLES of East Indian, Egyptian, and European petrol were miscible with castor oil in all proportions. Fractions distilled at 52° and 90° C. gave critical solution points of 32.2° and 31.6° C., whilst the fraction 104°—144° C. dissolved in all proportions in castor oil. Normal paraffins differ but little in their critical temperatures of solution (47.3°—47.9° C.), whilst benzene, toluene, and pentamethylene are miscible with castor oil in all proportions. The presence of aromatic and naphthene compounds lowers the miscibility temperatures of the paraffins. Variations in the castor oil also affect the critical temperature of solution.—C. A. M.

Fatty acids obtained by the oxidation of paraffins. F. Fischer and W. Schneider. Ber., 1920, 53, 922—925.

PURE paraffin or crude expressed paraffin was heated with dilute sodium carbonate solution in a steel autoclave at about 170° C., whilst simultaneously compressed air was pumped through the mixture. The paraffins were oxidised more rapidly under these conditions than at atmospheric pressure, and the yield of fatty acids was as high as 90% of the paraffin used. The rate of reaction varies with the temperature, and is proportional to the pressure of the air. Iron, manganese, and

copper had approximately equal catalytic effects, but other substances investigated were less active. The highest yields per given time were best obtained by working to secure complete neutralisation of the sodium carbonate solution rather than complete utilisation of the paraffin. The acids formed are true monobasic fatty acids of sp. gr. less than 1 and completely soluble in light petroleum; unlike the commoner members of this series, however, they contain an uneven number of carbon atoms. The following have been definitely identified:— $C_{15}H_{31}O_2$, m.p. 65°—66° C.; $C_{17}H_{33}O_2$, m.p. 58°—59° C.; $C_{19}H_{37}O_2$, m.p. 50°—51° C.; $C_{21}H_{41}O_2$, m.p. 38° C. Examination of a patented process for the oxidation of paraffin by blowing air through the substance heated at 135°—145° C. in an iron vessel in the absence of water showed that the actual products are the acid anhydrides which can be separated from unaltered paraffin by means of acetone.—H. W.

Paraffins; Oxidation of — [to fatty acids etc.]. A. Grün. Ber., 1920, 53, 987—996. (Cf. Kelber, J., 1920, 220 A.)

PURE paraffins, or mixtures such as vaseline or petroleum, are readily oxidised under suitable conditions by air or even by gaseous mixtures containing as little as 1% of oxygen. The statements by various authors that the process can be catalytically accelerated by oxygen-carriers are, in general, erroneous and depend, in part at any rate, on insufficiently extended analysis of the products. Thus, a series of experiments carried out with a paraffin (m.p. 52° C.) at 160° C. showed that metallic oxides such as lime and barium oxide, salts with an alkaline reaction, and certain neutral substances such as carbon and fuller's earth are particularly harmful, whilst acidic substances, such as stearic acid, are, according to the properties of the hydrocarbon used, either helpful or at any rate not markedly inhibitive. If the conditions of reaction are not carefully chosen, one carbon atom after another of the intermediate products can be oxidised away, and the whole series of acids from those of high molecular weight to the volatile acids and ultimately formic acid, or carbon dioxide may be obtained. Particularly when oxygen is used, the action is likely to lead to violent explosions due to the intermediate formation of peroxides. The products of oxidation differ quantitatively and qualitatively according to the conditions of the experiment. When the oxidation is not too vigorous, the product consists of mixtures of waxes with more or less free acid, alcohols, and some unsaponifiable matter. The composition of the mixture of acids depends on factors such as temperature, duration of action, etc., and the product may consist mainly of the series C_{16} to C_{22} or higher or of such in which C_{18} to C_{24} or C_{26} predominate. Hydroxy fatty acids are also formed and are present partly in the form of esters or estolides, whilst unsaturated acids, probably derived from the hydroxy-acids by loss of water, are also found. The unsaponifiable matter consists to a small extent of unchanged paraffin, but chiefly of higher alcohols together with other compounds, probably aldehydes, but possibly also containing ketones. (Cf. J.C.S., Aug.)—H. W.

PATENTS.

Oils, fats, and the like; Treatment of —. G. Calvert. E.P. 143,321, 21.2.19.

OILS or fats are agitated by spraying etc. with glycerin at about 140° C. until free fatty acids and other inedible constituents are esterified into edible glycerides. The treatment may be carried out under pressure or in a current of dry non-injurious gas to remove water, or in the presence

of a catalyst. The oil may also be simultaneously deodorised or hydrogenated.—C. A. M.

Grease; Apparatus for extracting — from organic matter. K. A. Stahlgren and T. L. Shannon. U.S.P. 1,341,523, 25.5.20. Appl., 11.3.19.

GREASE is extracted from organic substances by means of a solvent in a rotary drum provided with an agitator (which also serves as a means of heating) adjacent to the walls of the drum and which is rotated in an opposite direction to the drum.

—C. A. M.

Oils; Manufacture of edible and technical —. Byk-Guldenwerke. G.P. 317,717, 18.5.18.

ALKYL esters of fatty acids are polymerised, with or without the simultaneous action of oxygen or active light rays, or fatty acids, which have been thickened by oxygen or active rays, are converted into alkyl esters. The ethyl ester of linseed oil fatty acids when heated to 300° C. yields a product with iodine value about 100 and a viscosity between that of olive and castor oils.—C. A. M.

Fats; Process of extracting — from moist, granular or pulverulent substances. A. Lorenz. G.P. 319,664, 25.7.17.

THE material is extracted in a closed vessel containing a perforated false bottom by means of a solvent introduced from below. The false bottom rests on a central support, which serves as distributor of the solvent, and is perforated with large holes opening obliquely downwards. The bearing for the axle of the agitator is also constructed in the central support, and is cooled by the cold solvent.—C. A. M.

Copra-drying apparatus. J. D. McCord, Assr. to San Pablo Oil Mills, Ltd. U.S.P. 1,341,655, 1.6.20. Appl., 31.10.17. Renewed 2.1.20.

XIII.—PAINTS ; PIGMENTS ; VARNISHES ; RESINS.

PATENTS.

Resins; Process for purifying —. B. T. Brooks, Assr. to The Commercial Research Co. U.S.P. 1,340,873, 25.5.20. Appl., 14.5.18.

RESINOUS material is subjected to the action of the chlorhydrin of a gaseous elefine, the liquid portion separated from any solids accompanying it, and then evaporated to recover resinous material, the distillate being condensed. Steam is passed through the solid material to extract any liquids remaining in it, the extract is concentrated and united with the condensed distillate, and the resultant liquid employed for treatment of a fresh charge of resinous material.—A. de W.

Varnish; Process of manufacturing —. C. H. Bennett. U.S.P. 1,341,103, 25.5.20. Appl., 20.1.19.

A QUANTITY of varnish "body" is cooked remote from a closed blending chamber, then transferred to the blending chamber, and a quantity of solvent introduced. The solvent vapours are led off from the blending chamber by means of vapour pressure, condensed, and returned to the chamber (*cf.* U.S.P. 1,298,159; J., 1919, 428 A).—A. de W.

[*Resinous*] moulded products; *Process of producing —.* L. T. Richardson, Assr. to The Cutler-Hammer Mfg. Co. U.S.P. 1,342,067, 1.6.20. Appl., 6.6.18.

A PHENOLIC substance is mixed with a non-volatile methylene compound in proportions to afford about

one phenol group to one methylene group, and the mixture is heated under anhydrous conditions to form an initial condensation product in which less than half the phenolic substance has combined. This is mixed with a filler, the mass compacted, and the condensation reaction completed by further heating.—B. M. V.

Paint and process of making the same. J. Wingen. U.S.P. 1,341,250, 25.5.20. Appl., 12.2.19.

SEE E.P. 133,852 of 1919; J., 1919, 916 A.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Vulcanisation and depolymerisation. E. Seidl. Gummi-Zeit., 1920, 34, 797—798.

THE beneficial effect accruing to rubber by vulcanisation by the hot process is accompanied by a less marked adverse "depolymerisation" effect, which is masked by the former. Rise in temperature, however, increases the rate of the former change more than that of the latter, so that by vulcanisation at a higher temperature to the same percentage of combined sulphur, the proportion of the depolymerisation effect relative to the beneficial effect is decreased and the product possesses greater strength.—D. F. T.

Rubber membrane; Process of diffusion through a —. H. A. Daynes. Proc. Roy. Soc., 1920, A, 97, 286—307.

THE passage of a gas through a rubber membrane is a simple diffusion process in which the surface resistance is practically negligible. The mean diffusion constant is 11.4×10^{-6} cm. per sec. at 20° C. The absorption coefficient is 0.035 (mean), and this has a low temperature coefficient, 0.5% per 1° C. (*Cf.* J.C.S., Aug.)—J. F. S.

PATENT.

Rubber compounds; Manufacture of —. H. Wade. From Goodyear Tire and Rubber Co. E.P. 143,610, 24.2.19.

SEE U.S.P. 1,301,693 of 1919; J., 1919, 472 A. Other ingredients than barium sulphate can be incorporated in rubber mixings in a similar manner.

XV.—LEATHER; BONE; HORN; GLUE.

Tanning materials; True tanning value of vegetable —. J. A. Wilson and E. J. Kern. J. Ind. Eng. Chem., 1920, 12, 465—469.

TANNIN is defined as that portion of the water-soluble constituents of the material which will precipitate gelatin from solution and which will form compounds with hide fibre which are resistant to washing. To determine the true tanning value of vegetable substances 12 g. of hide powder, of known hide substance content (nitrogen $\times 5.62$), is shaken for 6 hrs., with 200 c.c. of an aqueous solution of the tanning material; if, after this shaking, the solution gives a turbidity with gelatin-salt solution, the operation must be repeated, using less of the tannin solution. The tanned powder is washed with water and squeezed, about 12 washings being required to remove non-tannin substances (ferrie chloride test), then dried at room temperature for 24 hrs., and analysed for water, fat (chloroform extract), and hide substance. The difference between the percentage sum of these constituents and 100 is taken as the percentage of tannin. Analyses of quebracho, oak bark, sumach, etc., by this method and by the official method of the American Leather Chemists' Association show that the latter method may give results in error to the extent of 43—220%.—W. P. S.

Tanning materials; "Modified shake method" of analysis of —. G. Baldracco and S. Camilla. J. Soc. Leather Trades' Chem., 1920, 4, 101—111.

A REPLY to Parker's criticisms (J., 1920, 123A). The results obtained with the official "shake method" are liable to considerable variation (cf. Jedlicka, Collegium, 1916, 557). Non-tannin filtrates obtained from the official method of analysis of certain tanning materials may contain tannins which are not precipitated by the ordinary gelatin-salt reaction, but are precipitated by the gelatin-acetic acid reagent (0.9 g. of gelatin dissolved in 50 c.c. of distilled water on the water-bath and mixed with 10 c.c. of glacial acetic acid). One drop of this reagent added to 4–5 c.c. of the non-tannin filtrate will precipitate 1 part of tannin in 150,000. The "modified shake method" is carried out with a lightly chromed hide powder which does not alter on keeping and can be easily prepared. The method shows no anomalies and non-tannin filtrates are obtained which are always free from tannin, as shown by the gelatin-acetic acid reagent.—D. W.

Tannins. II. Hamameli-tannin. H. K. Freudenberg and D. Peters. Ber., 1920, 53, 953–961.

THE previous conclusion (J., 1919, 296A) that hamameli-tannin is an ester-like compound of gallic acid (2 mols.) and a hexose is confirmed. The latter consists of an aldo-hexose to the extent of 93% at any rate, but it has not been definitely identified. Various improvements in experimental technique are described, including the purification of hamameli-tannin, the preparation of tannase free from dextrose, the examination of the course of the reaction by measurement of the gradually increasing acidity of the solution, and the replacement of lead carbonate and basic lead acetate by prepared clay for the removal of gallic acid and tannin. (Cf. J.C.S., Aug.)—H. W.

PATENTS.

Liming and other tanning operations upon hides or skins. C. M. Owen. U.S.P. 1,342,866, 8.6.20. Appl., 11.8.19.

SEE E.P. 128,451 of 1918; J., 1919, 649A.

Alkali silicate. G.P. 318,516. See VII.

XVI.—SOILS; FERTILISERS.

Soils; Acidity and acidimetry of —. Investigation of acid soils by means of the hydrogen electrode. H. G. Knight. J. Ind. Eng. Chem., 1920, 12, 457–451.

DETERMINATIONS were made with a new type of hydrogen electrode cell. When an acid soil is added to a neutral salt solution the hydrogen ion concentration reaches a maximum almost at once, but secondary reactions later cause a decrease in the concentration. If a free base is present in the salt solution, it is neutralised rapidly as indicated by the change in hydrogen ion concentration, but the hydrogen ion concentration of the solution continues to rise for an unknown period. There is no sharp break in the progress of base absorption by an acid soil such as would warrant differentiation of active and latent acidity, or immediate and eventual lime requirement. The changes in log C approach nearly straight line functions with progressive addition of base in the presence of a neutral salt solution; different indicators will give differences in lime requirement for soils depending on the slope of the log C curve, and the greatest differences occur with soils containing much organic matter. Insoluble products are formed when a base is added to an acid soil, calcium producing less soluble products than does potassium, but calcium and potassium hydroxides have equivalent

power in neutralising acid soils. The absorption of bases by acid soils is due largely to relatively insoluble acids.—W. P. S.

Soils; Lime requirement of — according to the Veitch method, compared with the hydrogen-ion concentration of the soil extract. A. W. Blair and A. L. Prince. Soil Sci., 1920, 9, 253–259.

WITH the samples of the particular soil examined (a loam soil which had received varying applications of lime) there appeared to be a fairly close correlation between the hydrogen ion concentration of the soil extract and the lime requirement of the soil as determined by the Veitch method. Samples which gave extracts with a hydrogen-ion concentration of about pH 6.7 or over were alkaline by the Veitch method. With soils containing a high percentage of organic matter certain inconsistencies appeared, however, due possibly to the presence of buffer substances.—W. G.

Soils; Hydrogen-ion concentration measurements of — in connection with their "lime-requirements." J. S. Joffe. Soil Sci., 1920, 9, 261–266.

THE results obtained are in fairly close agreement with those of Blair and Prince (preceding abstract).—W. G.

Soil; Relative absorption by — of sodium carbonate and sodium chloride. T. H. Kearney. Soil Sci., 1920, 9, 267–273.

WHEN equal volumes of solutions of equal concentration of sodium carbonate and of sodium chloride are added to the same weight of sand and allowed to remain in contact for 24 hrs., electrical resistance measurements indicate that the sodium carbonate is absorbed to a far greater extent by the sand than is the sodium chloride. Over the concentration range 0.1 to 1.0%, on the average, approximately 63% of the sodium carbonate, but only 23% of the sodium chloride was absorbed. This factor, as well as the partial formation of the less injurious bicarbonate, needs to be taken into account in investigating the comparative toxic effect of alkali salts on plants grown in soil cultures.—W. G.

Wheat. Blish. See XIXA.

PATENTS.

Nitrifying bacteria; Preparation of liquid cultures of —. F. M. Wood. U.S.P. 1,341,030, 25.5.20. Appl., 24.7.19.

CULTURES of nitrifying bacteria for soil fertilisation are prepared by mixing crushed leguminous nodules with water, adding a mixture of equal parts of garden soil and well-rotted manure, and exposing the liquid to a "sun-warmed atmosphere until a strong emulsion is obtained."—J. H. L.

Fertiliser material; Process and product of making —. P. McG. Shuey, Assr. to Savannah Chemical Co. and F. J. Gibson. U.S.P. 1,341,598, 25.5.20. Appl., 2.11.18.

MOLTEN nitre-cake is mixed with a phosphate-bearing material and an organic nitrogenous material.—W. J. W.

XVII.—SUGARS; STARCHES; GUMS.

Sugar; Effect of varying the amount of "inoculum" and concentration on the deterioration of — by moulds. N. Kopeloff. J. Ind. Eng. Chem., 1920, 12, 455–457. (Cf. J., 1920, 380A.)

DETERIORATION increases with an increase in the number of mould spores inoculated into the sugar and with a decrease in concentration of the films surrounding the sugar crystals. At moisture ratios of less than 0.18 there is little, if any, de-

terioration with a mould infection of less than 5000 spores per grm.; at moisture ratios above 0.18 deterioration occurs with upwards of 100 spores per grm. Under similar conditions *Aspergillus Sydowi*, Bainier has a greater effect than *Penicillium expansum* or *A. niger*.—W. P. S.

Dextrose; Action of hydrocyanic acid on —. J. Bongault and J. Perrier. *Comptes rend.*, 1920, 179, 1395—1397.

WHEN an excess of potassium cyanide acts on dextrose the action is slow, the whole of the dextrose being combined only after at least two days at 20° C., and the time required increases with the dilution of the solution. If an excess of dextrose acts on potassium cyanide the whole of the cyanide is destroyed in 10 hrs. at 20° C., and the mixture is no longer toxic.—W. G.

Wood charcoal. Pickles. See IIb.

PATENT.

Fruit jellies etc. G.P. 303,995. See XIXA.

XVIII.—FERMENTATION INDUSTRIES.

Enzymic extract, polyzyme; Properties of a specially prepared — comparing its starch liquefying power with malt diastase. J. Takamine, jun., and K. Oshima. *J. Amer. Chem. Soc.*, 1920, 42, 4261—1265.

POLYZIME is an aqueous extract of diastatic enzymes, sp. gr. 1.03—1.06, containing many other enzymes, made from a specially prepared culture of *Aspergillus oryza* on media, consisting mainly of wheat bran. Its amylolytic power by Wohlgemuth's method is D 40°/30 min., 3.000; D 40°/24 hrs., 115.000; whilst Lintner's saccharogenic value at 21° is 43, and at 50°, 150°. Its diastatic power remains unchanged at the ordinary temperature for 6 months, but is diminished after 3 hrs. above 40° C.—J. K.

Acidity of worts, beers, and other physiological liquids; New method for determining —. III. Investigation of carbonates and phosphate-carbonate mixtures in presence of capillary active indicators of acid and alkaline nature. W. Windisch and W. Dietrich. *Woch. Brau.*, 1920, 37, 35—38, 43—46, 54—56, 70—71. *J. Inst. Brew.*, 1920, 26, 388—389. (*Cf. J.*, 1920, 128 A, 461 A.)

THE surface-tension methods previously described were applied to the study of the equilibria produced in solutions containing potassium, magnesium, or calcium carbonate (or bicarbonate), and monopotassium phosphate (*cf. J.*, 1920, 293 A). Eucupine dihydrochloride and undecylic acid were employed as "indicators."—J. H. L.

Filter pulp; Purification of — [in breweries]. R. Heuss. *Z. ges. Brauw.*, 1920, 43, 145—147, 154—156, 161—163. (*Cf. Foster, J.*, 1916, 1170.)

AN investigation of the purification of pulp from beer filters, including an account of a biological control of the usual method of purification as practised in ten different breweries. Provided the cakes have been completely disintegrated in the preliminary washing with cold, or preferably lukewarm water, sterilisation can usually be attained by heating for ½ hr. at about 80° C. If the apparatus contains less than 40—50 pts. of water to 1 pt. of dry pulp, longer heating or a higher temperature is desirable. Old pulp appears to be more difficult than new to sterilise, possibly owing to the presence of knots. After sterilisation the pulp always becomes more or less re-infected before it is replaced in the filter. The utmost cleanliness is necessary, and the whole series of operations—

washing, sterilising, pressing and replacement in the filter, should be completed with the least possible delay.—J. H. L.

Pyruvic acid, Fermentability of — under the conditions of the fraction method. [*Fermentation of pyruvate-sulphite by yeast.*] C. Neuberg and E. Reinthur. *Ber.*, 1920, 53, 1039—1052.

THE behaviour of the pyruvic acid-sulphite complex has been further studied, particularly with respect to its action under the influence of widely-varying classes of yeast and yeast preparations. The complex is at least as readily fermented, as is pyruvic acid itself (*cf. Zerner, J.*, 1920, 276 A). All types of yeast and yeast preparations decompose pyruvic acid in the presence of an equivalent or greater amount of sulphite and under suitable conditions of permeability (which appear to be comparable with the osmotic requirements of sugar fermentation); the pyruvic acid-sulphite complex, in spite of its stability, is more readily fermented than the unstable, sugar-sulphite compound. (*Cf. J.C.S.*, Aug.)—H. W.

Wines; Process of treating [clarifying] white —. L. Monnier. *Ann. Chim. Analyt.*, 1920, 2, 147—148.

CERTAIN white wines examined by the author contained a bright blue sediment consisting of ferric ferrocyanide; potassium ferrocyanide and gelatin had evidently been used for clarifying the wine. —W. P. S.

Wine vinegar; Determination of total solids in —. Aschoff and H. Haase. *Z. öffentl. Chem.*, 1920, 26, 4—6.

INDIRECT determination of the total solids in wine vinegar by the method of Lehmann and Gerum (*Z. Unters. Nahr. Genuss.*, 1912, 23, 267) gives more accurate results than a direct determination. The sp. gr. (*d*) of the vinegar is determined, the acidity titrated with N/1 sodium hydroxide solution, and the number of c.c. multiplied by 0.00018 ($=d'$). The difference between *d* and *d'* gives the sp. gr. of the extract solution, and from this the total solids are obtained by means of Windisch's table.

—C. A. M.

Alcohol; [Temperature] corrections for densities of —. H. D. Richmond. *Analyst*, 1920, 45, 222.

A TABLE gives the figures necessary to correct the sp. gr. to 15.5°/15.5° C., when the determination has been made between 10° and 15° C., or between 16° and 25° C., and covers a range of sp. gr. from 0.79 to 0.99.—W. P. S.

PATENT.

Grain; Process for steeping and malting —. O. Rummel. G.P. 318,701, 5.12.17.

GRAIN is treated with cellulose waste lyes or fermented cellulose lyes, which may be added to the steeping water. This shortens the steeping process, promotes oxidation, and prevents mould growth.

—C. A. M.

XIXA.—FOODS.

Wheat; Effect of premature freezing on composition of —. M. J. Blish. *J. Agric. Res.*, 1920, 19, 181—188.

PREMATURE freezing, i.e., freezing of the immature growing plant, affects the composition of wheat and the flour milled therefrom, the percentage of non-protein nitrogen, reducing sugars, and acid-reacting substances being higher in each case than in the corresponding sound products. The non-protein nitrogen of frozen wheat contains a considerably higher percentage of α-amino-nitrogen than that of sound wheat.—W. G.

Pearl barley. Its manufacture and composition. J. A. LeClerc and C. D. Garby. J. Ind. Eng. Chem., 1920, 12, 451—455.

THE pearling process consists in subjecting the barley to a scouring action between a revolving mill-stone and a surrounding sieve, the latter moving slowly in the opposite direction to the stone; from 5 to 6 pearling operations are necessary, and in each a certain amount of endosperm is removed, in addition to the husk and bran. The first two pearlings yield a product known as "pot barley" and cause a loss of 22% of the whole material, 25% of the protein, 41% of the fat, and 50% of the mineral constituents. In continuing the operations to produce pearl barley (6 pearlings) 65% of the barley material is removed, together with 74% of the protein, 80—85% of the fat, P_2O_5 , K_2O , CaO , and MgO , and 97% of the crude fibre and SiO_2 .—W. P. S.

Rangoon beans [; Hydrocyanic acid in —]. W. Koenig. Chem.-Zeit., 1920, 44, 405—408, 414—415, 433—434.

HYDROCYANIC acid (14—65 mg. per 100 g.) was found in 59 samples of Rangoon beans (*Phaseolus lunatus*). The enzyme in the beans is not destroyed by boiling, and the whole of the hydrocyanic acid is liberated from the glucoside by exposing the cooked beans for 24 hrs. to the action of water. The glucoside is not decomposed by pepsin with dilute hydrochloric acid. In the case of beans containing not more than 30 mg. HCN per 100 g., the hydrocyanic acid may be removed by twice soaking the beans for 24 hrs. with 5 times the amount of cold water, and then boiling them twice for 1 hr. with the same excess of water, which is renewed as it evaporates. Beans thus treated lose about 14% in weight, but are then non-toxic. The husks of a sample contained 6:1 and the kernels about 40 mg. HCN per 100 g.—C. A. M.

PATENTS.

Grain; Process and apparatus for converting — into paste or dough. J. C. A. Pointe. E.P. 117,271, 5.7.18. Conv., 5.7.17. Addn. to 8381 of 1913 (J., 1914, 330).

THE grain is washed and macerated in water and then subjected to pressure between rubber-covered rollers and superposed perforated plates in order to separate the pulp and husks; salt, yeast, etc., may be added during the pulping process. The washing apparatus consists of a cylinder fitted with a vertical rotating shaft provided with helical blades and the macerator has a constant temperature water-jacket.—W. P. S.

Vegetables and the like; Method and apparatus for drying —. S. Vlessing. E.P. 143,074, 20.6.19.

VEGETABLES are subjected to the action of superheated steam (at 180°—200° C.) for 5—10 mins., and then dried on cruciform screens in an upward current of hot air. The screens are arranged in horizontal layers, and by periodically rotating them the vegetables are transferred from one layer to the next lower one, and so on.—W. P. S.

Meat and fish and like food preparations; Preservation and treatment of —. E. G. Peyton and R. Ferguson. E.P. 143,669, 24.3.19.

THE food is immersed for a time in an electrolytic bath, which may contain common salt in solution, and then desiccated for preservation. Prior to use the product may receive a further electrolytic treatment.—J. H. L.

Milk; Reconstructed — and process of making the same. A. W. Bosworth, Assr. to The Boston Floating Hospital. U.S.P. 1,311,010, 25.5.20. Appl., 1.7.18.

NON-HUMAN milk is treated by first separating the

fat, together with fat-soluble vitamins, and the casein, then removing from the serum practically all the calcium and phosphorus, without any of the dissolved vitamins, and finally re-combining the resulting serum with a portion of the fat and with protein and calcium and phosphorus in substantially the same proportions as are present in human milk.—J. H. L.

Humanised milk; Preparation of —. H. Timpe. G.P. 319,022, 2.1.19.

FRESH cow's milk is enriched with fat and treated with a mixture of sodium and potassium pyrophosphates (3 g. per l.). The precipitated less digestible portion of the casein and most of the calcium salts are separated by heating and centrifuging, and the milk intimately mixed with a solution containing 9% of lactose.—C. A. M.

Fruit juices and jellies; Preparation of — from fruits and other vegetable products with the simultaneous manufacture of preserves. O. and C. Biemann. G.P. 303,995, 25.11.16.

FRUITS etc. are treated with the runnings from sugar refineries etc. (e.g., a solution of molasses) in a connected series of diffusion vessels.—C. A. M.

Lupin seeds; Process of preparing a flour free from bitter principles from —. J. D. Riedel. G.P. 319,591, 30.11.17.

LUPIN seeds are treated with a solvent for fats in the presence of alkali and then with strong alcohol, to obtain a flour suitable for human food.—C. A. M.

Preserving peas, beans, and other pulse. A. Bruff. E.P. 143,804, 20.12.19.

Drying vegetables and other materials; Processes and apparatus for —. A. A. Goubert. E.P. 144,080, 27.5.19.

SEE U.S.P. 1,305,599 and 1,305,600 of 1919; J., 1919, 597 A, 562 A.

XIXB.—WATER PURIFICATION; SANITATION.

Sewage; Purification of — by activated sludge. R. Cambier. Comptes rend., 1920, 170, 1417—1419.

A VERY slight trace of chloroform in the air bubbled through the sewage during purification by the activated sludge process is sufficient to inhibit the formation of nitrates. Similarly if air saturated with chloroform is bubbled through the sewage for 1 hr. and then pure air for 4 hrs. to remove the chloroform, on the introduction of the sludge and the subsequent bubbling of air, the ammonia disappears but no nitrates are formed. Further, attempts to isolate nitrifying organisms from activated sludges were not successful. These results are opposed to the view that nitrification takes place during the purification of sewage by the activated sludge process.—W. G.

PATENTS.

Water purification and softening; Manufacture of base-exchanging compound for —. J. Crossfield and Sons, Ltd., and H. J. Wheaton. E.P. 142,974, 21.3.19.

A COMPOUND of silica and soda for water purification and softening is prepared by neutralising dilute sodium silicate solution to the extent of about 50% of the sodium. The gelatinous mass obtained is dried at a low temperature, washed to remove soluble salts, and granulated. A suitable product contains 12.6% Na_2O and 87.4% SiO_2 .—W. J. W.

Sewage and other foul liquids; Apparatus for aerating —. R. Ames. E.P. 143,369, 28.4.19.

IN aerating tanks a movable device for raising

the sludge to the surface comprises a series of vertical pipes fixed in a carriage, the lower ends of the pipes being bell-mouthed, and the upper ends being provided with mushroom-shaped flanges. By means of an internal concentric pipe, and an air lift consisting of a conical box with apex below and slotted to allow air to escape upwards, or other suitable device, the sludge is raised to the surface through the bell-mouthed pipes, and then flows in a cascade over the mushroom flanges back into the tank, becoming aerated in the process.—W. J. W.

Sewage and other liquids; Purification of —. W. Jones. U.S.P. 1,341,561, 25.5.20. Appl., 8.8.19.

SEE E.P. 132,826 of 1918; J., 1919, 876A.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Salvarsan and neosalvarsan; Examination of —. Utz. Süddeutsch. Apoth.-Zeit., 1920, 60, 187—188. Chem. Zentr., 1920, 91, 11, 772. (Cf. J., 1920, 246A.)

SALVARSAN preparations may be conveniently identified by Gaebel's reaction, according to which this compound gives the diazo reaction with α -naphthylamine, but not with β -naphthylamine. Salvarsan and neosalvarsan can be distinguished from one another by the following reactions: 5 c.c. of neosalvarsan solution (1:1000) with 0.1 c.c. of dilute hydrochloric acid gives a precipitate, soluble on boiling and not re-precipitated on cooling. Salvarsan gives no such precipitate. With an equal volume of acetic acid neosalvarsan solutions give a cloudiness changing to an orange-coloured precipitate on heating, whilst salvarsan gives a negative result. With ferric chloride both preparations give a green to red coloration, and with excess the salvarsan solution becomes dark red, and neosalvarsan violet. Mercuric chloride gives with salvarsan a heavy white precipitate, and with neosalvarsan an orange precipitate, both of which become somewhat grey or dirty on heating and rapidly sink to the bottom. Ammonium molybdate yields with salvarsan an olive-green precipitate and with neosalvarsan an olive coloration becoming dark green, but no precipitate. Platinic chloride gives only with neosalvarsan a flocculent precipitate which gradually settles. Picric acid produces with salvarsan solutions an intense yellow precipitate which is not dissolved on heating, whilst with neosalvarsan only a slight opalescence is produced after long standing. On the addition of a concentrated solution of sodium bisulphite the yellow salvarsan solution immediately becomes greyish-green, followed by a dirty grey cloudiness; on warming, the liquid becomes first green and then yellow, and finally a lemon-yellow precipitate is formed. Neosalvarsan gives no reaction with this reagent.—G. F. M.

Sympathomimetic amines. I. Amino-derivatives of naphthalene. A. Madinaveitia. Anal. Fis. Quim., 1920, 18, 66—78.

PHYSIOLOGICAL tests with amines of similar constitution to adrenaline but containing a naphthalene instead of a benzene nucleus, showed that a naphthalene compound is 40 times more active than a benzene one, and that introduction of a hydroxyl in the nucleus increases the physiological action of the compound.—W. R. S.

Saccharin analysis. O. Beyer. Chem.-Zeit., 1920, 44, 437—438.

The formula $x = 2.01329 \times c - 100/0.00845$ may be used to calculate the percentage (x) of *o*-benzoic-sulphimide in commercial saccharin when 1 g. of

the sample is titrated with $N/10$ potassium hydroxide solution using phenolphthalein as indicator; c is the number of c.c. of the alkali solution used.—W. P. S.

Salicylic acid; Melting point of —. W. J. Bush and Co. Perf. Essent. Oil Rec., 1920, 11, 207.

PURE salicylic acid has m.p. approximately 159° C., and products of m.p. 158° — 159° C. are regularly obtained in current British manufacture.

—G. F. M.

Chlorobenzenes; Catalysing action of aluminium in the preparation of —. J. Meunier. Comptes rend., 1920, 170, 1451—1452.

FOR the preparation of chlorobenzene aluminium is an excellent catalyst. To obtain a steady absorption of chlorine 1 pt. of aluminium per 1000 pts. of benzene by weight is used. If the action is stopped when the sp. gr. of the liquid reaches 1.008 a yield of 87% of monochlorobenzene can be obtained.

—W. G.

Chloroform and other volatile substances; Determination of —. A. Schlicht and W. Austen. Z. öffentl. Chem., 1920, 26, 55—57.

TO determine chloroform in a mixture of the same with alcohol, 5 c.c. of the sample is diluted with water to 200 c.c., shaken with 60 c.c. of ether, and, after separation, the sp. gr. of the ether is determined. Each increase of 0.0001 in the sp. gr. corresponds with 0.1786 g. of chloroform in the alcohol-chloroform mixture. The sp. gr. of the latter gives an approximate measure of the alcoholic strength, after the chloroform has been determined and an allowance made for its sp. gr.

—W. P. S.

Camphor; Examination of Chinese crude —. E. R. Dovey. Analyst, 1920, 45, 220—221.

MOISTURE is determined by the carbide method, allowing 3 hrs. for the evolution of the gas. To determine water and oil, 100 g. of the sample is pressed between layers of lint in a steel cylinder, 2 in. in diam. and 6 in. deep, having a perforated bottom plate and a piston worked by a strong screw thread; after 15 mins., the cake is removed and weighed. The moisture content of the pressed cake is determined, and from this are found the amounts of expressed water and oil. The total quantity of oil is calculated on the assumption that the residual water and oil in the pressed cake are in the same proportion as they are in the expressed liquid.

—W. P. S.

Oil of chenopodium from various sources; Composition of —. E. K. Nelson. J. Amer. Chem. Soc., 1920, 42, 1204—1208.

THE need for distilling chenopodium oil in a rapid current of steam, keeping the condenser water warm, and rejecting the water separating from the oil in the receiver, in order to avoid as far as possible decomposition of ascaridol (cf. J., 1908, 710; 1911, 1279) is confirmed. By distillation with low-pressure steam, oil of low ascaridol content was obtained, there being a distillation residue of 7—25%, soluble in water and probably consisting of glycols (cf. J., 1913, 379), as against 2.3%, with more rapid distillation and the precautions stated above. The proportion of ascaridol, determined by distillation under 3—6 mm. pressure, in oil from Carroll County, Maryland, U.S.A., amounted to 60—77%, from Java 70%, from Florida (dry plants) 41%, from Eastern U.S.A. (wild plants) 20%, whilst samples a year old were found to have deteriorated little, if at all. *p*-Cymene, *l*-limonene, and probably α -terpinene are also present in the oil.

—J. K.

Rubiera multifida; [Essential] oil of —. E. K. Nelson. J. Amer. Chem. Soc., 1920, 42, 1286.

A SAMPLE of the light yellow oil from California, sp. gr. 0.8512 at 25°/25° C., $[\alpha]_D^{25} = +35.75$, contained 85% of terpenes, largely β -phellandrene, and also anethole.—J. K.

Tea leaves; Unsaturated alcohol of the essential oil of freshly fermented —. P. van Romburgh. Proc. K. Akad. Wetensch. Amsterdam, 1920, 22, 758–761.

The alcohol appears to be a hexene-3-ol-6. (Cf. J.C.S., Aug.)—J. C. D.

Alkali in organic salts; Estimation of — as sulphate. C. F. van Duin. Chem. Weekblad, 1920, 17, 283–284.

THE alkali salt is evaporated with concentrated sulphuric acid until fuming ceases. The resulting mixture of sulphate and pyrosulphate is dissolved in a little ammonium carbonate solution, the solution is evaporated to dryness, and the residue gently ignited. In this way losses by spurring are avoided, and any carbonaceous matter is easily burned off.—W. S. M.

Bromine chloride and ethylene. Delépine and Ville. See VII.

Conductimetric titrations. Kolthoff. See XXIII.

PATENTS.

Acetic acid; Production of — from paraldehyde as the primary material. Soc. des Acieries et Forges de Firminy. E.P. 121,195, 18.2.19. Conv., 13.3.18.

PARALDEHYDE is directly converted into acetic acid by electrolytic oxidation in a cell provided with a porous cathode, e.g., gas carbon, and a refractory anode, such as magnetite or platinum, using a high current density and 10% sulphuric acid as the electrolyte. The paraldehyde is poured on to the surface of the acid and dissolves until a concentration of 10–15% is reached; the excess floats on the surface and dissolves as the oxidation proceeds. Oxidation is facilitated by the addition of a small quantity of a salt of a metal capable of several degrees of oxidation, such as manganese, cerium, molybdenum, or vanadium. The concentration of the acetic acid may be carried to about 40%, and it is then separated from the sulphuric acid by distillation.—G. F. M.

Ethyl acetate; Manufacture of — from paraldehyde as primary material. Soc. des Acieries et Forges de Firminy. E.P. 131,600, 18.2.19. Conv., 13.3.18.

By adding to the mixture of acetic acid and dilute sulphuric acid produced by the electrolytic oxidation of paraldehyde (preceding abstract), a quantity of ethyl alcohol equivalent to the acetic acid which it contains, and distilling the whole in a rectifying apparatus, esterification is effected and pure ethyl acetate distils. The liquid remaining, containing sulphuric acid and a little non-esterified acid and alcohol, may be again employed as a basis for a fresh electrolysis.—G. F. M.

[Hydr]oxyaldehydes and their ethers; Manufacture of —. A. Weiss. E.P. 139,153, 6.1.20. Conv., 18.2.19.

AROMATIC hydroxyaldehydes or their ethers are obtained by the interaction of an aromatic nitroso-compound such as nitrosobenzene, *p*-nitrosodimethylaniline, etc., and a mixture of formaldehyde with a phenol or phenol ether. For example, an excellent yield of vanillin is obtained by heating for several hours on a water-bath a mixture of

equimolecular proportions of guaiacol and formaldehyde and half a mol. of *p*-nitrosodimethylaniline, methyl or ethyl alcohol being added as a diluent, and gaseous hydrogen chloride being bubbled through continuously. The reaction mixture is then treated with sodium hydroxide, the alcohol distilled off, the by-product, *p*-aminodimethylaniline, extracted with benzene, and the residue acidified and again extracted with benzene to remove the crude vanillin, which is purified by distillation in vacuum.—G. F. M.

$\beta\beta$ -Dichloroethyl sulphide; Production of —. W. J. Pope, C. S. Gibson, and H. F. Thuillier. E.P. 142,875, 3.4.18.

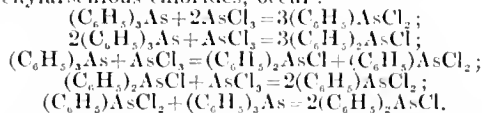
$\beta\beta$ -DICHLOROETHYL sulphide is obtained in upwards of 90% yield by passing dry ethylene into sulphur monochloride heated to and maintained at 50°–70° C. (cf. J., 1920, 383 A).—G. F. M.

Chloropierin; Production of —. K. J. P. Orton and W. J. Pope. E.P. 142,878, 9.5.18.

CHLOROPIERIN is obtained by the action of chlorine on picric acid or other suitable nitro-phenol, in presence of sufficient aqueous alkali, preferably sodium or potassium hydroxide or carbonate solution, to dissolve the nitro-derivative and neutralise the acid produced in the reaction: $C_6H_2(NO_2)_3OH + 11Cl_2 + 5H_2O = 3CCl_3(NO_2) + 13HCl + 3CO_2$.—G. F. M.

Arsenic compounds [mono- and di-arylarsonious halides]; Production of aromatic —. W. J. Pope. E.P. 142,880, 11.6.18.

MONO- and di-arylarsonious halides are obtained by bringing an arsenious halide in the liquid or vapour form into intimate contact with a triarylarsine heated to a high temperature under atmospheric pressure. According to the proportion of arsenious halide employed reactions expressed by the following equations, in the typical case of the phenylarsenious chlorides, occur:—



For these reactions a temperature approaching 350° C. is favourable. The products may be separated by physical or chemical means, or should the diaryl compound be required as the main product any mono-aryl compound present may be converted by gently boiling with triphenylarsine as expressed by the fifth equation, and, conversely, should the mono-aryl compound be required, any diaryl arsenious chloride present may be transformed according to the fourth equation.—G. F. M.

Arsinic acids; Process of preparing aromatic —. A. Mouneyrat. E.P. 112,917, 20.2.19.

AROMATIC arsinic acids are produced by the interaction of aromatic diazonium compounds with cold or warm aqueous or dilute alcoholic solutions of arsenious acid, in an acid, neutral, or alkaline medium in the presence of a copper salt and a reducing agent adapted to the acid or alkaline medium employed. In acid medium hypophosphorous acid or cuprous hydroxide are suitable, whilst in a neutral or alkaline medium sodium hydrosulphite, sodium formaldehydesulphoxylate, or even an excess of alkali arsenite, may be employed. The method of procedure is capable of numerous modifications in detail, of which examples are given.—G. F. M.

Benzoic acid; Production of —. W. P. Thompson. From Les Etabl. Poulenc Frères, J. B. Senderens, and J. Aboulenc. E.P. 143,392, 27.5.19.

BENZOIC acid is obtained from toluene by oxidation with a mixture of sulphuric acid and manganese dioxide, the reaction mixture being maintained at

60°–70° C., whereby only small quantities of benzaldehyde escape further oxidation, and only traces of carbon dioxide are formed by the further oxidation of benzoic acid. The operation is conducted in a closed jacketed vessel fitted with arrangements for steam heating and water cooling, with an agitator, and with a gas meter to record the evolution of gas. The sulphuric acid and manganese dioxide are introduced into the toluene in small quantities at a time in order to prevent a rise of temperature, and any notable evolution of carbon dioxide is at once checked by cooling the reaction mixture, which is, however, maintained at the highest temperature possible consistent with the above conditions. From 200 kg. of toluene on treatment with 400 kg. of manganese dioxide and 2000 kg. of sulphuric acid (52°–53° B., sp. gr. 1.56–1.58) there are obtained 100 kg. of benzoic acid, 8 kg. of benzaldehyde, and 100 kg. of recovered toluene.—G. F. M.

Thymol; Synthetic manufacture of —. M. Phillips. U.S.P. 1,332,680, 2.3.20. Appl., 8.9.19.

p-Cymene is nitrated, the nitro-compound reduced to the amino derivative (cymidine), the latter is sulphonated, the amino group eliminated, and the sulphonic acid subjected to alkali fusion.—L. L. L.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Coloured [photographie] sensitizers derived from quinolines, quinaldines, and lepidines containing dimethylamino and diethylamino groups. H. Barbier. Bull. Soc. Chim., 1920, 27, 427–439.

THE author has prepared the methiodides and ethiodides of *p*-dimethylaminoquinoline, of 6-diethylaminoquinoline, of 6-dimethylaminoquinoline, and of 6-dimethylaminolepidine, and finds that when these alkyl iodides containing a dialkylamino group are condensed amongst themselves or with alkyl iodides of other bases or with *p*-dimethylaminobenzaldehyde, they give a series of new colouring matters belonging to the cyanines. All of these cyanines, which contain the active auxochrome group, $N(CH_3)_2$ or $N(C_2H_5)_2$, can be used as photographic sensitizers. (*Cf.* J.C.S., Ang.)—W. G.

Photographic plate; Action of phosphorus, during oxidation, on the — and its ionising power. W. P. Jorissen. Rec. Trav. Chim., 1920, 39, 429–431.

A REPLY to Centnerszwer and Petrikalu (*Z. physik. Chem.*, 1912, 80, 235), in which it is shown that the effects obtained on a photographic plate by phosphorus in process of oxidation depend upon the velocity of the current of air removing the mist produced by the oxidation and on the temperature.—W. G.

XXII.—EXPLOSIVES; MATCHES.

Cellulose and its esters. I. Fractional precipitation of nitrocelluloses. J. Duclaux and E. Wollman. Bull. Soc. Chim., 1920, 27, 414–420.

NITROCELLULOSE can be divided into a number of fractions, which have widely differing viscosities in acetone solution, by fractional precipitation from its acetone solution by means of aqueous acetone or water. By repeating this process with the extreme fractions, samples were obtained, the viscosities of which in 2% acetone solution were as 46:1, and at one end a nitrocellulose was obtained, which in 1% acetone solution was as viscous as pure glycerol. These fractions obey the law of mixtures in so far as their viscosities are concerned, and the nitrogen content of the different fractions from one and the same operation is practically constant. A better

method of separation of the fractions is probably by filtration through a series of ultra-filters. The different nitrocelluloses can best be characterised and differentiated by osmotic pressure measurements.—W. G.

Powders and explosives; Analysis of —. *Differentiation of trinitroglycerin and dinitroglycol.* Marquoyrol and E. Goutal. Bull. Soc. Chim., 1920, 27, 443–448.

DINITROGLYCOL may be differentiated from trinitroglycerin either by the steady spontaneous loss in weight of the former when exposed in a desiccator over sulphuric acid at 12° C. at the ordinary pressure, the latter not losing in weight, or by their different effects in depressing the freezing-point of benzene, the former lowering the freezing-point in 10% solution by 3.06° C. and the latter by 2.13° C. Either of these methods is applicable for the estimation of the percentages of these two substances present in a mixture, and the results are not appreciably vitiated by the presence of impurities such as vaseline, diphenylamine, dimethyldiphenylurea, diethyldiphenylurea, or dinitrotoluene. For a correct interpretation of the results it is advisable to determine in the ether extract the substances other than dinitroglycol or trinitroglycerin.—W. G.

Nitration of m-nitrotoluene. Marquoyrol, Kohler, and Jovinet. Bull. Soc. Chim., 1920, 27, 420–424.

WHEN *m*-nitrotoluene is nitrated with a mixture of 16 pts. of sulphuric acid (98%) and 2.25 pts. of nitric acid (88%) for 8 hrs. at 100° C., a mixture of 2.3.4-, 3.4.6- and 2.3.6- or 2.3.5-trinitrotoluenes is obtained.—W. G.

Trinitration of o-xylene. Marquoyrol and P. Loriette. Bull. Soc. Chim., 1920, 27, 424–426.

By passage through its mono- and dinitro-derivatives without purification of these, but using fresh nitration mixtures at each stage, *o*-xylene gave the two trinitro-*o*-xylenes previously described by Crossley and Renouf (*Chem. Soc. Trans.*, 1909, 202).—W. G.

Mercury fulminate in mixtures for detonators; Direct estimation of —. Marquoyrol. Bull. Soc. Chim., 1920, 27, 448.

A QUANTITY of 1–1.5 g. of the mixture is extracted on a filter with 25–30 c.c. of a 5% solution of potassium cyanide. The filtrate is collected in the dish of a Riche's apparatus, and the mercury is deposited on the dish by electrolysis and weighed as such after washing with water and alcohol.—W. G.

Chemicals [nitrates and aluminium] used in pyrotechnics; Short commercial analytical methods for determination of purity of —. H. B. Faber and W. B. Stoddard. J. Ind. Eng. Chem., 1920, 12, 576–578.

FOR the determination of small amounts of sodium in potassium nitrate, 1 g. is dissolved in 5 c.c. of water, 1 c.c. of 2N nitric acid added, and then 10 c.c. of an acid solution of potassium nitrate, bismuth nitrate, and caesium nitrate (Ball, J., 1910, 46, 902), several precipitations being made simultaneously in Erlenmeyer flasks. After connecting the flasks, coal gas is passed through them, and the series is then closed at one end by a glass plug and at the other by a Bunsen valve. The flasks are allowed to stand in a cool place for 48 hrs., and the precipitate is collected in a Gooch crucible and washed with acetone, filtration being conducted rapidly to reduce exposure to the air. The crucible is dried in air bath at 100° C. for 30 mins., and the weight of sodium calculated from weight of precipitate $\times 0.03676$. Alternatively the caesium sodium

bismuth nitrate may be determined volumetrically by titration with potassium permanganate. The determination of nitrate in commercial salts is effected by decomposition with tungstic anhydride. 1 g. of nitrate is carefully heated with 5 g. of tungstic anhydride in a platinum crucible over a Bunsen burner, the percentage of pure nitrate being calculated from the loss, due to liberation of oxygen and oxides of nitrogen. For the estimation of aluminium in flake or powder, its reducing power on litharge is utilised. 3 g. of aluminium is mixed with 100 g. of litharge and 30 g. of borax glass, 25 g. of the latter being added for covering. The mixture is fused in a muffle, and after 20 mins. the molten lead is separated, freed from slag, and weighed, its weight $\times 0.0872$ representing the aluminium.—W. J. W.

Nitration of naphthalene. Pascal. See III.

PATENTS.

Explosives for detonators; Manufacture of —. W. Rintoul, J. Weir, A. G. Lowndes, and Nobel's Explosives Co., Ltd. E.P. 142,898, 10.1.19.

THE sensitiveness to shock of explosives such as lead azide is reduced by precipitating the azide from solution as an amorphous, or abnormally crystalline, or micro-crystalline powder, in presence of a colloid, such as gelatin. The fine powder obtained may be "corned" by addition of gum or starch. —W. J. W.

Priming mixture. A. S. Cushman. U.S.P. 1,325,928, 23.12.19. Appl., 20.8.18.

A MIXTURE of a "nitrated fuel body," e.g., tetranitromethylaniline (tetryl), and a variable proportion of a non-combustible basic oxide, such as litharge, together with other known ingredients (antimony pentasulphide, lead thiocyanate, potassium chlorate).

Loose compounds [explosives]; Manufacture of —. J. E. Bloom. U.S.P. 1,333,701, 16.3.20. Appl., 3.12.17.

IN the production of loose compounds, including explosives, nitro-substitution products, composite nitro explosives, nitrocellulose, etc., the components are dissolved in suitable liquid mobile media and the solutions blended and mixed under insulated conditions in an alternating current field or fields of regulated frequency and strength, and under suitable conditions of temperature, speed, and time.—L. L. L.

Explosive propellent powder. F. I. du Pont. U.S.P. 1,341,207, 25.5.20. Appl., 28.4.19.

A PROPELLENT powder consists of finely-divided ammonium nitrate embedded in a small proportion of a substance which forms a matrix for it, a non-porous mass being obtained on hardening.

—W. J. W.

Detonating fuse. W. C. Cope, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,341,705, 1.6.20. Appl., 4.6.19.

THE detonating mixture is composed of trinitrophenylmethylnitramine and trinitrotoluene.

—W. J. W.

Pyroxylin; Solvent for use with — and compositions containing the same. E. M. Flaherty, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,341,710, 1.6.20. Appl., 11.9.18.

A MIXTURE containing amyl acetate, *n*-butyl alcohol, and benzene is used as a solvent for nitrocellulose.

—W. J. W.

XXIII.—ANALYSIS.

Platinum wire in flame and bead tests; Substitutes for —. C. C. Kiplinger. J. Ind. Eng. Chem. 1920, 12, 500.

THE "lead" from a blacklead pencil is heated to redness, dipped in borax, and again held in the flame so that a hanging drop of borax glass is formed at the end. The reducing action of the carbon does not interfere with subsequent bead tests. The filter paper method of Ehringhaus (J., 1920, 315 a) is rendered more useful by inserting the rolled strip into a glass tube containing the solution etc. under examination. The mouth of the tube is constructed so that the paper fits it tightly, and about 3 cm. of the strip is allowed to project so as to form a wick.—C. A. M.

Bulb; Connection — for distillations. C. M. Clark. J. Ind. Eng. Chem., 1920, 12, 366.

THE upper tube of an anti-spray bulb passes a short distance into the bulb, and a baffle-plate, in the form of an inverted watch-glass, is attached to the lower end of the tube; the edge of the baffle-plate reaches to within 5 mm. of the inner wall of the bulb, forming a narrow channel for the passage of the vapours. A hole in the portion of the upper tube inside the bulb allows the vapours to escape through the tube to the condenser.—W. P. S.

Melting points; Apparatus for the determination of —. L. M. Dennis. J. Ind. Eng. Chem., 1920, 12, 366—368.

TO obtain a better circulation and more uniform temperature of the sulphuric acid in the type of apparatus proposed by Thiele (J., 1907, 435), the lower portion of the tube is inclined slightly downwards and is then constricted and bent upwards to the point where it enters the main or vertical tube. —W. P. S.

Densimeter; New industrial — for gases. E. Hauser. Anal. Fis. Quim., 1920, 18, 79—82.

A DENSIMETER for continuous readings, having no metallic parts, consists of a glass bell jar through which the gas is passed. A cylinder within the enclosed space contains water (for moist gases) or paraffin (for dry gases) in which is immersed an areometer provided with a closed glass bulb at the top of its graduated stem. The areometer sinks if the gas be lighter, and rises if heavier, than air. —W. R. S.

Conductometric titrations; Use of — in neutralisation analysis. I. Neutralisation of acids and bases. I. M. Kolthoff. Z. anorg. Chem., 1920, 111, 1—27.

TITRATIONS are carried out by running comparatively strong alkali solution into the dilute acid solution and determining the conductivity at intervals. The neutralisation curve is obtained by plotting conductivities as ordinates against volume of alkali added as abscissae. When a strong acid is titrated with a strong base, the neutralisation curve is a straight line descending sharply to the neutral point and then rising sharply as a second straight line as the solution becomes increasingly alkaline. When a comparatively weak acid, such as salicylic acid, is titrated with a strong base, the neutralisation curve passes through a minimum before the neutral point is reached, and this point is not sharp. It may be found, however, by determining the conductivity-concentration curve of the salt, which cuts the neutralisation curve at the neutral point. A very weak acid may be titrated with a strong base if the dissociation constant is not less than 10^{-10} when the concentration of the solution is 0.1N, or 10^{-9} for a 0.01N solution. The neutralisation curve then consists of a rising straight line which changes direction more or less

sharply at the neutral point. Boric acid and phenol were successfully titrated with sodium hydroxide. A weak acid can be titrated with a weak base if their dissociation constants are greater than 3×10^{-6} . Thus acetic acid can be titrated with ammonia, but boric acid cannot because, on account of the hydrolysis of the neutral salt, the neutralisation curve is rounded off on either side of the neutral point. (Cf. J.C.S., ii., 420.)—E. H. R.

Conductometric titrations; Use of —, in neutralisation analysis. II. Simultaneous titration of different acids or bases. I. M. Kolthoff. Z. anorg. Chem., 1920, 111, 28—51.

A STRONG and a weak acid in equal concentrations in 0.1N solution can be titrated together and good results obtained if the dissociation constant of the weaker acid is less than 4×10^{-4} . With weaker solutions or greater relative concentration of the weaker acid, the dissociation constant of the latter should be smaller. The neutralisation point of the strong acid is best found as the intersecting point of the salt conductivity curve and the neutralisation curve (cf. preceding abstract). The dissociation constant of the weaker acid may be lowered, and sharper results obtained, by addition of alcohol. Two weak acids can be titrated together in equal concentrations when the ratio of their dissociation constants is smaller than 0.001, provided the ionic mobilities of the anions are not too closely similar. Thus oxalic and tartaric acids or oxalic acid and phenol can be titrated together, but not acetic acid and phenol, because the change of direction of the curve at the point of neutralisation of the first acid is too slight. When a dibasic acid is titrated, the first neutralisation point can hardly be detected, but oxalic acid is exceptional, on account of the wide difference between its first and second dissociation constants. (Cf. J.C.S., ii., 421.)—E. H. R.

Potassium hydrogen phthalate as a standard in volumetric analysis. W. S. Hendrixson. J. Amer. Chem. Soc., 1920, 42, 724—727.

POTASSIUM hydrogen phthalate for use as a standard in volumetric work (J., 1915, 1118) is best prepared from phthalic anhydride and potassium carbonate, and is obtained absolutely pure by three crystallisations; it contains no water of crystallisation and is not hygroscopic. A saturated solution contains 10.23% of the salt at 25° C. and 36.12% at the boiling point.—J. F. S.

Titration curves; Colorimetric determination of —. L. J. Gillespie. J. Amer. Chem. Soc., 1920, 42, 742—748.

TITRATION curves are obtained colorimetrically without the use of a "buffer" mixture. A colour standard is used, consisting of a pair of test-tubes containing together 10 drops of indicator solution of suitable strength, the drop ratio in the two tubes being varied from 1:9 to 9:1. One of the tubes contains dilute alkali and the other dilute acid. Reference to a table gives the hydrogen exponent corresponding to the drop ratio, or the exponent can be calculated from the ratio $pH = k + \log(\text{drop ratio})$, where the drop ratio is the ratio of the number of drops of the indicator solution in the alkali tube to that in the acid tube, and k is a constant depending on the indicator. It has the following values: tetrabromophenolsulphonophthalein, 4.1; methyl red, 5.0; dibromo-*o*-cresolsulphonophthalein, 6.3; dibromothymolsulphonophthalein, 7.1; phenolsulphonophthalein, 7.7; *o*-cresolsulphonophthalein, 8.1; and thymolsulphonophthalein, 8.8.—J. F. S.

Potassium; Estimation of — as perchlorate. G. P. Baxter and M. Kobayashi. J. Amer. Chem. Soc., 1920, 42, 735—742.

THE method previously described (J., 1917, 354) is modified by using absolute alcohol containing 1%

perchloric acid for the initial extraction of sodium perchlorate from the precipitate before washing it with a saturated solution of potassium perchlorate. (Cf. J.C.S., ii., 388.)—J. F. S.

Manganese and zinc; Detection of — in the presence of phosphates and oxalates. E. Schmidt. Ber. deuts. Pharm. Ges., 1920, 30, 217—218.

ZINC and manganese are precipitated as sulphides by ammonium sulphide in the presence of phosphoric or oxalic acid, and not as phosphates or oxalates, as stated by Wester (cf. ante), and their separation from one another may then be effected by the difference in solubility of the respective hydroxides in ammonia and ammonium chloride solution. (Cf. J.C.S., ii., 289.)—C. A. M.

Radium; Practical methods for the determination of —. III. Alpha-ray method, gamma-ray method, miscellaneous. S. C. Lind. J. Ind. Eng. Chem., 1920, 12, 469—472. (Cf. J., 1915, 610; 1916, 113).

IMPROVEMENTS in the construction of the interchangeable electroscope (*loc. cit.*) are described, and consist of the substitution of amber or amberoid insulation instead of sealing-wax for both insulators, the use of metal stopcocks brazed into the chamber, and adjustable fittings for the microscope holder. Whilst the alpha-ray method is trustworthy for qualitative and very rough quantitative purposes, it is not sufficiently accurate for scientific control or when a mineral is sold on the result obtained by the method. The three chief sources of error are the variation in the amount of loss of radium emanation by gaseous diffusion from the ore, the variability of the radium-uranium ratio in the ore, and the position of the radioactive material in the individual grains. In the gamma-ray method, the radium preparation must be confined in a closed vessel to prevent escape of gas; the measurement may be made after the vessel has been closed for a month or more, the gamma-radiation having then reached a constant maximum which is directly proportional to the quantity of radium present.—W. P. S.

Molybdenum; Application of rotating zinc "reductor" to determination of —. W. Scott. J. Ind. Eng. Chem., 1920, 12, 578—580.

MOLYBDIC acid in dilute sulphuric acid solution may be reduced at 20°—30° C. by a rotating zinc cylinder, the containing beaker being kept covered during the reduction by a split cover-glass which serves to retain enough hydrogen to act as a protective covering for the zinc. After reduction the solution may be titrated in the same beaker with permanganate, or it may be transferred to another beaker containing ferric alum solution and syrupy phosphoric acid.—W. J. W.

Nitric nitrogen; Apparatus for the determination of — by Devarda's method. J. Erlich. Ann. Chim. Analyt., 1920, 2, 143—144.

THE apparatus consists of a reaction flask, a condenser, and a receiver; an anti-spray bulb is placed between the flask and the condenser, the bulb being partly filled with glass beads. A central tapped tube extends downwards through the bulb to the bottom of the flask, so that a current of air may be drawn through the apparatus to carry over the last traces of ammonia.—W. P. S.

Diphenylamine reaction [for nitrates]; Colour changes of the —. E. M. Harvey. J. Amer. Chem. Soc., 1920, 42, 1245—1247.

A MODIFIED diphenylamine reagent for use in microchemical tests for nitrates in plant tissues consists of 0.5 g. of diphenylamine, 7.5 c.c. of sulphuric acid (95—96%), and 2.5 c.c. of 10% aqueous potassium chloride solution; or, if it is particularly de-

sirable to avoid darkening of plant tissues, 5 c.c. of sulphuric acid, 3 c.c. of glacial acetic acid, and 2 c.c. of 12% aqueous potassium chloride solution may be used at some sacrifice of colour intensity. A diagram is given showing the variation of colour, shade, and intensity with strength of sulphuric acid. The proportion of diphenylamine is variable between fairly wide limits, but in excess it interferes with the reaction; variations of temperature between 20° and 50° C. have little effect; potassium chloride causes not only an intensification of the blue colour, but an extension in both directions of the concentrations of sulphuric acid which produce it, and is more suitable for this purpose than sodium chloride owing to the greater solubility of its acid sulphate. The stability of the blue colour once produced, even in concentrations of acid in which normally it would not be produced, renders the order of mixing of some importance. (*Cf.* J.C.S., Aug.) — J. K.

Benzidine sulphate; Solubility of — in water. C. S. Bisson and A. W. Christie. J. Ind. Eng. Chem., 1920, 12, 485—486.

The solubility of benzidine sulphate in water at various temperatures was as follows:—At 0° C., 0.049; at 25° C., 0.098; at 50° C., 0.141; and at 80° C., 0.290 (by evaporation), and 0.252 (by titration) g. per l. Hence in washing benzidine sulphate in the quantitative determination of sulphate the minimum amount of cold water should be used. (*Cf.* J.C.S., ii., 385.)—C. A. M.

Gas analysis; Combustion apparatus for use in —. E. R. Weaver and P. G. Ledig. J. Ind. Eng. Chem., 1920, 12, 368—370.

A combustion pipette having a capacity of about 150 c.c. has two short side tubes between which a platinum spiral is strung; the ends of the spiral extend down the side tubes and connect with platinum leads sealed through glass caps closing the ends of the tubes; the caps are cemented on to the tubes and are readily removed when necessary. A combustion capillary may be constructed on the same principle; the capped side tubes are fused on to opposite sides of the capillary, one below the other, and a platinum spiral is strung between them. This portion of the capillary is surrounded by a small water jacket.—W. P. S.

See also pages (A) 507, *Nitrogen in coal etc.* (Parker). 509, *Acetylene* (Willstätter and Maschmann, also Arnold and others); *Petroleum oils* (Hess). 512, *Wool etc.* (Krais and Biltz); *Bast fibres* (Haller). 513, *Wood* (Dore). 514, *Arsenic in sulphuric acid* (Kohr). 515, *Chlorides* (Bolam). 516, *Arsenic in sulphur* (Davis). 519, *Mercury* (Place). 522, *Tanning materials* (Wilson and Kern). 523, *Tanning materials* (Baldracco and Camilla). 524, *Alcohol* (Richmond); *Incense* (Aschoff and Haase). 526, *Salvarsan etc.* (Utz); *Saccharin* (Beyer); *Chloroform etc.* (Schlicht and Austen); *Camphor* (Dovey). 527, *Alkali in organic salts* (Van Duin). 528, *Explosives* (Marqueyrol and Goutal); *Mercury fulminate* (Marqueyrol); *Chemicals for pyrotechnics* (Faber and Stoddard).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Acton and McKean. Separation of solids from liquids. 19,131. July 7.

American Drying Processes, Inc. Desiccation. 19,982. July 9. (U.S., 27.3.19.)

Balthasar. Jets for evaporating liquids. 20,008. July 9. (Belg., 11.6.18.)

Bennett. Separation of solids of different specific gravity etc. 19,830. July 9.

Bloomfield. Still. 18,108. July 2.

Bowrey, and Duckham and Co. Centrifugal separators. 18,739. July 5.

Chem. Fabr. vorm. Weiler-ter Meer. Centrifugal pumps for acids. 18,203 and 18,396. July 2. (Ger., 28.9.16 and 19.11.17.)

Clark. Pulverising, mixing, etc. apparatus. 19,455. July 8.

Continuous Reaction Co., Skelley, and Smith. Means for mixing granular etc. substances. 18,321. July 2.

Dobblestein. Production of briquettes. 20,011. July 9. (Ger., 21.3.14.)

Dorr Co. Selective recovery of organic materials from liquids. 19,267. July 7. (U.S., 1.7.19.)

Dow. Drying apparatus. 20,100. July 9.

Elektro-Osmose A.-G. Process for extracting materials. 18,521. July 3. (Ger., 3.7.19.)

Fairlie. Filling material for reaction spaces. 19,919. July 9. (U.S., 23.3.18.)

Freytag. Washing gases and vapours. 18,319. July 2. (Ger., 19.8.18.)

Granichstädten and Sittig. Hydration and production of catalysts. 19,511. July 8. (Austria, 7.7.17.)

Hutchins. Drying apparatus. 18,495. July 3.

Juer and Smeu. Apparatus for dissolving materials with acids etc. 20,025. July 9. (Austria, 20.12.15.)

Krupp A.-G. Acid-resisting pipes, boilers, etc. 18,030. July 2. (Ger., 17.6.18.)

Martini u. Humeke A.-G. Producing inert gases for fire-prevention. 19,579. July 8. (Ger., 23.9.16.)

Mazza. Separating elements of gaseous mixtures. 19,236. July 7. (Ital., 7.9.15.)

Müller. Devices for separating constituent parts of heterogeneous mixtures. 17,561. June 28. (Ger., 10.2.19.)

Norma Comp., and Schweickardt. Grinding-machines. 19,890-1. July 8. (Ger., 26.2 and 1.3.18.)

Norton Co. Grinding machines. 19,659—19,661. July 8. (U.S., 21.7.14 and 28.6.15.)

Petzel. Separation of gas mixtures. 20,214. July 9. (Ger., 9.1.14.)

Schmidt. Reaction towers. 19,290. July 7. (Ger., 11.4.19.)

Soc. l'Oxylythe. Washing and drying gases. 18,379. July 2. (Fr., 22.1.19.)

Soc. Anon. Entreprises Simon-Carves. Rotary furnaces. 20,273. July 9. (Fr., 4.6.18.)

Teichner. 18,394. See X.

United Filters Corp. Rotary suction filters. 17,538. June 28. (U.S., 24.5.17.)

Vivies. Apparatus for washing or extracting liquids. 19,745. July 8. (Fr., 26.3.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

13,032 (1919). Brinjes and Goodwin, and Harris. Mixing and/or agitating machines. (115,230.) July 7.

4032 (1920). Fabry. Continuous stills for dissociating chemical solutions by heat. (146,058.) July 14.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Akt.-Ges. f. Brennstoffvergasung. Treatment of coking coals for extraction of nitrogen. 20,164. July 9. (Ger., 17.11.16.)

Avray. Gasoline etc. producing apparatus. 19,631. July 8. (Ger., 11.12.13.)

Bale and Perkin. Apparatus for destructive distillation of carbonaceous material. 18,145. July 2.

Bates. Fuel. 18,914. July 6.

Berzlit Ges. Distillation of low-grade fuels. 20,317. July 9. (Ger., 26.10.18.)

Bronn., and Rombacher Hüttenwerke. Treatment of coke-oven gases. 18,709 and 19,015. July 5 and 6. (Ger., 24.10 and 21.11.14.)

Brown and Oswald. Apparatus for extracting oil from sandstone, shale, etc. 19,202. July 7.

Brownlee. Apparatus for treating hydrocarbon oils. 17,578. June 28. (U.S., 11.7.17.)

Burnell. Conversion of liquid hydrocarbons into motor spirit. 19,073. July 7.

Cannon. Carbonising. 19,262. July 7.

Collin A.-G. Sealing retorts and oven chambers. 18,166. July 2. (Ger., 28.4.19.)

Collin A.-G. Recovering by-products from fuel gases. 19,592. July 8. (Ger., 3.3.19.)

Coppée and Co. Regenerating furnaces. 19,741. July 8. (Belg., 20.1.19.)

Coppée and Co. Coking ovens. 19,742. July 8. (Belg., 28.4.19.)

Cordes. Converting heavy hydrocarbons into lower-boiling lighter products. 19,616. July 8. (Ger., 28.7.14.)

Cummins. Vertical gas-retort settings. 18,825. July 6.

Dempster, Knight, and Siddall. Combined coal carbonising and gas-making plant. 18,076. July 2.

Deuts. Erdöl A.-G., Koettwitz, and Seidenschmur. Obtaining lubricating oils from bituminous or asphaltic materials. 18,874-5. July 6. (Ger., 6.11 and 3.3.16.)

Evans, Hollings, Stanier, and South Metropolitan Gas Co. Removing sulphur from gases. 20,332. July 9.

Ges. f. Maschinelle Druckentwässerung. Drying raw peat. 17,612. June 28. (Ger., 2.1.14.)

Ges. f. Maschinelle Druckentwässerung. Utilising coal slimes. 17,613. June 28. (Ger., 2.1.14.)

Goldschmidt A.-G. Separate recovery of constituents of distillation gas from fuel. 18,266. July 2. (Ger., 26.10.17.)

Haersolte van Haerst. Coke-ovens. 19,308-10, 19,743-4. July 7 and 8. (Ger., 14.7, 15.8, 1.3.16, 31.3.17, 1.10.18.)

Hollings, and South Metropolitan Gas Co. Removing sulphur from gases. 20,333. July 9.

Hunt. 17,940-1 and 18,855-6. See XX.

Hyde. Steaming horizontal gas retorts. 18,102. July 2.

Klönne. Gas-producer furnaces. 17,781. June 29. (Ger., 12.4.20.)

Linck. Gas-producers. 20,166. July 9. (Ger., 2.2.18.)

Nelson. Cracking hydrocarbon oils etc. 18,524. July 3.

Pintsch A.-G. Gas-producers. 18,863 and 19,478. July 6 and 8. (Ger., 26.3.14 and 30.4.18.)

Standard Oil Co. Obtaining products from petroleum. 18,574. July 5. (U.S., 20.10.13.)

Standard Oil Co. Distilling hydrocarbons. 19,709. July 8. (U.S., 26.6.14.)

Still. Purifying coal gas. 19,518. July 8. (Ger., 10.12.17.)

Still. Separating constituents from coke-oven etc. gases. 19,740. July 8. (Ger., 1.11.18.)

Still. Recovery of ammonia from products of distillation of coal etc. 19,739. July 8. (Ger., 3.12.18.)

Still. 19,126. See VII.

Strache. Preparation of acids from petroleum etc. 19,958. July 9. (Austria, 20.1.17.)

Teichner. Oxidising hydrocarbons. 20,277. July 9. (Austria, 15.5.19.)

Verity. Gas-producers. 18,457. July 3.

Welford. Treatment of oil shale etc. 17,814. June 30.

COMPLETE SPECIFICATIONS ACCEPTED.

21,389 (1918). Colman and Yeoman. Extraction and recovery of carbon bisulphide. (145,099.) July 7.

1425-6 (1919). Perry. Apparatus for distilling carbonaceous material. (145,101-2.) July 7.

4576 (1919). Hood. Purification of petroleum oils. (145,818.) July 14.

7182 (1919). Harger. Gas-producers. (145,136.) July 7.

7898 (1919). Antrobus. Gas-producers. (145,164.) July 7.

7986 (1919). Brockway. Apparatus for washing and scrubbing coal gas. (145,856.) July 14.

8193 (1919). Spicer (Wells). Gas-producers. (145,873.) July 14.

8627 (1919). Forward. Process of refining oil by distillation. (145,894.) July 14.

9686 (1919). Rock. Extracting hydrocarbons from coal etc. (145,906.) July 14.

9722 and 16,961 (1919). Forwood and Taplay. Treatment of hydrocarbons. (145,198.) July 7.

13,733 (1919). Gibson and Wyman. Production of enriched water-gas. (145,947.) July 14.

15,124 (1919). U.S. Industrial Alcohol Co. Motor fuel for aeroplane and other motors. (128,915.) July 14.

728 (1920). Crowther. Furnace or kiln for burning spent iron oxide. (145,366.) July 7.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

British Dyestuffs Corporation, Green, Napier, and Porter. Manufacture of phthalic acid and phthalic anhydride, and catalyst for use therein. 19,210. July 7.

Farbw. vorm. Meister, Lucius, u. Brüning. Manufacture of pyridine bases. 18,758, 18,943, 19,128. July 5, 6, 7. (Ger., 22.6.17, 30.8 and 23.12.18.)

Farbw. vorm. Meister, Lucius, u. Brüning. Manufacture of anthraquinone derivatives. 18,944 and 19,519. July 6 and 8. (Ger., 11.7.18, 11.3.20.)

Friederich. Manufacture of trinitroresorcin. 18,362. July 2.

Juer and Smeu. Machine for nitrating etc. 20,026. July 9. (Hungary, 18.11.16.)

Melamid. Converting tar oils into neutral oils for lubricating. 18,833. July 6. (Ger., 11.3.19.)

Phillipson. 19,913. See XIX.

Schroeter. Hydrogenation of naphthalene. 19,394, 19,409, 19,449. July 8. (Ger., 24.2.15, 7.12.15, 13.5.16.)

Schroeter, and Tetralin Ges. Hydrogenation of naphthalene. 19,514 and 19,750. July 8. (Ger., 1.8.16.)

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

A.-G. f. Anilinfabr. Manufacture of dyestuffs of the acridine series. 18,202. July 2. (Ger., 22.4.15.)

British Dyestuffs Corp., Levinstein, and Imbert. Manufacture of phenylglycine or compounds thereof. 18,278. July 2.

Cassella und Co. Manufacture of vat dyestuffs. 19,693-4. July 8. (Ger., 4.7.18 and 24.3.19.)

Cassella und Co. Manufacture of a colour of the anthraquinone series. 20,256. July 9. (Ger., 15.3.15.)

Farbw. vorm. Meister, Lucius, u. Brüning. Manufacture of azo dyestuffs. 18,760-1. July 5. (Ger., 30.6 and 23.4.14.)

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

- Carmuel (Bayer u. Co.). Protecting wool etc. from moth. 17,607. June 28.
 Cellon-Werke. Production of moulded articles from cellulose acetate. 19,957 and 20,278. July 9. (Ger., 25.1.19.)
 Deutsch-Koloniale Gerb- u. Farbstoff-A.-G. 19,154. See XV.
 Enge. Manufacture of fibrous material from wood etc. 18,503. July 3.
 Ges. f. Verwertung Chem. Produkte. Obtaining balloon materials etc. 17,638. June 29. (Ger., 21.8.17.)
 Hottenroth and others. Treatment of artificial threads etc. 19,315. July 7. (Ger., 7.10.18.)
 Hottenroth and others. 19,314. See XVII.
 Humphrys. Treating flax straw. 17,616. June 28.
 Jeroch and others. Utilising spent sulphite cellulose lyes. 18,477. July 3. (Ger., 24.1.17.)
 Johnston and Sutherland. Degumming and cleansing vegetable matter from fibres. 19,412. July 8.
 Moeller. Treatment of cellulose. 17,776-7. June 29. (Ger., 22.8.18 and 24.2.19.)
 Müller. Manufacture of artificial threads, sheets, etc. from viscose. 17,806. June 28. (Ger., 6.9.18.)
 Soc. Suisse des Ferments. Degumming textile materials. 17,721. June 29. (Fr., 30.6.19.)
 Verwertung Inlandischer Produkte Ges. Treating water reeds etc. for textile purposes. 19,460. July 8. (Ger., 20.12.16.)
 Wohl. 18,528 and 18,744. See XVII.
 Wolkinson. Treatment of vegetable fibre. 19,072. July 7. (Fr., 17.4.19.)
 Zellkoll Ges. Sizing paper pulp. 18,495. July 6. (Ger., 11.8.16.)
 Zellkoll Ges. Sizing paper, pasteboard, etc. 18,949. July 6. (Ger., 7.12.15.)
 Zellstoff-fabrik Waldhof. Treatment of wood etc. 19,313. July 7. (Ger., 19.10.16.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 8559 (1914). Govaerts and Dryepont. Treatment of ramie, flax, hemp, etc. plants. July 7.
 29,043 (1919). Watremez. Securing vegetable fibres to accelerate and facilitate subsequent bleaching. (135,516.) July 7.

VI—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

- Bennert. Preparation of dye-vats. 17,945. June 30. (Ger., 21.12.15.)
 Boardman and Petrie. Dyeing apparatus. 17,823. June 30.
 Carl, and Jager Ges. Producing marbled effects on textiles. 20,097. July 9. (Ger., 13.5.19.)
 Farb. vorm. Meister, Lucius, u. Brüning. Printing with insoluble dyestuffs. 19,129. July 7. (Ger., 15.1.19.)
 Freiburger. Treatment of fabrics. 17,503. June 28. (Ger., 13.2.17.)
 Kaufmann. Apparatus for dyeing linen etc. 20,056. July 9. (Fr., 18.10.13.)
 Mohr and others. Bleaching-device. 20,250. July 9. (Ger., 14.9.16.)
 Ornstein. Bleaching vegetable substances. 19,042. July 6. (U.S., 2.12.16.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 6817 (1919). Hunt. Apparatus for bleaching treatment of open fabrics. (145,113.) July 7.
 14,232 (1919). Calico Printers' Assoc., Rouse, and Nelson. Printing cotton fabrics and yarns. (145,240.) July 7.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

- Aragay and Bonneau. Simultaneous production of caustic soda and hydrochloric acid. 18,267. July 2. (Spain, 30.1.19.)
 Bea. Manufacture of copper sulphate. 19,675. July 8. (Fr., 3.8.17.)
 Bea. 20,316. See X.
 Bengough. Opening up water-insoluble compounds of the alkalis, alkaline-earths, metals, etc. 18,311. July 2. (Ger., 23.2.18.)
 Chem. Fabr. Rhénania, Grob, and Stuer. Manufacture of nitrogen-containing products from acetylene and ammonia. 19,039. July 6. (Ger., 20.11.13.)
 Clerc and Nihoul. 18,398. See X411.
 Collin A.-G. Producing ammonium sulphate from cyanogen compounds. 17,974. June 30. (Ger., 17.5.19.)
 Coppet. 19,630. See XIII.
 Dempster, Knight, and Siddall. Plant for making hydrogen. 18,438. July 3.
 Dumont. Lime kilns etc. 17,855. June 30.
 Forster und Co. Manufacture of sulphuric acid. 19,121. July 7. (Ger., 24.6.18.)
 Frischer. Obtaining oxides of nitrogen from ammonia. 17,522. June 28. (Ger., 23.11.16.)
 Heibronner. 20,269. See XIII.
 Howorth (Akt. Høyangsfaldene Norsk Aluminium Co.). Preparation of aluminium oxide. 17,765. June 29.
 Jaubert. Manufacture of hydrogen. 19,483. July 8. (Fr., 9.3.18.)
 Kersten. Decomposition of alkali chlorides. 19,459. July 8. (Ger., 10.5.16.)
 Mitsubishi Kogyo Kabushiki Kaisha. Synthetic production of cyanides. 18,674. July 5. (Japan, 7.7.19.)
 Mond (Metallbank u. Metallurg. Ges.). Production of zinc oxide. 19,959. July 9.
 Robertson. Obtaining volatilisable metal oxides. 19,388. July 8. (U.S., 16.12.18.)
 Sonneck, and Unione Italiana fra. Consum. o Fabric. di Concimi e Prod. Chimici. Manufacture of sulphuric acid. 18,746. July 5.
 Still. Treating crude gas liquor to obtain ammonia solution. 19,126. July 7. (Ger., 6.12.15.)
 Still. 19,739. See II.
 Verein Chem. Fabr. Mannheim. Manufacture of hydrofluoric acid. 19,704. July 8. (Ger., 27.12.18.)
 Verein Chem. Fabr. Mannheim. Manufacture of sulphur dioxide. 19,705. July 8. (Ger., 8.8.19.)
 Westling. Production of manganese dioxide. 18,979. July 6. (U.S., 14.3.18.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 7403 (1919). Gould (Balfour-Guthrie Investment Co.). Producing cyanogen and ammonia. (145,824.) July 14.
 20,042 (1919). Thorssell and Lunden. Apparatus for making nitrogen products. (145,299.) July 7.
 26,253 (1919). Norsk Hydro-Elektrisk Kvaestof-aktieselskab. Production of alumina. (134,531.) July 14.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

- Feldenheimer, Plowman, and Schidrowitz. Treatment of clay. 20,339. July 9.
 Frink. Glass-melting furnaces. 18,118 and 18,136. July 2.
 Frink. Glass-annealing furnaces. 18,163. July 2.
 Grimwade, and Grimwades, Ltd. Composition and process for forming pottery moulds. 19,059. July 6.
 Kern. 18,755. See XI.
 King, King, and King and Son. Enamels. 17,519. June 28.

Moorshead. Glass furnaces. 19,427. July 8.
Pollak (Werke Gantenbach). Production of a substitute for black glass and insulating material. 17,750. June 29.

Rhodin. Preparation of sands for bleaching. 19,246. July 7.

Rosenthal. Colouring glass. 18,577. July 5. (U.S., 21.1.14.)

Schott u. Gen. Reducing size of bubbles in glass. 19,445. July 8. (Ger., 7.12.14.)

IX.—BUILDING MATERIALS.

APPLICATIONS.

Blériot. 19,749. *See* XI.
Bub. Preserving wood. 19,553 and 20,039. July 8 and 9. (Ger., 7.2.14 and 19.12.13.)

Crozier. Manufacture of cementitious articles. 17,566 and 17,730. June 28 and 29.

Dossetter. Wood substitutes etc. 19,725. July 8.

Link. Production of artificial trass. 17,693. June 29. (Ger., 5.11.18.)

Marcusson. Oil-proof concrete bodies. 17,944. June 30. (Ger., 1.7.18.)

Wade (Bombrini Parodi-Delfino). Manufacture of cements. 18,736-8. July 5.

COMPLETE SPECIFICATION ACCEPTED.

6004 (1916). Sanders and Sanders. Coating or impregnating cement, stone, bricks, etc. (145,806.) July 14.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Ampère Ges. Production of ferro-tungsten. 19,363. July 7. (Ger., 6.5.18.)

Ampère Ges. Production of ferro-chromium poor in carbon. 19,364. July 7. (Ger., 6.5.18.)

Avestaran é Irazusta. Conglomerating pulverulent mineral residues. 17,690. June 29.

Bea. Treatment of old brass to obtain copper and oxide of zinc. 20,316. July 9. (Fr., 3.8.17.)

Bensa. Medium for preventing oxidation of iron etc. 19,841. July 9. (Ital., 5.11.18.)

Bonnard. Refining tin and antimony. 18,179. July 2. (U.S., 15.3.20.)

Boorne. Electrode for depositing molten metal. 17,901. June 30.

British Thomson-Houston Co. Production of metal films. 20,032. July 9. (U.S., 6.6.14.)

Dagory. Removing copper deposits from bores of guns. 20,181 and 20,200. July 9. (Fr., 19.6 and 11.10.17.)

Difenthaler. Production of grey cast iron. 19,988. July 9. (Ger., 9.5.16.)

Elmore. Extraction of lead and zinc from ores. 19,295. July 7.

Gauschemann. Smelting - furnaces. 20,007. July 9. (Ger., 9.9.15.)

Goldberg. Electrolytically working concentrated ores or alloys containing copper and nickel. 17,760. June 29. (Ger., 9.7.18.)

Grinlinton. Coating metals. 18,692. July 5. (Australia, 4.7.19.)

Hackspill and Staehling. Manufacture of alkali metals. 20,023. July 9. (Fr., 5.11.13.)

Lancaster. Removing iron etc. from residues or waste. 18,531. July 5.

Lance. Extraction of metal in the form of oxide from pyritic ores. 18,380. July 2. (Fr., 16.5.16.)

Maddick and Williams. Manufacture of zine powder. 19,260. July 7.

Manuf. de Prod. Chimiques du Nord. Removal of dust from gases from ore-roasting. 18,964. July 6. (Ger., 6.5.14.)

Marks (Ore Roasting Development Co.). Treating ores. 18,967. July 6.

Maschinenfabr. Esslingen. Protecting iron alloys against burning when smelted. 17,525. June 28. (Ger., 19.11.17.)

Metallbank u. Metallurg. Ges. Lithium-aluminium alloy. 19,956. July 9. (Ger., 15.2.19.)

Metallbank u. Metallurg. Ges. Furnace for annealing metals. 20,156. July 9. (Ger., 14.3.19.)

Metallind. Schiele u. Bruchsal. Production of aluminium castings. 18,712 and 19,720. July 5 and 8. (Ger., 16.12.18, and 19.3.19.)

Milliken. Alloys. 19,013-4. July 6.

Otsuka. Apparatus for concentrating ores by flotation. 18,378. July 2. (Ger., 30.12.17.)

Schaufelberg. Compound for hardening iron and steel. 18,300. July 2.

Schlotter. Electrolytic production of tin deposits. 20,248. July 9. (Ger., 15.3.17.)

Tashero. Process for carburising steel and iron. 18,628. July 5. (Japan, 13.8.18.)

Teichner. Manufacture of nickel catalyst. 18,394. July 2. (Ger., 10.11.16.)

Zilewski. Zinc-extracting furnaces. 20,131. July 9. (Ger., 28.3.16.)

COMPLETE SPECIFICATIONS ACCEPTED.

7111 (1919). Bloxam (Soc. Anon. des Usines Guilini). Increasing hardness and tenacity of metals. (145,129.) July 7.

7956 (1919). Wood, and Minerals Separation, Ltd. Concentration of ores. (145,852.) July 14.

8165 (1919). Minerals Separation, Ltd. (Emerson). Concentration of ores. (145,870.) July 14.

11,688 (1919). Sulman and Ballantine. Production of ferro-alloys. (145,925.) July 14.

14,094 (1919). Farmer. Alloy. (145,956.) July 14.

21,697 (1919). Gaskill. Crucible and like furnaces. (132,266.) July 14.

24,731 (1919). Stenquist. Metal alloy for armouring electric cables. (136,143.) July 7.

24,851 (1919). McLain and Carter. Open-hearth furnaces. (146,020.) July 14.

1829 (1920). Honhorst and Sofge. Steel alloys. (138,327.) July 14.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Blériot, Ltd. Manufacture of electrical insulating cement. 19,749. July 8. (Fr., 8.11.17.)

Boorne. 17,901. *See* X.

Brown. Electrostatic separation of finely divided discrete material. 19,159. July 7.

Champion Ignition Co. Insulating material. 18,843. July 6. (U.S., 22.8.17.)

Fery. Primary batteries. 19,942. July 9. (Fr., 1.12.14.)

Gef. f. Torfisolation. Manufacture of insulating bodies from peat. 20,103. July 9. (Ger., 31.8.15.)

Goldberg. 17,760. *See* X.

Kern. Manufacture of fireproof insulating material from clay etc. 18,755. July 5.

Miles. Electric furnaces. 18,958-9. July 6. (U.S., 25.1.18.)

Mond (Internat. Precipitation Co.). Apparatus for electrical treatment of gases. 17,744. June 29.

North. Electrically separating dust from gases and vapours. 17,723. June 29. (Ger., 27.8.17.)

Pollak. 17,750. *See* VIII.

Rickets. Electrically inducing chemical action. 18,627 and 19,549. July 5 and 8.

Schlotter. 20,248. *See* X.

COMPLETE SPECIFICATIONS ACCEPTED.

8795 (1919). Steiger. Electric furnaces. (125,391.) July 14.

18,004 (1919). Piquerez. Electric furnace. (132,232.) July 14.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Blichfeldt. 17,784. *See* XIX.

Dorr Co. Treatment of soapy liquors. 19,251. July 7. (U.S., 1.7.19.)

Eisenstein, and Schlicht A.-G. Oxidation of oils. 19,634 and 20,202. July 8 and 9. (Czecho-Slov., 3 and 16.4.19.)

Elektro-Osmose A.-G. Purifying glycerin etc. 18,752. July 5. (Ger., 17.1.19.)

Faitelowitz. 20,070. *See* XV.

Imhausen. Production of soap powder. 18,879. July 6.

Imhausen. Washing medium. 20,213. July 9. (Ger., 10.12.15.)

Miles. Manufacture of emulsified products. 17,762. June 29. (U.S., 10.1.19.)

Reichsausschuss f. Pflanzliche u. Tierische Oele u. Fette Ges. Production of fat. 18,478. July 3. (Ger., 10.5.15.)

Rogers. Extraction of oils from fatty substances. 19,883. July 9. (U.S., 7.3.19.)

Soc. Rocca, Tassy, et de Roux. Neutralising oils and fats. 17,539 and 17,540. June 28. (Fr., 13.11.19 and 3.5.20.)

Soc. Gén. d'Evaporation. Treating residual waters containing fatty and soapy matters. 19,002. July 6. (Fr., 9.3.14.)

Soc. Gén. d'Evaporation. Decomposition of soapy waters. 19,003. July 6. (Fr., 9.3.14.)

Tseng. Manufacture of transparent soap 18,050. July 2.

Wado (Ordbrown). Treatment of oils and fats. 18,399. July 2.

Watkins (Egestorff). 18,461. *See* XIX.

Wilbuschewitsch. Continuous extraction of oil etc. 19,748. July 8. (Gr., 12.6.19.)

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Badische Anilin u. Sodafabr. Manufacture of artificial resins. 18,617. July 5. (Ger., 13. 6.19.)

Bucherer. Production of derivatives of condensation products of formaldehyde and phenols. 20,041 and 20,286. July 9. (Ger., 10.6.18 and 22.3.19.)

Clere and Nichoul. Manufacture of zinc sulphide. 18,398. July 2. (Fr., 20.5.19.)

Coppet. Manufacture of anhydrous zinc sulphide. 19,630. July 8. (Fr., 11.9.16.)

Dahl. Apparatus for manufacture of white lead. 18,079. July 2.

Filhol. Manufacture of varnishes etc. 19,238. July 7. (Fr., 26.6.19.)

Helbronner. Manufacture of zinc sulphide. 20,269. July 9. (Fr., 16.10.16.)

Jerome. Rendering painted materials impervious to moisture. 18,900. July 6. (Ital., 13.6.19.)

Lance. Manufacture of ultramarine blue and lacquered colours. 18,389. July 2. (Fr., 15.3.19.)

Miles. Manufacture of products from resins. 17,762. June 29. (U.S., 10.1.19.)

Mond. 19,959. *See* VII.

Nishizawa and Shibata. Drying Japanese lacquer. 18,370. July 2. (Japan, 29.4.19.)

Plöniss u. Co. Preparation of fireproof and waterproof paint. 19,828. July 9. (Ger., 16.9.16.)

Thompson. Making white lead. 19,755. July 8. (Ger., 13.10.14.)

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Goodyear Tire and Rubber Co. Manufacture of rubber products. 18,915 and 18,919. July 6. (U.S., 29.5.17 and 25.10.18.)

Haddan (Waitz). Recovering caoutchouc and textile from rubber fabric. 20,050. July 9.

Kendall and Thomson. Preparation and manipulation of rubber prior to vulcanisation. 18,421. July 3.

Tilche. Vulcanisation of rubber. 20,267-8. July 9. (Fr., 18 and 11.7.19.)

Wade (Goodyear Tire and Rubber Co.). Manufacture of rubber products. 18,916 and 18,920. July 6.

Wade (Goodyear Tire and Rubber Co.). Process of vulcanising caoutchouc. 18,917-8. July 6.

Waitz. Recovering rubber and textile material from rubber fabric. 19,585. July 8. (Ger., 5.7.17.)

COMPLETE SPECIFICATION ACCEPTED.

15,121 (1919). Marks (Acushnet Process Co.). Reclaiming rubber etc. from fabrics coated therewith. (145,251.) July 7.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Badische Anilin u. Sodafabr. Manufacture of tanning preparations. 18,467. July 3. (Ger., 19.2.19.)

Breuer. Manufacture of leather. 18,083. July 3. (Ger., 14.12.17.)

Carmichael and Ockleston. Unhairing hides. 18,006. July 2.

Chem. Fabr. Worms. Manufacture of tanning agents. 20,027. July 9. (Ger., 20.7.16.)

Czechon. 19,477. *See* XIX.

Deutsch-Koloniale Gerb- u. Farbstoff A.-G., and Römer. Obtaining tanning materials from cellulose waste sulphite lyes. 19,154. July 7.

Faitelowitz. Extracting albumen and fat from bones. 20,070. July 9. (Ger., 13.2.19.)

Knorr. Glues. 20,139 and 20,140. July 9. (Ger., 13.8.17 and 20.9.18.)

Korn. Leather substitute. 19,964. July 9. (Fr., 9.5.19.)

Tatham. Manufacture of tanned and rubberised leather. 18,763. July 5.

Vos. 19,827. *See* XVI.

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Akt.-Ges. f. Anilinfabr. Manufacture of artificial fertilisers. 17,608 and 17,720. June 28 and 29. (Ger., 29.1 and 16.3.18.)

Chem. Fabr. Rhenania, and Voerkelius. Manufacture of manures. 20,170. July 9. (Ger., 31.5.18.)

Chem. Fabr. Rhenania, and Voerkelius. Nitric acid superphosphate. 20,171. July 9. (Ger., 12.4.19.)

Lidholm and others. Producing cyanamide from calcium cyanamide. 17,983. June 30. (Sweden, 19.9.19.)

Vos. Manufacture of manures from waste leather. 19,827. July 9. (Belg., 17.12.13.)

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

Betavit Ges. Preparation of sugar beets etc. 19,707. July 8. (Ger., 13.12.17.)

Hottenroth and others. Obtaining sugar etc. from wood etc. 19,314. July 7. (Ger., 18.4.17.)

Wohl. Converting cellulose materials into soluble carbohydrates. 18,526. July 3. (Ger., 14.3.17.)

Wohl. Saccharifying cellulose-containing materials. 18,741. July 5. (Ger., 7.4.17.)

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Dubourg. Brewing beer. 18,333. July 2. (Fr., 18.1.19.)

Fleischmann Co. Production of yeast. 20,293. July 9. (U.S., 7.1.19.)

Fleischmann Co., and Harrison. Production of yeast. 20,294. July 9.

Scherdel and Wohl. Production of yeast. 17,797. June 29. (Ger., 15.1.15.)

Vasseux. Production of yeast from peat. 19,515. July 8. (Fr., 19.1.18.)

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Beaumont, Goudie, and Harkness. Making of jams, jellies, etc. 18,890. July 6.

Bielmann and Bielmann. Manufacture of jellies from fruits etc. 19,887 and 20,352. July 9. (Ger., 24.11.16 and 14.3.19.)

Blichfeldt. Manufacture of margarine. 17,781. June 29.

Czochron. Obtention of albumen from leguminous seeds. 19,477. July 8. (Ger., 30.5.19.)

Elektro-Osmose A.-G. (Graf Schwerin Ges.). 17,609. See XX.

Elektro-Osmose A.-G. (Graf Schwerin Ges.). Process for obtaining the yellow and white constituents of eggs in a dry state. 18,947. July 6. (Ger., 9.1.18.)

Matzka. Utilisation of fruit etc. refuse. 19,881. July 9. (Ger., 8.6.18.)

Philpison. Coal tar disinfectants. 19,913. July 9. (U.S., 15.3.18.)

Ricciardi. Drying-kilns for alimentary pastes etc. 17,876. June 30.

Sykes. Apparatus for dehydrating food products. 18,852. July 6. (U.S., 4.2.18.)

Townsend. Manufacture of food. 19,605. July 8.

Vollbrot Patent Verwertungs-Ges. Process for manufacture of baked articles direct from corn. 18,344. July 2. (Ger., 3.10.14.)

Vollbrot Patent Verwertungs-Ges. Manufacture of food. 18,345. July 2. (Ger., 8.9.17.)

Vollbrot Patent Verwertungs-Ges. Apparatus for manufacture of baked articles direct from corn. 19,041. July 6. (Ger., 9.11.15.)

Watkins (Egestorff). Process for manufacture and preservation of cattle food, fish-livers, and oil therefrom. 18,461. July 3.

Wolff. Production of germ-free air. 19,499. July 8. (Ger., 12.9.17.)

COMPLETE SPECIFICATION ACCEPTED.

19,049 (1919). Adams. Separation of sludge and scum from sewage and other liquids. (145,291.) July 7.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Akt.-Ges. f. Anilin-Fabr. Manufacture of oxyarylaldehydes. 17,719. June 29. (Ger., 6.2.18.)

Bader, British Cellulose and Chemical Manufacturing Co., and Nightingale. Manufacture of aromatic derivatives. 18,114. July 2.

Bader, British Cellulose and Chemical Manufacturing Co., and Nightingale. Manufacture of alkyl derivatives. 18,115. July 2.

Bergius, and Goldschmidt A.-G. Production of ethylene chloride. 19,963. July 9. (Ger., 29.2.16.)

Boehringer Sohn. Process for obtaining effective ingredients of lobelia. 17,795-6. June 29. (Ger., 4.4 and 2.9.14.)

British Dyestuffs Corporation, and others. 19,210. See III.

Du Pont de Nemours and Co. Process for production of alkyl anilines. 18,085. July 2. (U.S., 7.7.17.)

Elektro-Osmose A.-G. (Graf Schwerin Ges.). Process for preparing proteins charged with immune substances. 17,609. June 28. (Ger., 11.7.18.)

Elektro-Osmose A.-G. (Graf Schwerin Ges.). Process for purifying glycerin etc. 18,752. July 5. (Ger., 17.1.19.)

Ellis (Soc. Chim. Usines du Rhône, anc. Gilliard, Monnet et Cartier). Manufacture of oxalaldehydes etc. 19,208. July 7.

Faitelowitz. 20,070. See XV.

Farbw. vorm. Meister, Lucius, n. Brüning. Manufacture of arseno compounds of the pyrazolone series. 18,759. July 5. (Ger., 15.12.17.)

Goldschmidt A.-G., and Matter. Production of ethylene chloride. 19,962. July 9. (Ger., 22.5.15.)

Grunstein. Manufacture of butyric aldehyde and butyl alcohol from erotonic aldehyde. 19,152. July 7. (Switz., 4.1.19.)

Grunstein. Manufacture of aldol from acetaldehyde. 19,153. July 7. (Switz., 18.3.19.)

Hunt. Production of reactive acid liquor, alcohols, esters, etc. from gaseous hydrocarbons. 17,940. June 30.

Hunt. Production of esters etc. from olefines. 17,941. June 30.

Hunt. Conversion of secondary alcohols into ketones. 17,942. June 30.

Hunt. Production of alcohols, esters, etc. from still gases of petroleum. 18,855. July 6. (U.S., 20.3.17.)

Hunt. Production of alcohols, esters, etc. from gaseous hydrocarbons. 18,856. July 6. (U.S., 11.3.19.)

Imray (Soc. of Chem. Ind. in Basle). Manufacture of aralkyl esters of 2-phenylquinoline-4-carboxylic acid. 18,756. July 5.

Kolshorn. Process for manufacture of salts of compounds of tannic or gallic acids and lactic acid. 17,554. June 28. (Ger., 13.6.19.)

Kolshorn. Manufacture of derivatives of *p*-aminophenol and of its *O*-alkyl ethers. 17,780. June 29. (Ger., 13.6.19.)

Matter. Production of polyvalent alcohols. 19,960-1. July 9. (Ger., 8.12.13 and 21.4.15.)

Tweedale and Tweedale. Manufacture of perfume and flavouring essences etc. 17,860. June 30.

Welmer. Manufacture of fumaric acid. 18,400. July 2. (Ger., 28.9.15.)

White (U.S. Sanitary Specialties Corp.). Decolorising materials. 18,351. July 2.

COMPLETE SPECIFICATION ACCEPTED.

8173 (1919). Confectionery Ingredients, Ltd., Matthews, King, and Kane. Manufacture of proto-catechuic aldehyde. (145,871.) July 14.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATION.

Shepherd. Method of obtaining a yellow separation print in colour photography. 17,892. June 30.

XXII.—EXPLOSIVES; MATCHES.

APPLICATIONS.

Buck. Liquid air for use as an explosive. 20,118. July 9.

Dubrisay. Manufacture of damp-proof matches. 18,195. July 2. (Fr., 3.7.19.)

Pinder and Semple. Manufacture of explosive. 19,115. July 7.

Von Herz. Explosives. 18,183. July 2. (Austria, 25.1.19.)

Wohl. Explosives. 17,605, June 28. 17,749, June 29. 18,711, July 5. (Ger., 14.1. and 1.3.19, and 9.1.20.)

XXIII.—ANALYSIS.

APPLICATIONS.

Siemens u. Halske A.-G. Photometric apparatus. 19,191. July 7. (Ger., 8.7.19.)

Simmance. Calorimetric apparatus. 18,762. July 5.

I.—GENERAL; PLANT; MACHINERY.

Treating volatile liquids with other liquids; Process for —. [Refining petroleum with sulphuric acid.] P. Mallet. Chim. et Ind., 1920, 3, 608.

To avoid loss of the volatile liquid when treated with another liquid with air agitation, and to prevent formation of explosive vapours, the process is carried out in a closed vessel. The air-vapour mixture may be utilised again for agitation, any corrosive effect of the vapours being overcome by passing the mixture through suitable absorbers before re-introducing it into the reaction apparatus.—W. J. W.

PATENTS.

Absorption, reaction, distillation, rectification, and like columns. L. Gay. E.P. 141,371, 7.3.19.

The plates of the column are traversed by two sets of vertical tubes, for passage of liquid and gas or vapour respectively, so distributed that on one and the same straight line there are alternately two gas tubes and one liquid tube, the former being located at the angles and the latter at the centres of regular hexagons. The gas tubes project from the upper surface of the plate; each is covered with a hood provided with openings in contact with the plates, while the liquid tubes project from the two surfaces of the plate, the portion projecting from the upper surface being slightly shorter than the gas tubes, while the portion projecting from the lower surface extends almost to the next lower plate. A common sleeve surrounds the lower part of a liquid tube of an upper plate and the upper part of a liquid tube of a lower plate, the sleeves being open at their upper ends and provided towards the top with long narrow vertical slots, and in their lower part with openings where they join the lower plates. The space between the plates is provided with filling material up to the level of the upper ends of the slots in the sleeves surrounding the liquid tubes.—J. S. G. T.

Filter. D. A. Corey. U.S.P. 1,295,006, 18.2.19. Appl., 7.5.17.

The filter is built up of a number of superposed sections each having a central tubular collar. A screen fits over the collar and extends radially outwards, the outer edge having an upright portion. A dished plate is also supported by the collar and extends outwards and upwards away from the screen; it is bent downwards at the circumference forming a perforated flange outside the screen. A filtering element is placed below each screen. Liquid enters from the interior of the collar, passes through perforations in the collar and upwards through the filter, into the chamber next above, and thence outwards through the perforated rim of the dished plate. The sections are easily separated for cleaning.—W. F. F.

Filter-press. H. T. Shriver. U.S.P. 1,342,829, 8.6.20. Appl., 22.9.19.

Is a plate and frame filter press in which each alternate plate is connected with a wash channel, automatic non-return valves are provided in the plates to prevent liquor flowing into the wash channel in the direction opposite to the wash water.—B. M. V.

Filter [press]. E. F. Atkins, Assr. to H. T. Shriver. U.S.P. 1,342,839, 8.6.20. Appl., 23.6.19.

Each plate of a plate and frame filter-press is provided with an outlet leading to the exterior, and a closed conduit extends along the plates and frames. By means of a multiple-way valve on each plate, the outlet to the exterior of the plate may be

opened and communication prevented between the outlet and the conduit; the outlet to the exterior of the plate may be closed, and the outlet connected with the conduit; or the outlet may be closed and communication between it and the conduit prevented.—J. S. G. T.

Filter. E. J. Sweetland, Assr. to United Filters Corp. U.S.P. 1,343,182, 8.6.20. Appl., 15.3.15. Renewed 6.6.19.

The filter itself is supported independently of its casing, which is formed in sections, and means are provided for moving the sections towards and away from the filter.—B. M. V.

Tunnel kiln. G. H. Benjamin. U.S.P. 1,294,756, 18.2.19. Appl., 21.5.17. Renewed 10.7.18.

The kiln combines the features of the "direct fire" and the "muffle" types, and means are provided for regulating the temperature at various points along its length. Two pairs of hot blast stoves are arranged one on either side of the kiln at the middle of its length, and hot air is delivered from them into longitudinal flues and thence through ports into the kiln. Combustion chambers are arranged on the inner side of the kiln walls, near the middle, and means may be provided for directing the combustion products through one of each of the opposed pairs of hot blast stoves to produce a still higher temperature at the middle zone of the kiln. Any desired variation in temperature along the length of the kiln may be produced by providing electrical heaters in the air supply flues and bringing one or more into operation. The passage of current through the heating coils may be controlled automatically by the movement of a truck through the kiln.—W. F. F.

Liquids [oil-water emulsions]; Separation of —. C. W. McKibben. U.S.P. 1,296,988, 11.3.19. Appl., 17.8.18.

An apparatus for separating the constituents of an emulsion, such as oil and water, comprises a tall, vertical cylinder provided with a fixed electrode composed of a series of wires forming an inverted cone, and a similar inner rotating electrode mounted on a concentric vertical shaft. The outer electrode and the casing are connected to one of the terminals of a high potential transformer of constant current type, and the inner electrode to the other terminal. The emulsion is passed continuously through the apparatus from top to bottom, and the separated liquids are drawn off through an outlet at the side, near the bottom. If a series of such separators be employed in parallel with a single transformer, the current, which is of predetermined value, is automatically divided between the separators as required by the condition of the liquid in each separator.

—W. F. F.

Pulverising frangible substances. G. A. Overstrom. U.S.P. 1,300,192, 8.4.19. Appl., 21.8.16.

MATERIAL to be pulverised is fed continuously into a rapidly rotating receptacle mounted on the upper end of a vertical shaft. Under the action of centrifugal force the material accumulates against the sides of the receptacle, leaving an inverted conical space in the centre, and any further material received then rides over the surface of the accumulated material, and is discharged over the rim of the receptacle. Wear of the rotating receptacle, except the rim, is thus avoided, and the rim is renewable when required. The material is thrown against an outer ring, having internal teeth, and is thereby pulverised, and, to prevent unequal wear of the ring, it may be partly rotated at intervals. The ring is built up of several superposed similar sections, so that when the central section becomes

worn the sections may be rearranged. Rotation of the material accumulated in the receptacle relatively to the latter is prevented by radial ribs below the normal surface of the material.—W. F. F.

Mixing and grinding apparatus. R. D. Maddox. U.S.P. 1,301,070, 15.4.19. Appl., 26.11.18.

A GRINDING receptacle is constructed of a distorted cylindrical shape. The two ends are elliptical, with the major axes of the two ellipses at right angles to one another, and the side walls of the cylinder form a continuous surface between the two ellipses. A number of such cylinders rest with their peripheral surfaces on a pair of long, horizontal supporting-rollers, so that when the rollers are rotated the receptacles are also rotated and simultaneously rocked from end to end so as to mix and grind the material which is placed within them.

—W. F. F.

Centrifugal dryer. G. H. Elmore. U.S.P. 1,342,743, 8.6.20. Appl., 8.8.18.

THE basket of the rotor is double-walled, the inner wall being solid and the outer perforated. The material passes from an axial feed inlet through passages to the annular space, being assisted by plungers working across the outer ends of the passages.—B. M. V.

Pump [for corrosive liquors]. D. Farrand. U.S.P. 1,342,798, 8.6.20. Appl., 23.10.17.

A U-SHAPED pipe is filled in the bend and some distance up each limb with a liquid of greater sp. gr. than the liquid to be pumped. At the top of one limb is a trunk piston with a layer of oil under it, and at the top of the other limb are inlet and outlet valves and connexions for the liquid to be pumped.—B. M. V.

Separating suspended matter from gases; Means for —. W. A. Schmidt, Assr. to International Precipitation Co. U.S.P. 1,343,285, 15.6.20. Appl., 5.3.13.

THE apparatus comprises a longitudinal ionising portion, a charging electrode, and a collecting field-surface member, the field surface being substantially parallel to the ionising chamber.

—J. S. G. T.

Separating suspended particles from gases; Apparatus for —. W. A. Schmidt and G. C. Roberts, Assrs. to International Precipitation Co. U.S.P. 1,343,482, 15.6.20. Appl., 23.3.14.

A COLLECTING electrode presenting a relatively extended surface is arranged within a precipitation chamber parallel to the direction of the gas current therein. The collecting electrode may consist of a perforated screen or a number of screens arranged in parallel planes, discharge electrodes of relatively small surface being interposed. The collecting electrode may be provided with flanges to act as baffles to the gas current.—J. S. G. T.

Refrigerating apparatus. C. Delaygue. E.P. 29,635, 23.12.13.

Condensing apparatus [for refrigerating machines]. J. McOustra. E.P. 144,413, 26.3.19.

Corrosive fluid valves. Chemical Equipment Co., Asses. of H. E. La Bour and G. A. Grassby, jun. E.P. 124,445, 13.3.19. Conv., 13.3.18.

Rotary furnaces, retorts, and the like [; Driving mechanism for —]. E. H. Summers and E. S. Whittard. E.P. 144,815, 21.3.19.

Froth or scum; Method and apparatus for suppressing the formation of — when boiling liquids. E. Wirth-Frey and E. Jenny-Henz. E.P. 121,293, 5.12.18.

SEE U.S.P. 1,316,947 of 1919; J., 1919, 806 A.

Tunnel kilns. G. H. Benjamin. E.P. 143,653, 6.3.19. SEE U.S.P. 1,294,756 of 1919; preceding.

Centrifugal dryers. G. H. Elmore. E.P. 144,979, 6.2.20.

SEE U.S.P. 1,334,023 of 1920; J., 1920, 322A.

Drying apparatus. S. H. Meihuizen. U.S.P. 1,344,241, 22.6.20. Appl., 9.3.18.

SEE E.P. 114,620 of 1918; J., 1918, 357 A.

Solidifying or concentrating materials; Apparatus for —. J. W. Leitch. U.S.P. 1,343,833, 15.6.20. Appl., 14.11.18.

SEE E.P. 117,560 of 1917; J., 1918, 496 A.

Furnace. T. B. Kitson. U.S.P. 1,343,457, 15.6.20. Appl., 5.12.18.

SEE E.P. 122,672 of 1917; J., 1919, 177 A.

II.A.—FUEL; GAS; MINERAL OILS AND WAXES.

Coals; Oxidation of —. M. Godchot. Comptes rend., 1920, 171, 32-34.

THE coal used contained 37.40% of volatile matter, 58.60% of fixed carbon, and 4% of ash. The ash-free material contained 83.59% C, 5.78% H, 10.67% O+N. The coal gave 22.63% of pyridine extract, which contained 83.45% C, 5.84% H, 10.51% O+N. When heated in the air at 100° C. for one month the original coal gained 3.15%, the extract 1.22%, the residue 1.99%, and a mixture of the residue and extract gained 3.20%. These results do not appear to be consistent with bacterial action being the cause of the slow oxidation of coal.—W. G.

Coal analysis; Notes on —. F. S. Sinnatt. Bull. 4, Lancs. and Cheshire Coal Res. Assoc., 1920. 38 pp.

APART from methods for the sampling, and proximate and ultimate analysis of coal, the bulletin contains information on the subject of the inorganic partings which occur in most bituminous coals, and on the fusibility of coal ash (*cf.* Cobb, J., 1904, 11). Carbonates in coal may be determined by a modification of the Pettenkofer method, described previously (Analyst, Apr., 1913). The agglutinating power (Grounds and Sinnatt, J., 1920, 83 r) and the increase in volatile matter due to the addition of inert matter, such as stone-dust (following abstract) should also be determined. A summary is given of eight results obtained by carrying out the standard volatile matter test in crucibles of different materials, showing the figures to vary between 28.4% and 36.1% for one coal and 31.9% and 37.8% for a second coal. A method of calculating the oxygen content of the coal substance is given.—A. G.

Coal; Influence of the addition of inert matter upon the volatile matter evolved when — is heated. F. S. Sinnatt and A. Grounds. Bull. 3, Lancs. and Cheshire Coal Res. Assoc., 1920. 14 pp.

THE effect of the degree of fineness of the coal, the addition of inert matter in varying proportions, and the fineness of the inert matter added upon the percentage of volatile matter evolved when coal is heated has been studied. Contrary to Somermeier (J. Amer. Chem. Soc., 1916, 26, 1002), the authors found that fine coal evolves rather more volatile organic matter than coarse coal. The inert matter added to the coal was gas-carbon, graded to varying degrees of fineness. With a mixture of 1 pt. of coal to 4 pts. of gas-carbon, carbonised under standard conditions, varying the size of the gas-

carbon produced very little change in the percentage of volatile matter evolved. On adding the gas-carbon to the coal in varying proportions, however, and carbonising under the same conditions, the percentage of volatile matter increased with the addition of gas-carbon up to a point at which there were 20 pts. of gas-carbon to 1 pt. of coal. The results are published for eight typical Lancashire coals, the volatile matter evolved having increased from 31.2 to 41.0% (Arley), from 38.0 to 50.0 (Bacon Mine), from 35.3 to 46.4 (Garswood 9ft.), from 33.9 to 43.2 (Hall Hole), from 27.3 to 32.3 (Mountain Mine), from 31.8 to 43.0% (Pemberton 2 ft.), from 31.7 to 45.3 (Ravine Mine), and from 28.0 to 30.0 (Hoo Cannel). The bearing of these results on the subject of stonedusting in mines and on the "sandwich" system of utilising coal and coke for steam-raising is discussed.—A. G.

Fuels; Practical value of —. E. Damour, *Comptes rend.*, 1920, 170, 1578–1581.

IN determining the practical value of a fuel it is necessary to take into account several factors in addition to the calorific value. The relative practical value of two fuels is the inverse of the weights of the two fuels which will just replace one another and give the same effect in a given furnace. A close approximation to the practical value is given by $V = Pf(0 - t)[1 + a(0 - t)] \pm M$, where P is the calorific value of the fuel used, 0 is the temperature of combustion, t the temperature of the material heated, and M represents all such factors as the cost of working the fuel, gasification, pulverisation, crushing, management of the fires, etc.—W. G.

Heavy oil fuel; Industrial use of —. E. Chabanier, *Chim. et Ind.*, 1920, 3, 566–575.

THE requirements as to viscosity, ignition temperature, and the amount distilled between certain temperatures are discussed. The author compares various types of solid fuel and gas furnaces, and describes their conversion into oil furnaces. In long-flame furnaces the consumption of oil is about one-third of the fuel consumption in a gas furnace. The oxidising effects of the flame can be reduced as desired.—W. J. W.

Gasoline; Methods of removing — from natural gas. R. P. Anderson, *J. Ind. Eng. Chem.*, 1920, 12, 547–549.

LEAN natural gas, yielding less than 0.5 gall. of gasoline per million cub. ft., is usually compressed for transport. The absorption method of removing gasoline has been used with gas containing less than 0.1 gall. per million cub. ft. Compression of moderately rich gas (0.5–3 galls. per million cub. ft.) will remove part of the gasoline, and the remainder may be recovered by refrigeration or absorption methods. Rich gas (more than 3 galls. per million cub. ft.) yields a larger proportion of its gasoline on compression, owing to the higher initial partial pressure of gasoline constituents, and but little more will be recovered by either refrigeration or absorption methods.—C. A. M.

Natural gas; Partial condensation of — at liquid air temperatures. J. Satterly, *Trans. Roy. Soc. Canada*, 1913, 13, [iii.], 109–121.

IN the preliminary analysis of natural gas for helium it is passed into a tube cooled with liquid air in which hydrocarbons condense. The condensed product is usually solid with Western Canadian gases and Texas gas, but liquid with most of the Ontario gases. The liquidity is due to dissolved ethane. The pressures during treatment show that some nitrogen probably dissolves in liquid methane or in the condensed solid. When the

liquid air cooling is removed and the condensed product allowed to warm up, instead of the pressure increasing regularly it sometimes rises to a certain point, halts for a time, and then suddenly diminishes for several cm., keeps steady for a time, and then gradually descends. It is thought that as the pressure rises some nitrogen is suddenly forced into solution. The reverse effects are often noticed on putting the tube again in liquid air. In some ways the sudden evolution or solution of nitrogen by liquid methane resembles the occlusion and spitting of oxygen by molten silver, and the effect must be kept in mind in the design of liquefaction plants, as a sudden evolution of gas may upset the balance in the rectifying column.

—J. R. P.

Methane-nitrogen; Composition of the vapour and liquid phases of the system —. H. A. McTaggart and E. Edwards, *Trans. Roy. Soc. Canada*, 1919, 13, [iii.], 57–66.

IN connexion with the extraction of helium from natural gas (cf. preceding abstract), the temperature-composition diagram for the system methane-nitrogen at atmospheric pressure was obtained, and the constants in the formula $\log r' = a + b \log r$, where r = ratio methane/nitrogen in the liquid, and r' the corresponding ratio in the vapour, determined as $a = -0.47$, $b = +0.85$.—J. R. P.

Methane and ethane; Latent heats of vaporisation of —. J. Satterly and J. Patterson, *Trans. Roy. Soc. Canada*, 1919, 13, [iii.], 123–127.

APPROXIMATE values of the latent heats of vaporisation of methane and ethane have been determined: methane 130 cal. per g.; ethane 260 cal. per g. (See J.C.S., Aug.)—J. R. P.

PATENTS.

Motor fuel for aeroplane and other motors. U.S. Industrial Alcohol Co., Assees. of W. T. Schreiber. E.P. 128,916, 16.6.19. Conv., 25.6.18.

THE fuel consists of 35–50% of an alcohol, e.g., ethyl alcohol, 25–35% of an aromatic hydrocarbon, e.g., benzol, and 20–40% of an ether, e.g., ethyl or butyl ether.—A. E. D.

Extracting oils and hydrocarbon material from shale and similar bituminous rocks [in situ]; Process for —. D. T. Day. U.S.P. 1,312,741, 8.6.20. Appl., 17.1.18.

THE volatile hydrocarbon material is volatilised from shale etc. *in situ* by pumping hot gases of combustion, substantially free from oxygen, down a borehole, the combined gases and vapour being led out of the borehole and afterwards separated.

—B. M. V.

Oil-still. W. M. Duncan. U.S.P. 1,342,947, 8.6.20. Appl., 10.9.17.

THE still contains intake and discharge manifolds near the bottom and top respectively. The manifolds are connected through a centrifugal pump within the still, whereby oil may be pumped from the bottom and discharged on to the surface of the oil in the still.—A. E. D.

Motor fuels and light paraffin oils from shale; Method of obtaining — and benzene, toluene, and solvent naphtha from coal. E. W. Thurlow. U.S.P. 1,343,100, 8.6.20. Appl., 28.8.17.

SHALE or coal is mixed with finely divided metal and with a substance which evolves carbon dioxide when heated, and then subjected to destructive distillation.—A. E. D.

Firing furnaces with powdered fuel; Apparatus for —. J. J. C. Brand. E.P. 144,763, 11.3.19.

Coke or the like withdrawing or extracting apparatus as applied to inclined and vertical carbonising and the like retorts and the like; Mechanically operated —. J. Mitchell. E.P. 144,952, 5.11.19.

Sulphonated products of mineral oils and process of producing same. Sulphonic acids of mineral oils and process of producing same. Alkali metal sulphate and process of producing same. W. J. Mellersh-Jackson. From Twitchell Process Co. E.P. 143,681—3, 5.4.19.

SEE U.S.P. 1,301,662, 1,303,779, and 1,301,663; J., 1919, 471 A, 545 A.

IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Adsorbing materials [charcoal and carbonised lignite]; Density of —. S. McLean. Trans. Roy. Soc. Canada, 1919, 13, [iii.], 197—199.

THE density was determined by the volumometer method, using helium as the filling gas, since it is not adsorbed at ordinary temperature. The following mean results were found:—Coconut charcoal (heated to 400° C.), 1.53; lignite carbonised at 350°, 1.33; lignite carbonised at 450°, 1.46; lignite carbonised at 550° C., 1.44.—J. R. P.

Inverted incandescence gas burner; A study of the —. C. Killing. Gas World, 1920, 72, 586—589.

By inserting very fine platinum wires at various points in the flames of an upright and an inverted Bunsen burner respectively, it was found that in each case the highest temperature of the flame occurs just at the tip of the small inner green cone of the flame. The heat produced in the primary combustion is proportional to the proportion of air in the primary mixture. These results have been applied in the design of a burner in which the primary air-gas mixture flows downwards out of the nozzle, and the mantle is brought very close to the small green inner cone of the flame. The lower end of the burner nozzle is provided with wire gauze or a perforated grid of metal, steatite, or magnesia. The mantle is small, hemispherical, and "spiderless," and retains its shape in use. The efficiency of the burner in the vertical direction is 0.69 l. per Hefner. Burners employing a magnesia grid at the bottom of the burner nozzle give 148.3 Hefners vertically, 123.5 Hefners at an angle of 45°, and 82.5 Hefners horizontally for an hourly consumption of 82.2 l. measured at 0° C. and 760 mm.

—J. S. G. T.

III.—TAR AND TAR PRODUCTS.

Paracoumarone resin. King and others. See XIII.

PATENTS.

Phenols and "other aromatic hydrocarbons"; Process of removing — from water or other liquids. R. M. Leggett, Assr. to Ozone Co. of America. U.S.P. 1,341,913, 1.6.20. Appl., 23.12.18.

THE liquid containing phenol is "coagulated," and after settling and removal of the deposit is treated with ozone.—W. H. C.

Sulphonating benzene. Barrett Co., Assees. of C. R. Downs. E.P. 122,169, 28.11.18. Conv., 5.1.18.

SEE U.S.P. 1,279,295—6 of 1918; J., 1918, 763 A.

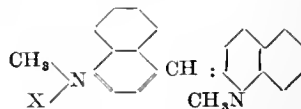
Benzene etc. from coal. U.S.P. 1,343,100. See IIA.

IV.—COLOURING MATTERS AND DYES.

Cyanine dyes. I. Constitution of the isocyanines. W. H. Mills and R. S. Wishart. Chem. Soc. Trans., 1920, 117, 579—587.

THE oxidation of 1,1-dimethylisocyanine acetate with cold aqueous permanganate gave 1-methyl-2-quinolone and cinchonic acid methochloride in accordance with the equation:

$C_{21}H_{19}N_2X + 3O = C_{10}H_7ON + C_{11}H_{10}O_2NX$, and it is concluded that the constitution of the dye is expressed by the formula:



The possibility that the isocyanines are virtually tautomeric compounds is, however, admitted, and a reversal of the normal order of basicity of the nitrogen atoms obtaining in the case of the simpler isocyanines is probably brought about by negative substitution in the quinoline half of the molecule, e.g., in 1'-methyl-2'-phenyl-1-ethylisocyanine iodide.

—G. F. M.

Morinda citrifolia; Constituents of —. J. L. Simonsen. Chem. Soc. Trans., 1920, 117, 561—564.

THE material investigated was obtained by acidification of the cold-water extract of the sparingly soluble barium salt of morindin (cf. J., 1918, 685 A). The yellow precipitate thus obtained was a complex mixture containing much resinous matter, but rubiadin monomethyl ether and alizarin α -methyl ether were isolated from it. The former substance is the methyl ether of 1,3-dihydroxy-4-methyl-anthraquinone, and is identical with the substance obtained by Barrowcliff and Tutin from *M. longiflora*, and erroneously described by them as a 1,3-hydroxymethoxy-2-methylantraquinone. Alizarin α -methyl ether was also obtained from *M. longiflora*.

—G. F. M.

PATENT.

Water-soluble dyes; Production of —. A. M. Hart. E.P. 144,336, 13.12.18.

WATER-SOLUBLE dyes are obtained from animal or vegetable dye-yielding materials, e.g., cochineal, turmeric, madder, etc., by extracting with a solvent, concentrating the solution, and heating this with a water-soluble metallic salt, e.g., alum, copper sulphate, potassium ferrieyanide, etc.

—A. J. H.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cotton; Prolonged action of a moderate heat on bleached — and some other substances. E. Knecht. J. Soc. Dyers and Col., 1920, 36, 195—198.

ALL the materials tested were exposed at 93° C. Bleached cotton yarn and bleached calico, on clock glasses, remained unchanged for the first few days and then turned greyish brown, with formation of oxycellulose. At the end of 336 hrs. the cotton yarn had decreased in strength 33%. When exposed in sealed tubes, yarn decomposed slightly and lost 50% of its strength, and had a remarkable shrivelled appearance. Other cellulose materials, as bleached linen and viscose silk, wood and filter paper, gave similar results. With scoured flannel and spun silk browning occurred after 24 hrs.—A. J. H.

Korean hemp and ramie; Proximate compositions of —. Y. Uyeda. J. Ind. Eng. Chem., 1920, 12, 573—576.

KOREAN hemp and ramie respectively gave the following results:—Loss on drying, 8.83, 10.50; benzene extract, 1.92, 0.86; alcohol extract, 1.20, 0.75; water-soluble substances, 4.50, 3.79; sol. in 1% NaOH, 18.53, 17.27; cellulose, 62.42, 65.88; and lignin, 3.32, 0.66%. Determination of lignin in hemp and ramie affords an indication of the completeness of the retting process. Lignin is partly attacked by alkali digestion. In the case of these fibres the lignin was thus reduced to 1.99 and 0.18% respectively.—C. A. M.

Balloon fabrics; Permeability of — to hydrogen and to helium. R. T. Elworthy and V. F. Murray. Trans. Roy. Soc. Canada, 1919, 13, [iii.], 37—45.

WITH rubbered fabrics the permeability to helium is less than to hydrogen, the ratio of helium to hydrogen being 0.66 by the interferometer method and 0.72 by the catharometer method. With skin-lined fabrics the ratio of helium to hydrogen permeability was found to be almost unity.—J. R. P.

Cellulose; Viscosity of solutions of —. W. H. Gibson, L. Spencer, and R. McCall. Chem. Soc. Trans., 1920, 117, 479—493.

THE viscosity of cuprammonium solutions of purified cotton can be controlled by controlling the conditions of the boiling process with sodium hydroxide. Using 2% caustic soda solution, the viscosity is approximately 50% higher than with 4% solution, but temperature is by far the most important factor in reducing the viscosity. Further, products of different viscosity are obtained from different classes of cotton waste under similar conditions of purification; for example, eastern cottons give products the viscosity of which is about half that of American cotton of similar grade. A close relationship exists between the viscosity of cellulose solutions and solutions of the nitrocellulose derived therefrom. The viscosity determinations were made with a hydrogen capillary viscosimeter and with the falling-sphere viscosimeter (see page 558 A), special precautions being taken in dissolving the cotton and filling the viscosimeter tube to prevent exposure to air or light, which causes progressive changes in the viscosity of the solutions. Variations in copper and ammonia content of the cuprammonium solution prepared by Ost's method (J., 1911, 1247) considerably influenced the viscosity of the cellulose solution. Copper hydroxide prepared by Dawson's method (Chem. Soc. Trans., 1909, 95, 370) showed a fairly definite solubility in ammonia, and an unsaturated solution of this form of the substance, containing 11 g. Cu and 200—210 g. NH_3 per l., was chosen as the standard cuprammonium solution.—G. F. M.

Pyroxylin solutions; Measurement of viscosity of —. E. F. Higgins and E. C. Pitman. J. Ind. Eng. Chem., 1920, 12, 587—591.

EFFLUX viscosimeters with small orifices are suitable for determining the viscosity of pyroxylin solutions of low viscosity, but the steel ball viscosimeter is only suitable for solutions of very high viscosity. The Stormer viscosimeter (in which the viscosity is determined by the speed of a revolving cylinder) is the most generally applicable, since it is unaffected by the density of the solution. (Cf. J.C.S., Aug.).—C. A. M.

PATENTS.

Textile fibrous material; Production of —. M. Hofsté. E.P. 136,804, 5.11.19. Conv., 17.12.18.

FIBROUS material (e.g., ramie) is treated with a concentrated alkaline solution (20—40%), boiled in

a saturated solution of an ammonium salt until the ammonia is completely expelled, washed, and dried. The liberation of gaseous ammonia effects the separation of the fibres.—A. J. H.

Vegetable fibres; Treatment of —. Gillet et Fils, Assees. of C. Schwartz. E.P. 144,563, 12.12.19. Conv., 12.6.19. Addn. to 136,568 (J., 1920, 513 A).

TEXTILE materials coated with a nitrated cellulose solution are washed with dilute solutions of acids, bases, or salts, instead of with pure water, in order to obtain a more rapid and complete precipitation of the nitrocellulose.—A. J. H.

Cellulose esters; Manufacture of dissolved or gelatinised —. G. Bonwitt. E.P. 138,078, 17.1.20. Conv., 6.6.17.

CELLULOSE esters, e.g., acetyl-, nitro-, nitroacetyl-cellulose, are dissolved or gelatinised by furfural or a homologue.—A. J. H.

Cellulose ester [acetate] composition, and method of making the same. Cellulose-nitrate composition. Cellulose-acetate composition. P. C. Seel, Assr. to Eastman Kodak Co. U.S.P. (A) 1,342,601, (B) 1,342,602, and (C) 1,342,603, 8.6.20. Appl., (A) (B) 7.2.18; (C) 26.4.19.

(A) A composition comprising cellulose acetate and a chloro substitution product of naphthalene. (B) A transparent flexible film consists of cellulose nitrate, camphor, and an amount of the chlorine derivatives of the cyclic hydrocarbons $\text{C}_{2n}\text{H}_{n+2}$ and their homologues, greater than half the weight of camphor. (C) A composition comprising cellulose acetate and ethyl propionate.—A. J. H.

Acetylcellulose composition, and method of making same. J. G. Jarvis. U.S.P. 1,313,135, 8.6.20. Appl., 13.3.19.

THE composition consists of 75 pts. of cellulose acetate, 25 pts. of naphthalene, and 60 pts. of acetone.—A. J. H.

Sulphite and like digesters; Methods of heating —. J. K. Ruths, and Aktiebolaget Vapora-kumulator. E.P. 144,084, 29.5.19. (Cf. U.S.P. 1,292,080; J., 1919, 283 A.)

THE period of digestion is shortened without affecting the quality of the product if steam of low pressure, obtained from an accumulator connected with the sulphite-boiler, be used for the steaming operation, and also for starting the digestion which is then completed with steam of a higher pressure.—A. J. H.

Soda-cellulose manufacture; Elimination of malodorous substances from the waste gases from —. C. G. Schwalbe. G.P. 319,594, 31.7.17.

THE gases are brought into contact with finely-divided wood, sawdust, or other vegetable waste product. Mercaptans etc. may be destroyed before this treatment by mixing the gases with an oxidising gas, such as chlorine, ozone, etc., or the wood or the like may be subsequently treated with an oxidising gas or liquid.—C. A. M.

Paper-making machines. Great Northern Paper Co., Assees. of C. E. Pope. E.P. 137,061, 24.12.19. Conv., 31.1.17.

Paper-making machine. A. J. Haug, Assr. to Improved Paper Machinery Co. U.S.P. 1,313,505, 15.6.20. Appl., 5.2.17.

Pulp digestion; Manufacture of caustic soda, more particularly from residues recovered from the alkali used in — and containing sodium carbonate. Dorr Co., Assees. of D. S. McAfee. E.P. 131,272, 31.3.19. Conv., 16.8.18.

See U.S.P. 1,308,184 of 1919; J., 1919, 630 A.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Dyeing machine. H. M. Dudley. U.S.P. 1,342,711, 8.6.20. Appl., 21.9.17.

A RECEPTACLE contains a series of fibre chambers having non-perforated sides and perforated top and bottom plates, the top plates being movable relatively to the bottom plates. The fibre chambers are arranged between two perforated plates spaced from the top and bottom plates respectively, and means are provided for forcing a liquid in either direction through the device.—A. J. H.

Dyeing machine. W. J. Going, Assr. to The Klaunder-Weldon Dyeing Machine Co. U.S.P. 1,343,260, 15.6.20. Appl., 8.5.16.

THE machine consists of a vat containing a removable framework which supports a number of removable yarn sticks and dye liquor deflectors.—A. J. H.

Hosiery dyeing machine. H. M. Dudley. U.S.P. (A) 1,344,120 and (B) 1,344,121, 22.6.20. Appl., 5.6.18.

THE fibre chambers are formed by perforated plates fixed lengthwise between two end plates attached to a perforated, hollow, rotary cylinder so as to form a reel. In (A) means are provided for continuously forcing a liquid through the machine.—A. J. H.

Fibre-treating machine. H. M. Dudley. U.S.P. 1,344,122, 22.6.20. Appl., 3.12.18.

A DYEING machine contains a series of fibre chambers each comprising two perforated plates, between which are a series of hollow cylinders. Means are provided for forcing a liquid through the fibres compressed between the plates.—A. J. H.

Cotton yarn or cloth; Process for treating [improving the lustre and texture of] — S. Kashitani. E.P. 144,083, 28.5.19.

COTTON yarn or fabric is treated for 1–50 min. in sulphuric acid of 52°–60° B. (sp. gr. 1.56–1.71), washed in water under vibration to remove loose fibres, immersed for 10–15 min. in a solution of caustic soda of 8°–15° B. (sp. gr. 1.06–1.12) to improve the lustre, neutralise the free acid, and maintain the colour, then treated with a 3% solution of borax to render it non-combustible and neutral. The fabric may be further softened by treatment with a 5% solution of glycerin, or with calcium chloride, glucose, or soap solution.—A. J. H.

Vegetable fibres; Process for the treatment of — Gillet et Fils., Asses. of C. Schwartz. E.P. 144,204, 12.12.19. Conv., 23.7.19. (Of. E.P. 136,568; J., 1920, 513 A).

CELLULOSE fibres coated with nitrated starch compounds have wool-like properties, are strong and soft, and dye directly with basic dyes. The fabric is treated for several minutes at –5° to 20° C. with a solution of starch in 65% nitric acid, then preferably treated with a solution of any suitable salt to aid the precipitation of the nitrated amylaceous substance, and afterwards washed. Alternatively the fabric may be first dressed with a starch paste, dried, treated with 65% nitric acid, the excess acid expressed, and washed. Print effects may be produced with the nitrated starch.—A. J. H.

Mercerisation [of cotton and artificial silk union fabrics]. S. M. Jones, Assr. to Arnold Print Works. U.S.P. (A) 1,343,138 and (B) 1,343,139, 8.6.20. Appl., 28.11.19.

COTTON union fabrics containing viscose silk are

mercerised without injury to the latter by a solution of caustic soda containing (A) formaldehyde or (B) phenol.—A. J. H.

Skein-mercerising machines; Means for electrically controlling — J. E. Wild and W. W. Caldwell, Assrs. to American Thread Co. U.S.P. 1,343,804, 15.6.20. Appl., 17.10.18.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Arsenic-free sulphuric acid. K. Scheringa. Pharm. Weekblad, 1920, 57, 421–422.

THE acid is heated to fuming with the addition of a small quantity of a sulphite. After dilution with an equal bulk of water, it is electrolysed for at least one day, and may then be used for analytical purposes after concentration.—W. S. M.

Reactions started by a primer. E. Berger. Comptes rend., 1920, 170, 1492–1494.

A NEW priming mixture consisting of 60% of sodium or potassium nitrate and 40% of commercial calcium silicide is suggested for starting such reactions as those of the thermit type. The mixture may even be mixed with gum arabic and applied to paper, such paper after drying being a useful form in which to light the mixture. This primer may be lit with a match and burns with a very high-temperature flame. It has been used successfully to start such reactions as the reduction of boron phosphate and arsenate by aluminium, the reduction of calcium and barium sulphates by red phosphorus, the preparation of silicon and boron fluorides from lead fluoride, and the reduction of sulphates by calcium silicide.—W. G.

Chlorides; Production of — by primed reactions. E. Berger. Comptes rend., 1920, 171, 29–32. (Of. ante.)

ALUMINIUM reacts spontaneously or by preliminary priming on a number of chlorides of metals and metalloids; in some cases magnesium is still more active. In a similar manner a considerable number of metals will act on organic chlorides such as carbon tetrachloride, tetra- and hexachloro-ethane, etc., to give the corresponding metallic chlorides which may be vaporised owing to the heat generated by the reaction when once started. Liquid organic chlorides may be first absorbed in kieselguhr or sawdust. Such mixtures, zinc being the metal used, found application in the war for the production of smoke clouds. Later, owing to the lack of organic chlorides, zinc chloride alone was used, being vaporised by means of a mixture of sodium nitrate and calcium silicide.—W. G.

Double salts; Application of a new physico-chemical method of analysis to the study of — R. Dubrisay. Comptes rend., 1920, 170, 1582–1584.

IF a mixture of solutions of sodium and potassium sulphates is mixed with phenol, the temperature of mixing observed agrees closely with that calculated additively from the temperature of mixing of the individual solutions with phenol. Where a mixture of solutions of potassium and zinc sulphates is mixed with phenol there is a difference between the observed and calculated temperatures of mixing varying with the proportions of the two salts present. It is a maximum when the salts are present in equimolecular proportions and indicates the presence, in solution, of a double salt.—W. G.

Chromium sulphate; Constitution of the lilac grey complex — A. Recoura. Comptes rend., 1920, 170, 1494–1497.

THE lilac grey chromium sulphate previously

described (*cf.* J., 1920, 107 A) is shown to consist when freshly prepared of 1 mol. of the green sulphate in its depolymerised state and 2 mols. of the violet sulphate. On standing the green sulphate rapidly and spontaneously polymerises and then masks the sulphate ions of the violet salt. (*Cf.* J.C.S., Aug.)—W. G.

Mercuric salts; Precipitation of — by hydrogen sulphide. P. Jolibois and P. Bouvier. *Comptes rend.*, 1920, 170, 1497—1498.

THE black precipitate obtained with excess of hydrogen sulphide and a dilute solution of mercuric chloride has the composition HgS , the white precipitate obtained when there is excess of mercuric chloride having the composition $2\text{HgS}, \text{HgCl}_2$. There is no indication of the existence of any other intermediate compound.—W. G.

Fluorite; Colouring matters of red and blue —. C. S. Garnett. *Chem. Soc. Trans.*, 1920, 117, 620—622. (*Cf.* J., 1919, 628 A.)

THE colour of "Blue John" appears to be due to films of organic colouring matter lying parallel to and in the plane of the faces of the otherwise colourless crystal and also at the crystalline junctions, and probably deposited on the surfaces of the crystals at intervals during their growth. A very intensely coloured specimen contained 0.27% of carbon, and on distillation gave off inflammable vapours, and a yellowish-brown oil collected in the condenser. The residual fluorite was colourless. The colour of red fluorite, on the other hand, is not destroyed by heat, but is readily extracted from the powdered material by boiling hydrochloric or nitric acid. The solution contained rare earths, and nothing but insignificant quantities of other elements.—G. F. M.

Tartaric acid compounds of antimony. A. Blanchetière. *Bull. Soc. Chim.*, 1920, 27, 477—490.

A STUDY of the solution of antimony hydroxide in a solution of tartaric acid confirms Jungfleisch's view that the process is one of esterification. The monostibiotartaric acid is best obtained by extraction of complexes, richer in tartaric acid, with acetone. (*Cf.* J.C.S., Aug.)—W. G.

Hydrogen peroxide; Catalytic decomposition of — by colloidal platinum. A. de G. Rocasolano. *Comptes rend.*, 1920, 170, 1502—1504.

USING electrosols of platinum, it is shown that the decomposition of hydrogen peroxide by colloidal platinum is not a unimolecular reaction. During the reaction the catalyst itself apparently undergoes some change. The altered catalyst can decompose further amounts of the peroxide, and the reaction is then unimolecular.—W. G.

Gas adsorption [by charcoal]; Effect of pressure on —. A. Pickles. *Chem. News*, 1920, 121, 1—2.

SIX kinds of charcoal, previously heated, were saturated with dry ammonia, and placed successively in a vessel, immersed in cold water, and connected with a manometer and water pump. In all cases the pressure at first increased and ultimately decreased. The denser charcoals, notably those from palm nut, coconut, and fruit stones, were the most efficient gas adsorbents. Iodine liberated by chlorine water from iodine residues was removed by warming the solution with charcoal dust, but only 17% I could be adsorbed from a solution of iodine in benzene. About 80% of benzoic acid was adsorbed from a solution in benzene. The most efficient decoloriser was birch charcoal dust.

—C. A. M.

Adsorption of gases by carbonised lignite. S. McLean. *Trans. Roy. Soc. Canada*, 1919, 13, [iii.], 187—199.

THE adsorption of air, carbon dioxide, oxygen,

hydrogen, and nitrogen by carbonised lignite was studied. Oxygen is adsorbed much more readily than nitrogen. The presence of water vapour decreases the amount of gas adsorbed. No adsorption is permanent, since there are but few cases where the last weight is greater than the first, and these may be due to experimental errors. The fact that with oxygen in most cases the last weight is less than the first indicates that the oxygen combines with the lignite forming carbon dioxide, which is given off when the lignite is heated.—J. R. P.

Helium and hydrogen in air; Use of the Jamin interferometer for the estimation of small amounts of —. J. C. McLennan and R. T. Elworthy. *Trans. Roy. Soc. Canada*, 1919, 13, [iii.], 19—25.

TWO methods of calibrating the Jamin interferometer for the determination of small amounts of helium and hydrogen in air are outlined, one based on the relation between the difference of path caused by the displacement of air in one tube by the mixture and the resulting shift of bands, and the second based on the change of refractive index of air in one tube caused by lowering the pressure of the air. The accuracy was $\pm 0.5\%$. With longer tubes a greater accuracy could be obtained.—J. R. P.

Helium content of mixtures of gases; Estimation of the — by the use of a catharometer. V. F. Murray. *Trans. Roy. Soc. Canada*, 1919, 13, [iii.], 27—35.

THE Shakespear "hydrogen purity meter," high and low percentage "hydrogen explorers," and a Shakespear "permeameter explorer" have been calibrated for use with helium.—J. R. P.

Helium mixtures; Continuous flow apparatus for the purification of impure —. E. Edwards and R. T. Elworthy. *Trans. Roy. Soc. Canada*, 1919, 13, [iii.], 47—52.

HELIUM containing at least 12% of impurity can readily be purified by passing in a continuous stream over charcoal at the temperature of liquid air or oxygen. The helium is not absorbed. The rate of flow can be increased to at least 10 l. per hr. without decreasing the efficiency of the absorption. The maximum possible rate of flow was not determined. By the use of two or more sets of charcoal tubes in parallel the process may be made continuous, as the charcoal may be revived without disturbing the flow.—J. R. P.

Hydrogen and helium; Combustibility of mixtures of —. J. Satterly and E. F. Burton. *Trans. Roy. Soc. Canada*, 1919, 13, [iii.], 211—215.

THE combustibility was tested by a lighted match and by a white-hot platinum spiral. Under the conditions of experiment the percentage of hydrogen could be raised to 26 before the mixture became inflammable; if the percentage exceeded 28 the mixture would burn.—J. R. P.

Natural gas. Satterly. *See* IIA.

Methane-nitrogen. McTaggart and Edwards. *See* IIA.

Conductometric titrations. Kolthoff. *See* XXIII.

PATENTS.

Carbonic oxide; Process and apparatus for treating gases containing — [in connexion with synthesis of ammonia]. General Chemical Co., Assees. of F. W. de Jahn. E. P. 124,760, 28.2.18. Conv., 3.11.17.

GASES consisting of 1 vol. of nitrogen and 3 vols. of a mixture of carbon monoxide and hydrogen, produced by passing the requisite quantities of

steam and nitrogen over red-hot carbonaceous material, are mixed with regulated quantities of steam, and the mixture is passed through a layer of iron oxide maintained at 550° C., whereby the carbon monoxide is catalytically oxidised to carbon dioxide with the liberation of an equal volume of hydrogen. After absorbing the carbon dioxide, the resulting mixture of nitrogen and hydrogen is used for the production of ammonia. The oxidation of the carbon monoxide is carried out in a specially constructed cast-iron chamber supported on three legs, which are so arranged on supports that they are capable of movement to allow for expansion when the chamber is heated. The gases are preheated before entering the reaction chamber by means of the flame from a producer-gas burner, and the heat of the issuing gases is used to preheat a further quantity of the original mixture.—A. R. P.

Alkali alumino-silicates; Decomposition of —. E. Bergve. E.P. 127,586, 9.5.19. Conv., 27.5.18.

NATURAL alkali alumino-silicates are heated to 800°–1200° C., without melting, and sulphur vapour is passed through the mass to produce "oxy-sulphosilicates" of the alkalis and aluminium. The mass is extracted with water in an autoclave, whereby hydrogen sulphide is evolved and soluble alkali sulpho-aluminate is formed, from which aluminium hydroxide may be recovered.—A. R. P.

Copper sulphate; Obtainment of — from ores containing copper. J. S. Ross. E.P. 143,973, 6.3.19.

COPPER carbonate ores are leached with an excess of 5% sulphuric acid, air, with or without superheated steam, being forced through the charge until the copper is dissolved. The liquor is drained off on to a fresh charge of ore and allowed to remain in contact therewith until it becomes basic, when it is again drawn off and concentrated to 52° Tw. (sp. gr. 1.26) after the addition of copper oxide or other basic substance. Air, with or without superheated steam, is forced through the liquid in minute jets, and the whole allowed to cool. After filtration the clear solution is used for the production of pure copper or copper sulphate crystals. The second charge of ore is used as the first charge in the next cycle of operations.—A. R. P.

Rock salt; Purifying —. L. W. Damman. E.P. 144,022, 16.9.19.

Rock salt is ground to grains of about 1 mm. size, and the finer, less pure material separated by sifting. The coarser material is further purified by a suitable grading operation, e.g., by a jiggling process in saturated brine, whereby the heavier impurities fall to the bottom, whilst the lighter particles of purified salt are collected separately, and, after drying, are suitable for table use.

—A. R. P.

Evaporating plant for the manufacture of salt from brine or other like purposes. M. Allen. E.P. 144,023, 9.4.19.

THE brine circulation in a series of open pans is accelerated by forcing the liquid by means of a steam pump, or the like, into a system of flow and return pipes arranged to pass through the flues or furnaces of each of the pans. The brine is discharged into a settling tank, which it enters and leaves at the top, thus allowing any solid impurities to settle. A by-pass is provided to the tank and to each of the evaporating pans, so as to cut any desired unit out of the circuit when required.

—A. R. P.

Salt; Method of, and apparatus for producing purified —. F. G. Wheeler, Assr. to Bleach Process Co. U.S.P. 1,342,519, 8.6.20. Appl., 16.11.18.

A SALT is separated from a solution containing other salts by passing the solution through an open

atmospheric cooler, whereby crystals of the required salt are deposited on the cool surface. Means are provided for removing the crystals from the cooler, and a centrifugal machine for separating them from the solution.—A. R. P.

Ammonium sulphate; Manufacture of —. S. M. Shadbolt and J. E. Grainger. E.P. 144,030, 16.4.19.

THE crystals of ammonium sulphate and mother liquor in the liquor pit are lifted by means of ammonia vapours mixed with steam to a neutralising chamber fitted with baffle plates over which the liquid flows while ammonia vapours are passed through the chamber, the excess of ammonia being returned to the saturator.—A. R. P.

Purifying [burner] gases and producing briquetting material. T. L. Briggs, Assr. to General Chemical Co. U.S.P. 1,301,372 and 1,301,373, 22.4.19. Appl., 1.6.16 and 15.5.17.

GASES derived from the burning of sulphur ore and containing sulphur dioxide, oxygen, nitrogen, and small quantities of sulphur trioxide and iron oxide dust, are filtered through a layer of the burnt ore, which is pulverised to such a size that it will pass through a sieve of 1 in. mesh, but not through a sieve of 0.25 in. mesh. The gases are passed horizontally through a vertical layer of the ore at a temperature of 900° F. (480° C.). The filtering layer is preferably about 18 in. thick, and the lower layer is periodically removed by vibration through a grate and the upper layer simultaneously replenished with fresh material. The material is contained between a pair of vertical screens, one of which is adjustable to and from the other to vary the thickness of the filtering layer.—W. F. F.

Fixing nitrogen; Process for —. C. T. Thorssell and H. L. R. Lundén, Assrs. to Aktiebolaget Kvaefveindustri. U.S.P. 1,342,834, 8.6.20. Appl., 9.7.19.

IN the production of cyanides by the interaction of nitrogen with a heated mixture of carbonaceous material and alkaline compounds in the presence of finely-divided metal, the latter is prepared by reducing compounds containing no other elements than the metal, hydrogen, oxygen, carbon, and nitrogen at a temperature below 550° C., and is immediately introduced into the reaction zone.

—J. S. G. T.

Ammonium perchlorate; Manufacture of — from sodium perchlorate and ammonium sulphate. G. C. Given, H. G. Quaid, and R. A. Long, Assrs. to Atlas Powder Co. U.S.P. 1,342,956, 8.6.20. Appl., 23.10.18.

TO a nucleus solution of sodium sulphate and ammonium and sodium perchlorates are added sodium perchlorate and ammonium sulphate. Anhydrous sodium sulphate separates and is removed, and ammonium perchlorate is then separated below 22° C.—A. E. D.

Manganese peroxide; Process of forming —. H. C. Chapman, Assr. to National Carbon Co. U.S.P. 1,343,390, 15.6.20. Appl., 29.6.17.

MANGANESE peroxide for use as a battery depolariser is prepared by mixing manganese carbonate with a salt containing chlorine and roasting the mixture.—W. J. W.

Brines; Process for separation of constituents of —. Process for separating compounds from brines. H. W. Morse, Assr. to American Trona Corp. U.S.P. (A) 1,343,400 and (B) 1,343,401, 15.6.20. Appl., 7.11 and 26.11.18.

(A) BRINE is evaporated to such a degree that on further heating the precipitated salts with the

liquor to 90°–100° C., potassium chloride and borax will redissolve, after which these salts are separated from solution by crystallisation. (u) The solution of potassium chloride and borax may be subjected to a rapid cooling in order to separate potassium chloride alone.—W. J. W.

Potassium salts; Process for recovery or purification of —. W. Hirschkind, Assr. to California Alkali Co. U.S.P. 1,313,508, 15.6.20. Appl., 8.4.18.

POTASSIUM salts are recovered from mixtures containing boric acid by extracting the latter with alcohol.—W. J. W.

Aluminium chloride; Process for producing anhydrous —. P. Danckwardt. U.S.P. 1,343,662, 15.6.20. Appl., 23.6.19.

A METAL chloride is electrolysed under such conditions that the nascent chlorine formed at the anode reacts with an aluminium compound, thereby producing aluminium chloride which volatilises and is condensed in suitable chambers.—A. R. P.

Chrome alum; Process for manufacturing —. G. H. Hultman. U.S.P. 1,343,725, 15.6.20. Appl., 11.11.19.

FERROCHROME is dissolved in sulphuric acid, and a soluble potassium salt is added to the solution. —W. J. W.

Air or other gaseous mixtures; Process for separating —. Ges. fur Linde's Eismaschinen A.-G. G.P. 319,992, 22.1.16. Addn. to 301,940.

THE heat exchange in the rectification column in the separation of the argon (E.P. 24,735 of 1914 and 109,789; J., 1915, 871; 1918, 507A) should be at least equal to the heat of evaporation of the total quantity treated in the column.—C. A. M.

Lead salts; Method and apparatus for producing —. W. J. Mellersh-Jackson. From E. A. Sperry. E.P. 144,819, 21.3.19.

SEE U.S.P. 1,308,948 of 1919; J., 1919, 631A.

Titanium compounds; Process of producing —. P. Farup, Assr. to Titan Co. A./S. U.S.P. 1,343,441, 15.6.20. Appl., 27.2.18. Renewed 4.4.19. SEE E.P. 115,020 and 116,266 of 1918; J., 1919, 175A, 296A.

Sodium sulphate; Removal of — from solution. F. A. Freeth and H. E. Cockledge. U.S.P. 1,343,443, 15.6.20. Appl., 30.10.18.

SEE E.P. 131,956 of 1918; J., 1919, 761A.

Pyrites kiln. E.P. 144,142. See X.

Titanium pigments and products. U.S.P. 1,343,446-7 and 1,343,468-9. See XIII.

VIII.—GLASS ; CERAMICS.

Glass furnace refractories; Preservation of — by water-cooling. E. B. Christmas. J. Soc. Glass Tech., 1920, 4, 128—138.

By adapting water-cooling devices to glass-furnaces, the durability of the refractory materials and the efficiency of the workmen may be greatly increased without introducing any radical changes into works practice. The water-cooling devices should be made of thin, welded steel plates (castings are unsuitable), and the effluent water should have a temperature of 180° F. (82° C.) or 5°–10° C. below the temperature at which precipitation of any saline matter in the water can occur.—A. B. S.

Glass; Development of various types of —. C. J. Peddle. J. Soc. Glass Tech., 1920, 4, 3—107.

LIME-SODA glasses of the types $100\text{SiO}_2, 40\text{Na}_2\text{O}, x\text{CaO}$ and $100\text{SiO}_2, 20\text{Na}_2\text{O}, x\text{CaO}$, in which x lies between 5 and 40, can be made at temperatures above 1300° C. They all attack the melting pot in proportion to the soda or lime, or both, present. The density, refractive index, and total dispersion rise on increasing either the lime or soda present, the lime having the greater effect. Lime-soda glasses containing more than 72% SiO_2 tend to devitrify, silica being separated. Glasses containing more than 10% CaO and more than 20% Na_2O devitrify readily, but with less than 15% Na_2O , up to 15% CaO may be present without devitrification. With more than 15% Na_2O the glasses become dim on exposure. All the lime-soda glasses in the first series weather too badly to be used as optical glasses, and all in the second series, except $100\text{SiO}_2, 20\text{Na}_2\text{O}, 20\text{CaO}$, are useless, as they devitrify too readily. Glasses of the latter type are not sufficiently durable for first-class optical glass, but are useful for jars, bottles, etc. In modern glass manufacture the tendency is to use mixtures too high in silica and soda and too low in lime. After continued use of cullet from which some alkali has been volatilised the glass works badly. Instead of the customary addition of soda—which gives a glass lacking durability—it is better to add lime so as to bring the composition of the glass up to $100\text{SiO}_2, 20\text{Na}_2\text{O}, 20\text{CaO}$. Lime-potash glasses corresponding to the formulae $100\text{SiO}_2, 40\text{K}_2\text{O}, x\text{CaO}$ and $100\text{SiO}_2, 20\text{K}_2\text{O}, x\text{CaO}$, where x lies between 5 and 40, are more brilliant, more brittle, and less coloured than the corresponding lime-soda glasses. They had to be founded above 1300° C., and attacked the melting pot. Their total dispersion, refractive index, and density increase irregularly with the lime and potash content, the lime having the greater effect on density, and the potash on the refractive index and total dispersion. Lime-potash glasses containing more than 34% K_2O devitrify readily. The solubility decreases as the lime and silica content rises and increases rapidly as the potash increases. The durability is increased by high lime and silica, and diminished by high potash. Soda-lime glasses devitrify more easily than potash-lime glasses, but a high lime content reduces devitrification. Soda-lime glasses are more soluble than the corresponding potash-lime glasses, and glasses containing equal weights of soda and potash are less soluble than either the corresponding soda-lime or soda-potash glasses. Weight for weight soda is a better flux than potash, and a soda glass is more fusible than the corresponding potash glass. Molecule for molecule, soda is more powerful than potash in inducing devitrification. The lime-potash glasses are not sufficiently durable for optical purposes. Lime-soda-potash glasses corresponding to $100\text{SiO}_2, 20\text{Na}_2\text{O}, 20\text{K}_2\text{O}, x\text{CaO}$ and $100\text{SiO}_2, 10\text{Na}_2\text{O}, 10\text{K}_2\text{O}, x\text{CaO}$ are not of first-class durability for optical instruments, though glasses corresponding to $100\text{SiO}_2, 10\text{Na}_2\text{O}, 10\text{K}_2\text{O}, 20\text{CaO}$ have a durability equivalent to Schott and Genossen's No. 0337 silicate crown glass. The refractive index and density of these glasses increase irregularly with an increase in either lime or alkali, and in these and other properties the glasses are intermediate between the lime-soda and lime-potash glasses. The best glasses in all the foregoing series can be represented by the simple formula $5\text{SiO}_2, 1\text{R}_2\text{O}, 1\text{CaO}$; glasses corresponding to the usual Benrath formula $6\text{SiO}_2, \text{R}'_2\text{O}, \text{R}''\text{O}$ are rather more difficult to melt, more durable, but devitrify more easily.—A. B. S.

Weathering of glass; Mechanics of —. F. R. von Richowsky, J. Amer. Ceram. Soc., 1920, 3, 309—312.

THE mechanism of weathering consists in the ad-

sorption or absorption of water in the following successive stages: true surface adsorption; solution of the adsorbed molecules in the glass; reaction of the dissolved water with the glass, forming a surface film; absorption of water by this film, and further reactions; extraction of soluble salts from the film; and solution of the silica skeleton film.—A. B. S.

Optical glass; Practical test for resistance of — to weathering. F. R. von Biehowsky. J. Amer. Ceram. Soc., 1920, 3, 296—308.

THE sample, in the form of half-inch cubes, is heated with a 5% solution of sodium hydroxide in a steel bomb at 225° C. for 2 hrs., or with hydrochloric acid (1: 1) in a sealed tube of Jena combustion glass at 175° C. for 6 hrs. The appearance of the glass and the loss in weight are noted. The results obtained by the two tests do not always place a series of glasses in the same order of stability.

—A. B. S.

Lead glass for table ware; Comparative effects of soda and potash on —. F. W. Hodkin and W. E. S. Turner. J. Soc. Glass Tech., 1920, 4, 120—125.

ON comparing lead-soda, lead-potash, and lead-soda-potash glasses, it was found that lead-soda glasses are the most fusible and lead-potash glasses the least fusible, but the most viscous, and that a lead-soda-potash glass containing soda and potash in equivalent proportions fuses more readily and is more durable than either a lead-soda or a lead-potash glass. The presence of 2 pts. of borax per 100 pts. of sand increases the durability of these glasses.—A. B. S.

Lime-soda glasses; Optical properties of some —. J. R. Clarke and W. E. S. Turner. J. Soc. Glass Tech., 1920, 4, 111—115.

THE refractive indices and dispersions for the C, D, F, and G lines, the value of v , and the limit of transmission of ultra-violet light in a series of glasses corresponding to $2\text{Na}_2\text{O} \cdot 0.6\text{SiO}_2$ to $\text{Na}_2\text{O} \cdot \text{CaO} \cdot 0.6\text{SiO}_2$ were determined. The refractive index for the D line is an additive property, the relation between refractive index and composition being linear, and all the refractive indices increase regularly as soda is replaced by lime. The total and partial dispersions and values of v were practically constant (about 59) in the glasses examined. The ultra-violet absorption band remains fairly constant at 2950 Å.U., the glasses being very transparent to ultra-violet light.—A. B. S.

Soda-lime glasses; Density of —. S. English and W. E. S. Turner. J. Soc. Glass Tech., 1920, 4, 126—128.

THE substitution of lime for soda increases the density of a glass, this property being additive. Winkelmann and Schott's factors of 2.3 for the density of SiO_2 , 2.6 for Na_2O , and 3.3 for CaO in a glass give low results and require revision.

—A. B. S.

Magnesia-containing glasses; Thermal expansion of —. S. English and W. E. S. Turner. J. Soc. Glass Tech., 1920, 4, 115—120.

WHEN magnesia replaces soda in a soda-magnesia glass, there is a great reduction in the thermal expansion. The thermal expansion appears to be an additive property relative to the composition of a glass. Assuming that the coefficient of linear expansion of fused silica is the same as that of silica in combination in glass, the linear expansion factors of various oxides in glasses are calculated as: SiO_2 0.05, Na_2O 4.32, CaO 1.63, MgO 0.45. Tabulated results for a series of lime glasses and magnesia glasses show that the observed expansions agree better with the expansions calculated with

the aid of the authors' factors than with those calculated by means of Schott's factors.—A. B. S.

Casting slip; Effect of the method of preparation on the viscosity of a —. V. S. Schory. J. Amer. Ceram. Soc., 1920, 3, 286—295.

THE viscosity of a casting slip may be varied by altering the percentage of alkaline reagent and of water and also by varying the mode of preparation of the slip. Slips with high viscosity were obtained by first adding the water to the blunger, then the sodium carbonate and silicate together, and then the clay. Slips of low viscosity were obtained by adding the sodium carbonate before the clay, and the sodium silicate last. When filter-press cakes are ground with water and alkaline reagents, slips of low viscosity are produced by the use of wet cakes, or if the sodium carbonate is added prior to the sodium silicate. Grinding produces slips of a higher viscosity than blunging. The firmest products were obtained by blunging, using the lowest practicable proportion of alkaline reagents.

—A. B. S.

Terra cotta; Satisfactory method of using barium hydroxide in —. M. E. Gates. J. Amer. Ceram. Soc., 1920, 3, 313—315.

A SOLUTION of barium hydroxide is preferable to barium carbonate as an anti-scum reagent on terra cotta bodies. The solution is supplied from a tank provided with a mechanical stirrer, through a siphon, the end of the shorter arm of which is attached to a float. The rate of delivery is varied by altering the length of the longer leg of the siphon.—A. B. S.

Titanium enamels. R. D. Landrum and L. J. Frost. J. Amer. Ceram. Soc., 1920, 3, 316—321.

TITANIUM oxide (rutile) is an opacifying agent, but an excess prevents the enamel remaining properly in suspension. Boric oxide appears to be a desirable constituent of titanium enamels. Titanium oxide is harmful in a ground coat, destroying the cobalt blue, and lowering the melting point and the adhesion of the enamel. Three good titanium enamels had the following formulae: (0.742 Na_2O , 0.078 K_2O , 0.180 CaO) 0.3 Al_2O_3 , (2.444 SiO_2 , 0.371 TiO_2 , 0.768 F_2); (0.672 Na_2O , 0.142 K_2O , 0.186 CaO) 0.31 Al_2O_3 , (2.521 SiO_2 , 0.398 TiO_2 , 0.794 F_2); (0.536 Na_2O , 0.146 K_2O , 0.318 CaO) 0.332 Al_2O_3 , (2.710 SiO_2 , 0.152 B_2O_3 , 0.270 TiO_2 , 1.067 F_2).—A. B. S.

PATENTS.

Printing surfaces. D. Motta, E. Rugiu, and A. Villain. E.P. 143,943, 2.1.19.

PLATES for printing are made of sheets of paper covered with a composition consisting of equal parts of calcium carbonate and magnesium silicate, with an organic agglutinant such as gum. The surface may be provided with a network or grain in relief to facilitate engraving and may afterwards be rendered insoluble by means of formalin.—A. B. S.

Porous refractory materials; Manufacture of —. L. Denis. E.P. 144,359, 5.3.19.

A POROUS refractory material suitable for surface combustion is obtained by calcining a mixture of 30 pts. of "rich refractory earth" containing 70—80% of sand with 30 pts. of magnesia, 25 pts. of "poor light earth" containing more than 80% of sand, and 5 pts. of sawdust. The mixture is heated to at least the temperature at which it will be used.

—A. B. S.

Kiln. C. H. Zwerman. U.S.P. 1,308,973, 8.7.19. Appl., 27.3.19.

Two tunnel kilns are arranged side by side with a common dividing wall, and each comprises a "water-smoking" zone, preheating zone, firing

zone, and cooling zone. The cooling and firing zones of one kiln are opposite the "water-smoking" and preheating zones of the other respectively. A number of burners supplied with producer gas deliver hot gas into the top of the firing zone, and the gases are discharged at the bottom into flues from which they pass into flues around the preheating zone of the kiln. The air in the "water-smoking" zone of one kiln is heated by passing it through flues adjacent to the cooling zone of the other kiln. Overheating of the materials treated and the roof of the tunnel is prevented by providing passages in the roof through which air is passed to preheat it on its way to the burners. This air enters horizontal flues below the kilns at their mid-points and passes in both directions to vertical flues at both ends of the kiln, through which it passes upwards to the flues in the roof. —W. F. F.

Glass; Apparatus for feeding or delivering molten material, such as —, in separate lumps or charges. W. L. Pratt. From C. H. Rankin, C. H. Blumenauer, and C. J. P. Pratt. E.P. 144,552, 6.11.19.

IX.—BUILDING MATERIALS.

Cement; Effect of storage on —. D. A. Abrams. Structural Materials Research Lab., Lewis Institute, Chicago. Bull. 6, June, 1920. 28 pp.

PORTLAND cement was stored for periods up to two years under varying conditions. Compression tests of concrete and mortar showed that the cement deteriorated on storage in cloth sacks, in paper bags, and in sacks covered with Portland cement or hydrated lime. The greater part of the deterioration occurred during the first three months, after which period the cement showed 80% of its original strength, after 6 months 71%, after 1 year 61%, and after 2 years 40%. The 7-day tests showed a greater deterioration than the 28-day and later tests. The strength of concrete is not permanently reduced to that shown by the 7-day and 28-day tests. The storage period is of more importance than the conditions of storage provided direct access of moisture is prevented. Storage of cement prolongs the time of initial and final setting; it affects the normal consistency only slightly. The deterioration appears to be due to the absorption of atmospheric moisture. Cement in temporary storage should be kept not less than 1 ft. above the ground and 1 ft. away from exterior walls. A bibliography on the storage of cement is included. —A. B. S.

PATENTS.

Fireproofing wood and other materials; Protecting and —. W. P. Thompson. From Arentox Co. E.P. 144,342, 5.2.19.

SEE U.S.P. 1,318,523-4 of 1919; J., 1919, 948 A.

Hydraulic ingredient and process of making same. F. M. Meyer, Assr. to The Chemical Foundation, Inc. U.S.P. 1,343,929, 22.6.20. Appl., 3.1.14. Renewed 22.10.19.

SEE F.P. 466,859 of 1913; J., 1914, 964.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Cast iron; Growth of grey —. M. Okôchi and N. Satô. J. Coll. Eng., Tokyo Imp. Univ., 1920, 10, 53-71.

THERE are two cases of growth in grey cast iron, one occurring at temperatures higher than that of transformation, the other during transformation.

The first case is explained as permanent expansion due to the pressure of occluded gases. The flakes of graphite in the iron absorb a large amount of gas, and the spaces round them are also filled with gas. When the iron is heated to a high temperature the pressure of the occluded gases is greatly increased, and if the high temperature is maintained plastic deformation takes place with corresponding increase in volume. A state of equilibrium is set up between the pressure of the gases and the resistance of the material. Raising the temperature disturbs this equilibrium and causes further deformation, while cooling reduces the gas pressure and causes penetration of gases along the flakes of graphite from the surface of the iron. The second case is also considered to be due to the pressure of occluded gases. The transformation from alpha to gamma iron is accompanied by contraction, which the pressure of the gases resists, so that the time for transformation must be extended. This requires repeated heatings, whereby the quantity of occluded gas is increased and the pressure of the gas gradually overcomes the contraction and produces increase in volume. Prevention of growth may be brought about by decarbonising the surface in order to prevent penetration of gases along the flakes of graphite. —J. W. D.

Carbide carbon in steel; Estimation of —. K. Inouye. J. Coll. Eng., Tokyo Imp. Univ., 1920, 10, 145-151.

Two methods of estimating carbide carbon in steel were investigated, viz., those of Ledebur and of Arnold and Read, and the amount of carbon in this form in steels having various microstructures was determined. The amount of carbide carbon increases with the rate of cooling, and the slower a steel is cooled below the A_1 the greater is the amount. This is indicated in the structure, steels having a sorbitic, pearlitic, and spheroidic structure respectively containing 0.51, 0.71, and 0.80% of carbide carbon. In carefully annealed steels only about 90-92% of the total carbon is recovered in the form of carbide, indicating that a certain amount of hardening carbon remains in solid solution. —J. W. D.

[Steel] guns; Erosion of —. M. Okôchi. J. Coll. Eng., Tokyo Imp. Univ., 1919, 9, 239-265.

THE causes of the erosion of guns are discussed, and additional reasons are given for the lower resistance to erosion of high-carbon steel compared with low-carbon steel and of nickel or nickel-molybdenum steel compared with ordinary gun steel (cf. J., 1919, 143 A). —J. W. D.

Nickel steels; Thermal change of the elastic properties of —. P. Chevenard. Comptes rend., 1920, 170, 1499-1502.

IN reversible nickel steels the coefficient of variation of the modulus of torsion is characterised by a rapid increase to a maximum, which corresponds to the minimum of dilatability, and then a gradual diminution. For irreversible alloys the results do not indicate any anomaly. Tempering and hammer hardening diminish the value of the modulus of torsion, which increases, however, with the temperature of annealing, there being a maximum of rapidity between 450° and 550° C. The thermoelastic coefficient is affected by the treatment which the alloy undergoes, but, contrary to what happens in the case of the dilatability, hammer hardening diminishes the amplitude of the anomaly, whilst tempering increases it. —W. G.

Iron; Corrosion of —. R. Kattwinkel. Z. angew. Chem., 1920, 33, 156.

AN incrustation found in a steam pipe was strongly magnetic and contained 99.1% Fe_3O_4 . —W. P. S.

Phosphorus; Determination of — in tin bronzes. L. Bertiaux. *Ann. Chim. Analyt.*, 1920, 2, 167—168.

FIVE g. of the alloy is dissolved in a mixture of nitric acid (36° B., sp. gr. 1.332), 15 c.c., hydrochloric acid (21.5° B., sp. gr. 1.175), 20 c.c., and water, 35 c.c., the solution boiled to expel nitrous vapours, treated with 25% ammonium chloride solution, 35 c.c., and 10% ammonium acetate solution, 20 c.c., again boiled, and the phosphoric acid precipitated by the addition of 10% molybdate solution. The precipitate is collected on a filter, washed with a hot solution containing 5% each of hydrochloric acid (21.5° B.) and ammonium chloride, and then with hot water until free from acidity. The precipitate may be weighed directly or its quantity determined by titration with sodium hydroxide solution or permanganate solution (after reduction with zinc), or it may be converted into ammonium magnesium phosphate and weighed as magnesium pyrophosphate.—W. P. S.

Cadmium and zinc; Experimental determination of the vapour pressure curves of molten — and the calculation of the chemical constants of cadmium, zinc, and monatomic bromine. H. Braune. *Z. anorg. Chem.*, 1920, 111, 109—147.

THE vapour pressure of molten cadmium up to 2 atm. is represented by $\log p = -5763/T - 1.28 \log T + 12.282$, whilst that of zinc is given by $\log p = -6655/T - 1.147 \log T + 12.046$. Both zinc and cadmium have a constant atomic heat over the range of temperature from the melting point to the boiling point. The values are 7.50 (cadmium), 7.24 (zinc). (Cf. J.C.S., Aug.)—J. F. S.

PATENTS.

Steel, particularly adapted for the manufacture of ball and roller bearings. Comp. d'Applications Mécaniques. E.P. 126,289, 23.4.19. Conv., 2.5.18.

THE steel contains C 0.85—1.5, Cr 0.5—2, and V 0.1 to 0.5%, a suitable composition being C 0.950, Mn 0.300, Si 0.150, Cr 1.200, V 0.250%, with sulphur and phosphorus as low as possible. To increase the tenacity and flexibility of the steel, and to facilitate certain forging operations, drawing, and annealing, one or more other metals, such as Ni, Co, Mo, Ta, or the like, may be added in a proportion not exceeding 0.25%.—T. St.

Steels; Alloy —. The British Thomson-Houston Co. From General Electric Co. E.P. 144,185, 4.11.19.

AN alloy containing about 16% B is produced by reducing a mixture of iron oxide and boric acid by aluminium, and this alloy is then added to molten steel to yield a product containing B 0.75—5%. By thus incorporating a relatively small amount of boron with chromium, tungsten, vanadium, etc., comparatively easily fusible special steels are produced which can be cast readily even into intricate forms substantially free from blow holes and other defects, and which still preserve the properties given by the alloying element exclusive of the boron.—T. St.

Iron ores; Treatment of —. C. R. Hayward, F. O. Stillman, and H. M. Schleicher, Assrs. to Moa Iron and Development Corp. U.S.P. 1,341,901, 1.6.20. Appl., 24.7.18.

IRON ore containing aluminium and one or more of the metals nickel, manganese, and chromium is subjected to a sulphating roast whereby a small proportion of the iron and a large proportion of the other metals are rendered soluble. On leaching, a richer iron ore residue is obtained and a liquor which on evaporation yields sulphates of iron and

the other metals. These salts are roasted, and from the resulting oxides alumina is recovered.

—J. W. D.

Iron and steel plates; Process for finishing —. C. Williams, Assr. to N. Nelson. U.S.P. 1,342,484, 8.6.20. Appl., 9.6.19.

THE plates, after pickling, are successively washed in a soapy solution, rinsed, annealed in the presence of sulphurous fumes, and subjected to the action of dry air. Finally, to give them a high polish, the plates are cold-rolled.—T. St.

Iron; Puddling —. T. F. Payne. U.S.P. 1,342,694, 8.6.20. Appl., 30.4.19.

THE metal is agitated in the furnace on horizontal longitudinal parallel lines whereby a combing or stratifying action is produced.—T. St.

Iron or steel articles; Process of colouring —. H. F. Jermain, Assr. to Winchester Repeating Arms Co. U.S.P. 1,342,910, 8.6.20. Appl., 7.1.20.

IRON or steel articles are made the anode and cathode alternately in an electrolytic bath consisting of a solution of an oxidising agent in an alkaline liquid, the opposing electrode being incorrodible. The articles are afterwards washed with hot water, dried, and rubbed with oil.—T. St.

Steel alloy. C. M. Johnson. U.S.P. 1,342,911, 8.6.20. Appl., 7.8.19.

THE alloy steel consists of iron with C 0.30—0.60, Mn not more than 0.80, Si 1.00—2.50, Cr 0.30—0.80, V 0.10—0.30, and Ni 1.50—3.50%.—T. St.

Ferrochrome; Production of low-carbon —. H. C. Sicard, Assr. to United States Ferro-Alloys Corp. U.S.P. 1,341,939, 1.6.20. Appl., 26.9.19.

A HIGH-CARBON ferro-chromium alloy is decarbonised by means of titanic oxide.—J. W. D.

Electric smelting furnace. I. Hole. E.P. 125,363, 6.3.19. Conv., 11.4.18.

CHARGING openings are arranged in the roof of the furnace on both sides of a central longitudinal row of electrodes, so that the sloping sides of the charge form a longitudinal groove at the bottom of which is the slag bath in which the electrodes dip without passing through the charge. A discharge outlet is provided so that the slag in the furnace is kept at a constant level.—A. R. P.

Furnace; Electro-metallurgical —. F. Louvrier. U.S.P. 1,342,636, 8.6.20. Appl., 5.4.19.

AN annular chamber surrounding the feeding column of the furnace leads into the reduction chamber, and means are provided for feeding coal dust on to shelves in this annular chamber.

—J. S. G. T.

Kilns for pyrites. J. L. Fairrie. E.P. 144,142, 14.8.19.

A NUMBER of kilns with fire-grates and working doors are arranged in a circle round a common flue into which the gases are drawn. The burnt ore falls on to sloping floors below the fire-grates and passes through swing doors to a common discharge pit at the base of the central flue, whence it is removed either continuously or intermittently by means of a conveyor, preferably of the drag har type. The conveyor can be withdrawn bodily for repairs. Additional air may be admitted through a port between the pit and the flue, which can be closed by an adjustable tile.—B. M. V.

Furnaces; Port construction for metallurgical —. G. F. Downs and H. F. Miller, jun. U.S.P. (A) 1,343,314 and (B) 1,343,315, 15.6.20. Appl., 30.3.18 and 15.5.19.

(A) Gas and air uptakes communicate with the in-

terior of the furnace, the air uptake by means of an air port, and the gas uptake by means of a water-cooled tubular metal flue opening into the air port. Adjustable means, operated from outside the furnace at a point above the air port, are provided for supporting the flue, so that its orifice is entirely surrounded by the air port. (u) The gas flue connecting the gas uptake with the interior of the air port is arranged to discharge across and at an angle to the upward path of the air coming through the air uptake.—T. St.

Electrolytic recovery of metals from their solutions. H. Wade. From The Central Mining and Investment Corp. E.P. 143,620, 21.2.19.

Is the electrolytic recovery of metals by passing the pregnant solution through a permeable cathode of fine porosity, oxygen is removed from the solution, e.g., by a vacuum pump or by a reducing agent, before passing it into the electrolytic cell.—J. W. D.

Metal alloy. C. B. Backer. U.S.P. 1,311,771, 1.6.20. Appl., 30.12.18.

As alloy consisting of Mg 85—98%, Al 15—14.0, and Zn 0.75—6.0%.—J. W. D.

Copper; Recovering — from its ores. C. J. A. Dalziel. U.S.P. 1,311,786, 1.6.20. Appl., 24.9.18.

Suitable proportions of finely crushed ore and an acid solution are converted into a pulp by aeration-agitation, and the pulp is caused to flow through a series of electrolytic cells, sedimentation in the cells being prevented by a blast of air introduced at the lower part of each cell. The barren pulp is discharged from the last cell.—J. W. D.

Copper; Process of extracting —. A. A. Ossa. U.S.P. 1,313,153, 8.6.20. Appl., 20.12.18.

Oxidised copper ores are leached with a solution containing sulphuric acid and the copper precipitated as iodide by hydriodic acid in presence of sulphur dioxide. The sulphuric acid formed in the latter reaction is used for the extraction of fresh ore, and hydriodic acid is regenerated from the iodide precipitate for addition to fresh copper sulphate solutions.—T. St.

Metallurgical process. [Treatment of copper ores.] W. E. Greenawalt. U.S.P. 1,344,127, 22.6.20. Appl., 28.11.17.

Mixed sulphide and oxide ores of copper are treated with a solvent for the oxides, a flotation agent is added, and the pulp electrolysed, using a current density high enough to give a loose deposit of copper which with the copper sulphide is floated by means of the gases liberated.—A. J. H.

Metallurgical process. W. E. Greenawalt. U.S.P. 1,342,804, 8.6.20. Appl., 21.12.16. Renewed 2.9.19.

As electrolyte containing ferric iron is agitated with a (copper) sulphide reducing agent practically free from iron. A portion of the mixture is continuously withdrawn, the sulphide separated and returned to the agitator, and the reduced electrolyte returned to the electrolytic bath.—J. S. G. T.

Chilian mill; Multiple-process —. C. C. Lane. U.S.P. 1,342,917, 8.6.20. Appl., 12.12.16.

In a Chilian mill the framework that drives the grinding rollers is provided with a heavy and evenly balanced monolith and above that a tank, which may be used for cyanide treatment of the ore pulp, the weight of both being supported by the grinding rollers and assisting grinding.—B. M. V.

[Crucible] furnace. A. C. Osborn, jun. U.S.P. 1,343,470, 15.6.20. Appl., 26.1.20.

A CRUCIBLE furnace comprises an upper section hinged to a lower section carrying a removable sup-

porting member for the crucible, and means for moving one section relatively to the other, gas jets forming a part of the upper section and connected to and fed by a supply pipe and a main supply pipe, and means for automatically providing an air-tight connexion between the two pipes when the upper section is moved to its operative position.—A. de W.

Ores; Treating —. G. L. Collord. U.S.P. (a) 1,343,249 and (n) 1,344,250, 15.6.20. Appl., 9.5.19.

(a) THE moist ore is treated with a liquid, less volatile than water, which will spread throughout the mass and form a thin film round the particles. The ore is then partially dried. (n) The moist ore is partially dried and then mixed with an oleaginous material to agglomerate the fine particles.—A. R. P.

Ores or other materials; Process and apparatus for concentrating —. S. H. Dolbear, Assr. to Minerals Separation North American Corp. U.S.P. 1,343,313, 15.6.20. Appl., 14.11.18.

A COLUMN of liquid containing a mixture of floatable and unfloatable materials in a suitably divided condition is treated with a gas in such a manner that gas in the form of bubbles rises in the liquid and becomes attached to the floatable material. These bubbles are then diverted into a gaseous medium at a point where the pressure in the column is greater than in the gaseous medium.—T. St.

Zinc, copper, cadmium; Process of treating residues containing —. J. O. Elton and R. B. Caples, Assrs. to Anaconda Copper Mining Co. U.S.P. 1,343,619, 15.6.20. Appl., 5.1.20.

Zinc sulphate solutions containing copper and cadmium are treated with zinc to precipitate the latter two metals. The precipitate is treated with just sufficient sulphuric acid to dissolve the zinc and cadmium, and the filtered solution is again treated with zinc to precipitate the cadmium.—A. R. P.

Lead and like ores and products; Smelting —. T. W. Cavers, Assr. to Garred-Cavers Corp. U.S.P. 1,343,656, 15.6.20. Appl., 9.8.18.

Oxidised lead ores or the like are mixed with a carbonaceous reducing agent and smelted in a blast furnace by means of powdered coal fed into the blast.—A. R. P.

Metal-plating; Process and apparatus for —. R. J. Piersol. U.S.P. 1,343,842, 15.6.20. Appl., 3.1.20.

THE metal to be plated is treated with the plating metal *in vacuo* at a temperature between the melting points of the two metals.—T. St.

Furnace; Galvanising —. C. A. Porath. U.S.P. 1,343,843, 15.6.20. Appl., 7.7.19.

THE galvanising material is contained in a tank above a layer of a substance of higher specific gravity, and means are provided for heating the bottom of the tank.—T. St.

Melting furnaces; Lids for —. A.-G. Brown, Boveri & Co. E.P. 132,785, 2.9.19. Conv., 14.9.18. Addn. to 131,887.

Furnace for hardening high-speed steel and the like. S. H. Gush. E.P. 144,486, 23.6.19.

Electric arc welding operations; Metallic electrodes for use in —. C. J. Holslag. E.P. 144,333, 11.12.18.

Steel alloy. Stahlwerke R. Lindenburg A.-G. E.P. 144,326, 16.8.16. Addn. to 103,810.

SEE G.P. 312,311 of 1916; J., 1920, 160 A.

Manganese-steel sheets; Manufacture of —. R. A. Hadfield. U.S.P. 1,344,392, 22.6.20. Appl., 17.8.17. SEE E.P. 133,131 of 1918; J., 1919, 866 A.

Alloy. C. G. Fink, Assr. to General Electric Co. U.S.P. 1,342,993, 8.6.20. Appl., 22.11.15.

SEE E.P. 13,207 of 1915; J., 1916, 695.

Solder for aluminium. F. F. Boisselier. U.S.P. 1,344,165, 22.6.20. Appl., 14.2.19.

SEE E.P. 124,715 of 1919; J., 1920, 338 A.

Copper ores. E.P. 143,973. See VII.

Electrodes. E.P. 143,619. See XI.

XI.—ELECTRO-CHEMISTRY.

Albumins and antitoxins. Ruppel. See XX.

PATENTS.

Electrodes for electrolytic recovery of metals from solution. H. Wade. From The Central Mining and Investment Corp. E.P. 143,619, 24.2.19.

A PERMEABLE electrode consists of a sheet formed of felted organic fibres, a fabric reinforcement, and comminuted conducting material (graphite) incorporated with the fibre.—J. W. D.

Electrical treatment of gases; Apparatus for —. H. W. Squires, Assr. to Research Corp. U.S.P. 1,342,651, 8.6.20. Appl., 22.6.18.

THE gas to be treated is passed through a passage-way, annular in cross-section, between a pair of sloping electrodes.—J. S. G. T.

Electrical furnaces; Heating of material or the performance of chemical processes in —, together with apparatus therefor. T. A. F. Holmgren and C. T. Thorssell, Assrs. to Aktiebolaget Kvafveindustri. U.S.P. 1,342,809, 8.6.20. Appl., 24.7.19.

IN electric resistance furnaces employing alternating current supplied to two or more heating circuits, a phase difference approximately equal to 180° or 360° divided by the number of current circuits, is introduced into the currents supplied to the respective circuits.—J. S. G. T.

Resistance element. T. W. Case. U.S.P. 1,342,842, 8.6.20. Appl., 15.3.20.

A RESISTANCE element is composed of a compound of thallium and bromine.—J. S. G. T.

Battery-plates; Active material for —. W. Gardiner and W. H. Duggan. U.S.P. 1,342,953, 8.6.20. Appl., 23.8.19.

A MIXTURE of lead oxide, kieselguhr, powdered pumice, and ammonium sulphite is incorporated with an acid solution.—A. E. D.

Battery materials [manganese oxide]; Purification of —. O. E. Ruhoff, Assr. to French Battery and Carbon Co. U.S.P. 1,343,585, 15.6.20. Appl., 1.3.17.

ORE containing manganese oxide is treated with a dilute solution of sulphuric acid, then mixed with other components of the battery material, and the resulting liquid removed.—J. S. G. T.

Electric furnace; Control of power absorbed in an —. D. F. Campbell, W. S. Gifford, H. S. Waite, and G. E. Taylor. E.P. 144,438, 25.4.19.

Electric arc furnaces [; System of control for three-phase —]. British Thomson-Houston Co., Ltd., and H. C. Hastings. E.P. 144,503, 17.7.19.

Electrolytic apparatus. E. O. Benjamin, Assr. to International Oxygen Co. U.S.P. 1,344,220, 22.6.20. Appl., 1.8.17.

SEE E.P. 137,553 of 1918; J., 1920, 271 A.

Separating emulsions. U.S.P. 1,296,988. See I.

Separating suspended particles. U.S.P. 1,343,285 and 1,343,482. See I.

Manganese peroxide. U.S.P. 1,343,390. See VI.

Steffens' waste liquors. U.S.P. 1,342,737. See XVII.

XII.—FATS; OILS; WAXES.

Catalysis. XIII. Contact potentials and dielectric capacities of metals in relation to the occlusion of hydrogen and hydrogenation. W. C. McC. Lewis. Chem. Soc. Trans., 1920, 117, 623—638.

FROM a consideration of the solubility of hydrogen in certain metals it is inferred that metals in general possess finite dielectric capacities, and values for a number of metals have been obtained from the critical increments of dissociation of hydrogen dissolved therein. The insolubility of hydrogen in certain metals is regarded as being due to the inability of these metals to dissociate the gas, and such metals may be expected to function as negative catalysts when deposited on the surface of active metals in hydrogenation. The dissociating power of a metal for hydrogen depends on both the dielectric capacity and the existence in the metal of radiation of a sufficiently short wave length. As this is related to the magnitude of the atomic latent heat of fusion of the metal, a measure of the dissociating power is given by the product of the dielectric capacity and the latent heat of fusion. The mechanism of hydrogenation of olein by nickel or other metals is regarded as consisting primarily of the dissociation and solution or adsorption of hydrogen by the particles of metal with which the olein molecules already activated by virtue of their temperature collide. A simple collision formula reproduces in so far as order of magnitude is concerned the observed unimolecular velocity constants obtained by Thomas (J., 1920, 10 τ). It is concluded that in the hydrogenation of olein and of similar substances adsorption of the unsaturated compound on the metal does not take place, the adsorption being restricted to the metal-hydrogen compounds.—G. F. M.

Cod-liver oil; The sulphuric acid test for —. P. J. de Kadt. Pharm. Weekblad, 1920, 57, 756—758.

COD-LIVER oil which has been bleached by means of bleaching agents such as fuller's earth, animal charcoal, etc. fails to give a violet coloration with sulphuric acid, the impurities in the oil which give this reaction having been absorbed by the bleaching agent. When the sulphuric acid test is made on a porcelain slab, the best results are obtained with acid of sp. gr. 1.72. It is, however, preferable to employ a 5% solution of the oil in carbon bisulphide and then to test it with sulphuric acid of sp. gr. 1.84.—W. J. W.

Soya bean oil. W. H. Low. J. Ind. Eng. Chem., 1920, 12, 572—573.

A SAMPLE of filtered soya bean oil had iodine value (Wijs) 138.45, and contained 94.96% fatty acids, 1.16% unsaponifiable matter, and 10.37% glycerol (calculated from the saponification value, after making allowance for free fatty acids).—C. A. M.

Soap solutions and gels; Direct experimental determination of the concentration of potassium and sodium ions in —. C. S. Salmon. Chem. Soc. Trans., 1920, 117, 530—542.

THE theory of colloidal electrolytes (J., 1918, 249 τ) is supported by the results of E.M.F. determinations, both from direction and magnitude of the diffusion potential between soap and potassium chloride and by comparison with known concentra-

tions of potassium and sodium chlorides. The concentrations of sodium and potassium ions already deduced from a comparison of conductivity data and osmotic activity are confirmed by direct measurements for a selection of typical cases at 18° and 90° C., the solutions studied being potassium laurate, potassium oleate, and sodium oleate. Gelation of sodium oleate does not influence the results obtained.—G. F. M.

Emulsions. 1. New method of determining the inversion of phases. S. S. Bhatnagar. Chem. Soc. Trans., 1920, 117, 512—552.

A METHOD for determining the inversion of phases and the identification of the two types of emulsion is based on the greater electrical conductivity of the oil-in-water type and the sudden rapid rise in the resistance of the emulsion at the point of inversion into the water-in-oil type, a sensitive milliammeter being used to obtain the indications. Both types of emulsion are formed by sodium or potassium hydroxide with olive oil and with paraffin oil, and the inversion points were determined in each case with different concentrations of alkali. At concentrations below $N/500$ the divalent alkalis gave results practically identical with the univalent alkalis, but at greater concentrations the electrolytic effect predominates over the volume-ratio effect and the formation of the water-in-oil type is favoured. Homogeneous emulsions of particles of uniform size were prepared. Using weak alkalis the volume ratio was constant at 7.4:26, and the equation for closest packing appears to hold in them. A preliminary study was made of the effect of different fatty acids and of a few electrolytes on the emulsion equilibrium.—G. F. M.

Oil-cake. Sabalitsebka. See XIXa.

PATENTS.

Oils or fats; Process for neutralising —. A. G. Bloxam. From M. Arbini. E.P. 144,176, 17.10.19.

OILS or fats are neutralised with dry caustic alkali or calcined alkali carbonate, or with alkali peroxide or percarbonate, with the exclusion of water, the water formed in the reaction being removed, e.g., by a vacuum pump. The neutralised oil is then separated from the dry soap (which is ready for use) by means of an anhydrous solvent (methylal or acetone).—C. A. M.

Hydrogenising oils and fats; Apparatus for —. Blair, Campbell and McLean, Ltd., D. A. Blair, and J. L. Ferguson. E.P. (a) 144,478, 23.8.19. (b) 144,479, 19.6.19.

(A) THE mixture of oil or fat and catalyst is placed in a circular steel high-pressure vessel fitted with a vacuum pipe and means for introducing hydrogen, heated externally by a steam, gas, or flue jacket. A pump raises the mixture from the bottom to the top of the vessel, wherein it falls on to the uppermost of a number of horizontal, preferably annular, dish-shaped plates or partitions, each having a central opening through which the liquid falls into the interior of one of a number of buckets or reservoirs fitted to and adapted to be rapidly rotated in unison on a central shaft. The walls of the buckets are provided with brushes having short flaring bristles fitted through and connected to the insides of the buckets, whereby the centrifugal force of rotation distributes the liquid against the brushes and then forces it outwards in a fine spray against the inner sides of the reaction vessel. The next lower annular partition then collects the sprayed oil, and its central opening in its turn directs it to the underlying bucket. The lower part of the rotating shaft carries a stirring device, which is in combination with angle iron or similar device attached to the reaction vessel to prevent the liquid attaining circular motion, and an agitator to pre-

vent settlement of the catalyst in the bottom of the vessel. (B) The apparatus described in (A) comprises in addition an inner vertical cylinder or sectional cylinders with inwardly projecting perforated baffles, perforated sectional division plates, and bevel division plates, arranged concentrically about the shaft which carries the distributing reservoirs, and geared to rotate in a similar or opposite direction to the central shaft. The outside of the cylinder has openings which act as gas suckers and force gas against the flow of spray of liquid directed by the central rotating reservoirs.—A. de W.

Fatty oils; Process of treating [hydrogenising] —. C. Ellis. U.S.P. 1,313,255, 15.6.20. Appl., 17.12.15.

FATTY oils containing unsaturated components are brought into contact with a stationary catalytic agent containing active catalyst materially over 1% of the weight of oil which would be in contact with the catalyst at any given time, in the presence of a counter-current of hydrogen at such pressure that no material amount of the metal of the catalyst can remain in combination with the fatty acid of the oil whilst the oily material is cooled to the temperature below which objectionable side reactions occur.—A. de W.

Hydrogen-circulation process [for hydrogenating oils]. C. Ellis. U.S.P. 1,342,668, 8.6.20. Appl., 14.10.16.

THE current of gas containing hydrogen which has circulated through the unsaturated material and catalyst is passed into a larger volume of gas suitable for hydrogenation, and at least the greater part of it left in contact with water for a longer period than it was in contact with the organic material.—C. A. M.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Varnishes; Wearing quality of — compared with their physical and chemical analyses. W. T. Pearce. J. Ind. Eng. Chem., 1920, 12, 552—555.

GOOD grades of exterior and interior varnishes were applied to outside doors exposed to different aspects, and after 9 and 12 months the lustre and condition of the coatings were noted, and compared with viscosity, sp. gr., elasticity, time of drying, etc., and with the composition (total resins, rosin, oils, turpentine, turpentine substitutes, and ash) of the original varnishes. The varnished panels were also tested by placing a moist felt sponge at one end, and if the varnish failed to whiten, the panel was placed in water for definite periods, and allowed to dry. There appear to be definite relationships between the values for viscosity, surface, elasticity, and the sponge test and the results of the service tests. Details of the methods of analysis are given.—C. A. M.

Paracoumarone resin; Use of — in varnishes. W. W. King, F. W. Bayard, and F. H. Rhodes. J. Ind. Eng. Chem., 1920, 12, 549—552.

PARACOUMARONE resins prepared by the polymerisation of the coumarone and indene in aromatic naphthas are now obtainable for commercial purposes of a lighter colour than formerly. Grades suitable for varnish melt between 125° and 145° C.; they retard the polymerisation of tung oil, and satisfactory varnishes may be made by heating a mixture of linseed and tung oils (5 to 40 galls.) with the coumarone resin (100 lb.) at about 2.5° C. per min. to 290°—320° C., then cooling the mixture, adding a suitable quantity of thinner (coal tar solvent), continuing the cooling to below 100° C., and finally adding drier solution (100 pts. of cobalt linoleate

and 84.2 pts. of raw linseed oil heated for 2 hrs. at 200° C., treated with 42.1 pts. of tung oil, again heated, and poured into 600 pts. of coal tar solvent). The resulting varnishes are clear, free from sediment, set in 3 hrs., and dry hard within 20 hrs. The films are brilliant, of a light colour, and resist the action of hot and cold water, ammonia, soap, vinegar, fruit acids, etc. Cobalt linoleate may be replaced by a manganese drier (acetate or linoleate) but a larger proportion is required.—C. A. M.

PATENTS.

Pigments and method of making same. V. M. Goldschmidt, Assr. to Titan Co. A./S. U.S.P. 1,343,446, 15.6.20. Appl., 27.9.18.

TITANIUM compounds are treated with liquids containing a foreign colouring substance in solution or suspension.—A. de W.

Pigment containing titanium-oxygen compound. V. M. Goldschmidt, Assr. to Titan Co. A./S. U.S.P. 1,343,447, 15.6.20. Appl., 10.5.17.

A SOLUTION containing titanium compounds and amorphous silica is evaporated to dryness and the residue heated to expel water and sulphuric acid, and to convert the titanium dioxide into crystalline form.—A. de W.

[Titanic oxide] pigments; Process of producing —. R. Nielsen and V. M. Goldschmidt, Assrs. to Titan Co. A./S. U.S.P. 1,343,468, 15.6.20. Appl., 16.5.17.

A MATERIAL suitable for use as or in the manufacture of a pigment comprising iron oxide with smaller amounts of titanium oxide and calcium sulphate, titanate, and ferrite, is obtained by evaporating a solution containing iron and titanium sulphates from which titanium hydroxides have been precipitated, heating the residue to drive off sulphuric anhydride, adding calcium carbonate to the residue, and so heating the resultant mixture that the SO₂ radicle present is combined to form calcium sulphate, and calcium titanate and ferrite are formed.—A. de W.

Titanium products and the process of producing same from precipitated titanium hydrates. R. Nielsen and V. M. Goldschmidt, Assrs. to Titan Co. A./S. U.S.P. 1,343,469, 15.6.20. Appl., 12.10.17.

THE undesired acid substance contained in titanium hydroxides is eliminated by treating them with a compound of a metal which reacts with the former to produce a number of compounds, one being a neutral and relatively insoluble salt and another a gas.—A. de W.

Turpentine substitute and process of making same. R. C. Bone, Assr. to Esteva-Ruiz and Co. U.S.P. 1,343,387, 15.6.20. Appl., 14.3.18.

KEROSENE is treated with calcium carbide and water simultaneously, the mixture allowed to settle, bleaching powder and hydrochloric acid are added to the liquid which separates, the resultant mixture is allowed to stand, and the supernatant liquid decanted and mixed with calcium oxide.—A. de W.

Phenolic condensation products; Manufacture of —. C. A. Nash. U.S.P. 1,343,575, 15.6.20. Appl., 10.3.19.

A MIXTURE of a fibrous filler and a relatively small quantity of a slowly-volatile solvent for a fusible phenolic condensation product is mixed with a binder consisting of a substantially anhydrous phenolic condensation product containing at least two phenolic groups to each methylene group and a non-volatile methylene body in sufficient quantity

to give to the moulding mixture approximately one phenolic group to each methylene group; the product is subjected to a quick-moulding operation to form a body, and the moulded article after removal from the mould is then hardened by prolonged heat treatment to a hard and substantially infusible form.—A. de W.

Linoleum, liverusta and the like; Preparation of a substitute for —. F. Eckl. G.P. 319,473, 20.6.18.

PLASTIC masses of glycerin and glue hardened with tannin are mixed while hot with a product obtained by boiling wood tar with lime. The resulting plastic material does not dry to a hard mass, but becomes coated with a resistant elastic skin.—C. A. M.

Linoleum substitute; Preparation of —. Deutsche Konservierungsges. m.b.H. G.P. 319,782, 29.11.16.

JUTE or other fabric is coated with cork meal, wood meal, etc., and a binding agent composed of highly chlorinated products of naphthalene, which is mixed in the usual way, while hot, with basic substances, and applied to the fabric with the aid of hot highly-chlorinated naphthalene.—C. A. M.

Printers' roller composition; Apparatus for reclaiming used —. T. H. Grozier. U.S.P. 1,344,270, 22.6.20. Appl., 10.11.19.

SEE E.P. 121,941 of 1918; J., 1919, 378A.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Latex; Method of coagulating —. T. Burney. From E. Nixon-Westwood. E.P. 144,133, 30.7.19.

LATEX is coagulated in a rectangular tank coated or enamelled with acid-resisting material and provided with removable vertical partitions which are introduced after the mixing of the latex and the coagulant; the partitions cause the formation of the coagulum in sheets of convenient size.—D. F. T.

Rubber; Process for vulcanising — and product obtained thereby. I. Ostromislensky, Assr. to New York Belting and Packing Co. U.S.P. (A) 1,342,457 and (B) 1,342,458, 8.6.20. Appl., 29.7 and 24.11.16.

(A) THE harmful effect of the vulcanising agent on an organic dye mixed with rubber is avoided by the additional presence of a substance capable of inducing vulcanisation and of preventing injury to the colouring matter. (B) Rubber is subjected to the action of sulphur and an organic vulcanising agent containing oxygen.—D. F. T.

Rubber vulcanisation and the product thereof. S. B. Molony, Assr. to Michigan Chemical Co. U.S.P. 1,343,224, 15.6.20. Appl., 17.6.19.

THE vulcanisation of rubber is accelerated by the presence of a compound of carbon bisulphide with amines derived from beet sugar residue.—D. F. T.

Dimethylbutadiene. G.P. 319,505. See XX.

XV.—LEATHER; BONE; HORN; GLUE.

Chamoising. IV. W. Moeller. Collegium, 1920, 69—79. Chem. Zentr., 1920, 91, 11., 786. (Cf. J., 1919, 782 A.)

THE author has prepared ozonides from oleic acid and marine animal oil by the Harries method, and investigated their tanning properties alone and in mixture with phenol, the composition of the leather thus produced and of the substances taken

up by the hide. He concludes that the tannage is effected by the undecomposed ozonides, and that only a portion of the oxygen is split off with the formation of peroxides, anhydrides, and lactones which constitute the peptised substance in the tannage.—D. W.

PATENTS.

Iron-tanned leather; Preparation of tough, durable — M., E., and R. Stecher. G.P. (A) 319,705 and (u) 319,859, 11.1.16 and 11.10.17. Addus. to G.P. 314,487 and 314,885 (J., 1920, 121 A, 199 A).

(A) SOLUTIONS of ferrous salts are oxidised with excess of chlorine compounds, preferably chlorates or chloric acid, free from iron. (u) The oxidised iron solution is mixed with solutions of vegetable tanning materials or synthetic tannins, and used for tanning purposes. This process gives a smoother grain than that of the original patent.—D. W.

Condensation products of N-aryl-sulpho derivatives of aromatic aminosulphonic acids [tanning agents]; Preparation of — Ges. für Chem. Ind. in Basel. G.P. 319,713, 12.2.15.

CONDENSATION products of formaldehyde with N-aryl-sulpho derivatives of aminosulphonic acids of the benzene or naphthalene series are readily soluble in water, precipitate albumin from its solutions, and tan animal hides.—C. A. M.

XVI.—SOILS; FERTILISERS.

Soils; The absorbent power of — with respect to manganese. P. Nottin. Comptes rend., 1920, 171, 44—47.

CALCITE is not acted on by a 1% solution of manganoous chloride, whereas aragonite and ktypeite are readily attacked, the calcium going into solution and the manganese being precipitated. The different absorbent powers of soils for manganese salts are to be attributed to a different crystallographic state of the calcium carbonate present in the soils.—W. G.

Soil bacteria; The potential biochemical activity of the spores of — J. R. Neller. Soil Sci., 1920, 9, 329—340.

THE accumulation of ammonia and the production of carbon dioxide resulting from inoculating a sterile soil with soil infusions which have been heated to 85° C. for 10 mins. indicate that the bacterial spores of the soil are capable of energetic activity when supplied with sufficient food and moisture. Evidence is not available as to how they would withstand competition from actinomycetes, non-sporulating bacteria, and fungi in a normal soil flora.—W. G.

Alkali content of soils as related to crop growth. F. T. Shutt and E. A. Smith. Trans. Roy. Soc. Canada, 1919, 13, [iii.], 233—242.

A CONTINUATION of work on the relation of alkali content of Canadian soils to crop growth (see J., 1919, 84 A).—J. R. P.

Nitrogen fixation; Symbiotic — as influenced by the nitrogen in soil. W. A. Albrecht. Soil Sci., 1920, 9, 275—327.

EXPERIMENTS with soya beans and cowpeas indicated that nitrogen fixation will take place in a soil containing large amounts of nitrogen either in the form of nitrates or as organic matter. No injurious effects on nitrogen fixation were observed following on applications of nitrates such as are used in practice. Nodules were produced when large amounts of organic nitrogen were present, and good growth of the leguminous plants was obtained even when sufficient organic matter was present to give large

losses of volatile nitrogen from the soil. Varying the amount of total nitrogen in a soil failed to exert any varying influence on the amount of nitrogen fixed.—W. G.

Nitrogenous fertilisers; Field experiments on the availability of —, 1908—1917. J. G. Lipman and A. W. Blair. Soil Sci., 1920, 9, 371—392.

EQUIVALENT amounts of nitrogen were applied to plots in the form of sodium nitrate, calcium nitrate, ammonium sulphate, calcium cyanamide, dried blood, dried fish, and concentrated liquid manure. One half of each plot was limed and the other half left unlimed. The average yields of dry matter and the percentage of nitrogen recovered were greater with the mineral nitrogenous fertilisers than with the organic manures. Of the four mineral fertilisers sodium nitrate gave the largest yields of dry matter and highest percentage recovery of nitrogen on the unlimed section, and ammonium sulphate the highest on the limed section. Similarly dried fish and dried blood gave respectively the highest returns of the organic manures on the two sections. In all cases there was an average loss of at least two-thirds of the applied nitrogen.—W. G.

Calcium cyanamide; Determination of cyanamide and dicyanodiamide in —. Marqueyrol, P. Lorient, and L. Desvergues. Ann. Chim. Analyt., 1920, 2, 164—167.

VOLUMETRIC methods for the determination of cyanamide and dicyanodiamide depending on the precipitation of the silver compounds in ammoniacal and nitric acid solution, respectively, are untrustworthy owing to the variable silver content of the precipitates. The silver precipitates, however, contain the whole of the cyanamide nitrogen, or the dicyanodiamide nitrogen, and determination of the nitrogen in the precipitates by the Kjeldahl method, as recommended by Caro, gives an accurate measure of the two forms of nitrogen. (Cf. J., 1911, 23, 1129.)

—W. P. S.

Plant juices; The reaction of —. A. R. C. Haas. Soil Sci., 1920, 9, 341—369.

THE reaction of the juice of a plant appears to be affected by changes in illumination, soil solution, age of the plant, and other conditions. The degree of maturity of the plant seems to be an important factor in the degree of actual acidity attained. It is shown that the actual reaction of the juice of many agricultural plants is distinctly acid. The juice of the younger portions of a plant usually has a lower actual acidity than that of the older portions.—W. G.

PATENT.

Sandy soils; Improvement of —. H. Stoltzenberg. G.P. 319,634, 13.5.19.

AMMONIUM fluoride is distributed over the soil, which is then harrowed. The treatment is repeated until the particles of sand have been sufficiently corroded, when they will have an increased absorptive power. The fluoride can be removed by irrigation, prolonged exposure, or chemical treatment.

—D. W.

XVII.—SUGARS; STARCHES; GUMS.

Raw sugars; Deterioration of Cuban — in storage. N. Kopeloff and H. Z. E. Perkins. J. Ind. Eng. Chem., 1920, 12, 555—558.

A NUMBER of bags of Cuban raw sugar were examined chemically and bacteriologically at intervals during 5½ months, at the end of which period it was found that loss of sugar had occurred in all owing

to the action of micro-organisms. In those samples in which initially the number of micro-organisms had been high, deterioration had subsequently been rapid, and some of the highest counts occurred with sugars having factors of safety (moisture/[100—polarisation]) less than 0.30 (cf. J., 1919, 592 A). It is suggested that it may be possible to predict the keeping qualities of a sugar from the number of micro-organisms present; when more than 100 per grm. are counted, and when the factor of safety is greater than 0.2, subsequent deterioration may be expected.—J. P. O.

Carbonatation of beet juices; Microscopical investigation of the —. H. Claassen. Z. Ver. deuts. Zuckerind., 1920, 203—223.

On carbonating a pure solution of calcium saccharate, it is possible to obtain the calcium carbonate in a crystalline or partly crystalline state; but in the case of raw beet juice treated with milk of lime under ordinary factory conditions the precipitate is almost entirely amorphous. During the second carbonatation, however, that is, after filtering off the scum and passing in carbon dioxide to remove the lime still in solution, the precipitate obtained is largely composed of well-formed crystals, mostly rhombohedral. From the point of view of ready filtration of beet carbonatation scums, it is of secondary importance whether the calcium carbonate be in the form of crystals or amorphous grain, the essential condition being the even admixture with the adsorbed colloids of a sufficient amount of calcium carbonate to render the mass porous.—J. P. O.

Beet syrup; Production of [edible] — in beet sugar factories. H. Claassen. Ver. Deuts. Zuckertech., May, 1920. Chem.-Zeit., 1920, 44, 443.

WHILST in beet syrup factories proper, the juice is extracted from the steamed beets by pressure, it is possible to employ the much less costly diffusion process, with certain modifications. The juice is generally acidified with sulphuric acid, but a finer flavour is produced by spontaneous acidification during the diffusion process, for which purpose diffusion may be conducted very slowly or the freshly charged slices may be steamed. The filtered acid juice may be concentrated without excessive frothing by employing a high vacuum and maintaining brisk ebullition. A further heating, e.g., at 104°—108° C., is necessary to effect sufficient inversion to prevent subsequent crystallisation. When the proportion of sucrose has been reduced to not more than 1.5—1.6 times the moisture-content, the syrup should be cooled rapidly to prevent further inversion. Syrups suitable for human consumption may also be similarly produced from beet factory syrups.—J. H. L.

Cane juice; Hot sulphitation of limed —. W. H. T. Harloff. La. Planter, 1920, 64, No. 1, 14.

In order to avoid the formation of calcium sulphite scale on the tubes of heaters in which limed juice which has been sulphited in the cold is raised to 50°—60° C., the following procedure is advised: After heating to 80°—85° C., the raw juice is limed and sulphited simultaneously, conditions being so adjusted that the inflowing gas thoroughly mixes the contents of the tank, while the rate of flow of the milk of lime is such that the liquid is substantially neutral or only very slightly acid to phenolphthalein paper. On completing saturation, the juice is passed through the heaters, and finally is allowed to settle as usual. In this way scale formation in the heater tubes is very much less, settling is more rapid, and the juice is lighter in colour than when the ordinary method is used.

—J. P. O.

Invert sugar; Determination of — in presence of sucrose, by Bruhns' method. P. Beyersdorfer. Z. Ver. Deuts. Zuckerind., 1920, 259—271.

A CRITIQUE of Bruhns' method (Zentr. Zuckerind., 1919, 34, 621) in which, under prescribed conditions, 20 c.c. of sugar solution is boiled with 20 c.c. of Fehling solution (Soxhlet's formula) and the un-reduced copper is determined by treating the cooled solution first with potassium thiocyanate and potassium iodide and then with acid and finally titrating with thiosulphate solution, the results being interpreted by means of tables according to the amount of sucrose present. With fairly pure beet products, containing from 0.05 to 1.3% of invert sugar, it gave results in close agreement with Herzfeld's method. The precise determination of the end point of the titration required considerable practice, and duplicate determinations differed in some cases by 0.2 c.c. in the volume of thiosulphate required. Comparative determinations of reducing sugar in beet molasses by Bruhns', Beyersdorfer's, Schrefeld's, and Hiller's methods, showed occasional small differences between all, which are attributed to the presence of reducing substances other than invert sugar in molasses.

—J. H. L.

Sucrose; Alteration in volume during the inversion of —. N. Schoorl. Z. Unters. Nahr. Genussm., 1920, 39, 113—121.

WHEN 1 mol. of sucrose is hydrolysed, the 18 g. of water required undergoes a contraction of 6.9 c.c., which corresponds with a contraction of 0.5 c.c. for 26 g. of sucrose per 100 c.c. of solution. The extent of the contraction varies slightly with the catalyst used; it is greatest with oxalic acid and invertase and least with hydrochloric acid. The relation of the contraction to the determination of sugars by means of the sp. gr. is discussed.—W. P. S.

Maple products; Analysis of —. II. Composition of maple syrup and of maple sugar sand and the detection of adulteration of the syrup. J. F. Snell. Trans. Roy. Soc. Canada, 1919, 13, [iii.], 221—231.

THE average content of calcium malate in washed maple sugar sand was found to be 70%, corresponding with 55% of malic acid (cf. J., 1911, 980). In samples received in 1914 an average of 47.4% of malic acid was found. The recovery in laboratory experiments was 75% of the acid as shown by analysis. Adulteration of maple sugar with refined sugar leaves the sucrose content unchanged, but reduces the percentages of non-sugar constituents. Detection of adulteration is based on the content of one or more non-sugar constituents. The salts are most abundant, and the conductivity method of estimating these is preferred to others except the volumetric lead method (J., 1916, 482). Tables of standard values are given.—J. R. P.

Sucrose; Biochemical preparation of — from gentianose. E. Bourquelot and M. Bridel. Comptes rend., 1920, 171, 11—15.

EMULSIN freed from invertase decomposes gentianose, giving dextrose and sucrose. The sucrose can be isolated in a pure crystalline state after converting the dextrose into β -methylglucoside.—W. G.

Radium radiation; Action of — on sucrose and agar. A. Fernau. Biochem. Zeits., 1920, 102, 246—251.

SUCROSE is inverted by the action of radium radiation. Agar submitted to the same influence liquefies with the production of sugars—most probably galactose and arabinose—which strongly reduce Fehling's solution. The chemical change in both cases is due to the primary action of the radium.

—S. S. Z.

PATENT.

[Steffens' process.] Method of recovering by-products from waste liquors [from —]. G. M. Bradford and J. E. Broadhead. U.S.P. 1,312,737, 8.6.20. Appl., 16.9.18.

THE waste liquor, concentrated to about 30° B. (sp. gr. 1.26), is subjected to the action of an electric current of sufficient intensity to produce an arc in the liquid, whereby evaporation and electrolysis are produced, followed by complete combustion. The volatile products are recovered as well as the residue, which contains potash.—J. H. L.

XVIII.—FERMENTATION INDUSTRIES.

Mucors; Action of different acids on the growth of —. Bettinger and Delaval. Bull. Assoc. Chim. Suer., 1920, 37, 254—261.

IN employing mucors for the saccharification of distillery mash the starch is first liquefied either by diastase or by acids (cf. J., 1920, 244A). In rice mash liquefied by diastase without any addition of acid, growth of the mucus is less than in mash treated with a small quantity of hydrochloric, sulphuric, oxalic, tartaric, phosphoric, succinic, or acetic acid, equivalent to about 5 l. of commercial hydrochloric acid per 1000 kg. of grain. The optimum amount varies with different acids, and is in general greater the lower the degree of ionisation of the acid; if the optimum is exceeded growth is more or less inhibited. Acetic and succinic acids, which are excretion products, act favourably on growth at very low concentrations, but with increasing concentration they exert an inhibitive action much greater than can be accounted for by their degree of ionisation.—J. H. L.

Yeast (Saccharomyces thermantitonus). H. von Euler and I. Laurin. Biochem. Zeits., 1920, 102, 258—268.

A STRAIN of *Saccharomyces thermantitonus* emanating from the Institut für Gärungsgewerbe, in Berlin, was examined by the authors for its growth, fermentation, and inversion capacities. They found that the limits of temperature characteristic for this species have altered after 15 years' cultivation in the laboratory on thin beer wort. A similar observation has been previously made by the authors on a strain of *Saccharomyces thermantitonus* which came from Jørgensen's laboratory. It is, therefore, concluded that the yeast during the years of cultivation in the laboratory adapted itself to a lower temperature.—S. S. Z.

Catalase; Chemical kinetics of —. E. Yamazaki. Sci. Rep. Tôhoku Imp. Univ., 1920, 9, 59—95.

THE enzymes from *Glycin hispida*, Maxim, and from the blood of cattle catalyse hydrogen peroxide according to the same laws as that from *Phyllostachys mitis*, Riv. (cf. J. 1920, 463 A), but the temperature coefficients are different. The rate of decomposition of hydrogen peroxide by catalase is reduced to zero by the addition of $N/1200$ mercuric chloride, potassium cyanide, hydroxylamine, and phenylhydrazine; strong oxidising agents like potassium chlorate, potassium nitrate, and potassium bichromate retard the reaction very much, whilst manganous sulphate, soluble starch, glycocoll, and sucrose accelerate it. (Cf. J.C.S., Aug.)—J. F. S.

Urease; Chemical kinetics of —. E. Yamazaki. Sci. Rep. Tôhoku Imp. Univ., 1920, 9, 97—136.

UREASE from soya beans decomposes urea in two stages thus: $CO(NH_2)_2 + NH_4CO_2NH_2 \rightarrow (NH_4)_2CO_3$. The reaction is retarded by the addition of electrolytes such as hydrochloric acid, silver sulphate, and mercuric chloride. (Cf. J. C. S., Aug.)—J. F. S.

Enzyme action; Mechanism of —. E. Yamazaki. Sci. Rep. Tôhoku Imp. Univ., 1920, 9, 137—142. A NUMBER of hypotheses of the mechanism of enzyme action are discussed.—J. F. S.

Wine analysis. W. Fresenius and L. Grünhut. Z. anal. Chem., 1920, 59, 49—79.

DIRECTIONS are given for determining the sp. gr., alcohol content, and total solids in the same sample of wine. A table is given for correcting the sp. gr. to 15°/4° C., when the determination has been made at 17.5°/4° C.—W. P. S.

Acetone and butyl alcohol; Seed-culture methods in the production of — by a fermentation process. H. B. Speakman. J. Ind. Eng. Chem., 1920, 12, 581—587.

IN the production of acetone and butyl alcohol by fermentation (J., 1919, 155 r) the acidity of the mash rises for 15 hrs. and then falls for the same period, except in the presence of certain organisms which cause abnormal rise in the acidity. The seed cultures should be transferred when the acidity begins to fall. The seed-culture plant is made of copper; this soon becomes coated with a black deposit, which reduces the formation of toxic metallic salts. Laboratory cultures are started from the products of a fermentation containing maize residues and spores of the bacillus in a tube which has been kept for 3 months in a dry place. Mash contained in tubes constricted in the middle is inoculated from this first tube, and the tubes sealed at the constriction. After 24 hrs. the tubes are opened and incubated at 37° C., the gas developed forming an anaerobic atmosphere. From these tubes flasks of 300 c.c. of 5% mash are inoculated next day, and larger quantities from these. The period of the final fermentation is reduced by keeping the number of generations between the spore culture and fermentation as few as possible. After inoculation of the main mash (24,000 galls.) from a seed tank (500 galls.), the valves of the vessel are closed until the fermentation begins, and a slight pressure is then maintained by regulating the flow of gas through the air-valve.—C. A. M.

PATENTS.

Alcohol; Manufacture of — [from rice straw]. A. B. C. Rogers and C. H. Bedford. E.P. 144,079, 27.5.19.

RICE straw, with or without rice husk, is steamed, preferably with superheated steam, then converted into pulp and digested with dilute acid, and the liquid obtained is neutralised and fermented. The steaming process may be effected in two stages, the uncondensed steam from the second stage being utilised for the preliminary steaming; and during the steaming process the material may be subjected to the action of hydrochloric acid vapour or atomised calcium hypochlorite and chlorine with or without hydrochloric acid. An arrangement of plant is also claimed.—J. H. L.

Alcohol-reduced beer; Manufacture of —. H. Heuser. Reissue 11,889, 22.6.20, of U.S.P. 1,302,551, 6.5.19. Appl., 3.7.19.

SEE J., 1919, 551 A.

XIXA.—FOODS.

Milk; Pasteurisation of —. Orla-Jensen. Milchw. Zentr., 1920, 49, 45—53. Chem. Zentr., 1920, 91, ii., 780.

THE low pasteurisation method (heating at 60°—70° C. for 30 mins.) is the best for milk intended for infants' food and for cheese making, since the enzymes are not destroyed. Cream for butter making should be heated at 95° C. for 1 min. and then

cooled rapidly, whilst milk used as a medium for the culture of lactic acid bacteria should be pasteurised by heating at 85° C. for 1 hr. (Cf. Ayers, J., 1916, 323.)—W. P. S.

Milk serum obtained by spontaneous curdling. J. Grossfeld. Z. Unters. Nahr. Genussm., 1920, 39, 140—145.

The serum obtained by allowing milk to curdle spontaneously in a closed vessel may be used for all the usual serum determinations which serve to detect the presence of added water in the original milk. The only correction necessary is that for the effect of the acidity on the sp. gr.; for each c.c. of N/10 alkali solution required to neutralise 10 c.c. of the serum, 0.00011 is added to the sp. gr. found.—W. P. S.

Milk; Influence of various carbohydrates on the curdling of —. E. Aschenheim and G. Stern. Biochem. Zeits., 1920, 102, 98—124.

MIXTURES of milk with water, oat jellies, or oatmeal extracts were curdled with rennet under conditions resembling those prevailing in the stomach. The mixture with water showed more consistent conditions of coagulation than the oat jelly and oatmeal extract mixtures. The oat jelly mixture produced, however, a curd resembling more the curd formed on curdling human milk than the oatmeal extract mixture. The addition of sugar reduced these differences in coagulation. Lactose influenced this condition in a more pronounced manner than "nutritive" sugar (Nährzucker) and sucrose.—S. S. Z.

Hydrocyanic acid produced by beans; Determination of —. A. Czapski. Z. anal. Chem., 1920, 59, 80.

Less than one-half of the free hydrocyanic acid actually produced is found in the distillate when paraffin wax is added to the distillation flask to prevent excessive frothing of the contents during distillation.—W. P. S.

Proteins; Chemistry of the —. E. Herzfeld and R. Klinger. Biochem. Zeits., 1920, 102, 89—98.

To obtain protein free from adhering impurities, such as the products of protein degradation, fats, lipoids, etc., the clear extract from the tissues is precipitated with 5—6 vols. of 90% alcohol and heated on a boiling-water bath. It is then filtered or centrifuged and the residue is treated with boiling absolute alcohol and again with boiling ether. The protein powder thus obtained is further extracted with boiling water to remove albumoses, peptones, and salts. For the detection and estimation of the higher polypeptides in protein solutions, the protein and the higher polypeptides are precipitated with boiling 90% alcohol, the precipitated fraction is extracted with boiling water; and the nitrogen is estimated in the aqueous extract. Using this method, it was found that all colloidal protein solutions contain appreciable quantities of albumoses. 100 c.c. of human serum contains a quantity of albumoses and peptones equivalent to 0.4 g. of Witte's peptone.—S. S. Z.

Beckmann's process; Nutritive value of straw and chaff hydrolysed by —. Replacement of protein by urea in the diet of growing animals. W. Völtz. Biochem. Zeits., 1920, 102, 151—228.

EXPERIMENTS on a full-grown sheep and three lambs showed that urea could replace the protein in the metabolism of ruminants. The urea is converted in the digestive tract of the animals into bacterial protein, of which 80—90% is eventually absorbed by the system. The requisite carbohydrate for the synthesis of the protein can be supplied by sugar or starch or even to some extent by cellulose. Straw

hydrolysed by Beckmann's process (J., 1919, 384 A) produces a food of higher nutritive value than that obtained by the Lehmann and Colsmann process.—S. S. Z.

Oil-cake; Importance of — as a fodder and, in particular, the value, utilisation, and toxicity of beech nuts. T. Sabalitschka. Ber. deuts. Pharm. Ges., 1920, 30, 259—277.

THE toxic action of beechnut meal is due to the presence of soluble oxalic acid to the extent of 0.51%, probably in the form of potassium hydrogen oxalate, a conclusion which is in full harmony with the physiological symptoms. The poison may be removed by extracting the cake with about five times its weight of cold water during several hours; the residue is still a valuable fodder, but should not be preserved, as it is particularly liable to attack by fungi.—H. W.

Rhubarb juice. J. Angerhausen. Z. Unters. Nahr. Genussm., 1920, 39, 122—130.

ANALYSIS of four samples of rhubarb (stalk) juice are recorded:—Sp. gr., 1.0187—1.0237; total solids, 3.69—4.96; total sugar (as invert sugar), 1.21—2.08; ash, 0.62—0.77; oxalic acid, 0.23—0.32; citric acid, 0.08—0.24; malic acid, 2.0%; free acidity (c.c. N/1 acid), 26.5—29.6. Free oxalic acid is not present in the juice. The free acidity decreases when the juice is fermented, and the whole of the combined oxalic acid may be removed by suitable treatment with calcium carbonate.—W. P. S.

Agar and radium radiation. Fernau. See XVII.

PATENT.

Yeast; Manufacture of foods or stimulants for promoting the growth of — after the yeast has been mixed with other ingredients into the form of dough. W. A. and E. W. Geere. E. P. 143,938, 4.8.17.

A YEAST food consists of a dry mixture of cereal germs which have not previously been heated, cereal meal, starch, ammonium carbonate, and a phosphate.—J. H. L.

XIXB.—WATER PURIFICATION; SANITATION.

Carbon dioxide; Determination of — in moorland waters. V. Rodt. Chem.-Zeit., 1920, 44, 469.

THE presence of free weak organic acids and of their salts in moorland waters interferes with the determination of the free and bicarbonate carbon dioxide. In many cases where the solvent action of the water on limestone is required Heyer's method (Gesundheitsing., 1912, 35, 669) is applicable. The water is shaken at intervals for 3 days in a closed flask with powdered marble, and an aliquot portion then titrated with N/10 acid, with methyl orange as indicator. The results are expressed in mg. CaO per l. The difference between the results of titration of the water before and after the treatment with marble gives the amount of CaCO₃ dissolved. (Cf. J.C.S., Aug.)—C. A. M.

Sewage; Purification of — by activated sludges. R. Cambier. Comptes rend., 1920, 171, 57—60.

THE ferrous sulphide normally present, or artificially introduced, into sludges, appears to be an important factor in the purifying and nitrifying action of these sludges on sewage. It is frequently noticed that when such sludges lose their activity a reddish colour due to ferric hydroxide is apparent. All the causes which tend to destroy the ferrous sulphide or modify its physical state equally tend to destroy the activity of the sludge. It was found that sludges which had become practically inactive

could be re-activated by the addition either of ferrous sulphide or of an equimolecular proportion of solutions of ferrous sulphate and ammonium sulphide.—W. G.

Dichloroethyl sulphide: Toxic action of —. A. Mayer, H. Magne, and L. Plantefol. *Comptes rend.*, 1920, 170, 1625—1628.

DICHLOROETHYL sulphide acts as a general poison. Its action on the various parts of the organism are described.—W. G.

PATENTS.

Germicide and insecticide. L. Cheeseman. U.S.P. 1,313,031, 8.6.20. Appl., 6.3.19.

THE product contains barium hydroxide and hydro-sulphide mixed with another alkaline-earth base.
—J. H. L.

Sewage and other impure liquids; Aeration of —. O. Stott and E. R. Jones. U.S.P. 1,313,797, 15.6.20. Appl., 21.6.19.

SEE E.P. 133,722 of 1918; this J., 1919, 961 A.

Polysulphide solutions [insecticides]; Method of treating —. E. C. Holton, Assr. to Sherwin-Williams Co. Reissue H.890, 22.6.20, of U.S.P. 1,251,908, 29.1.18. Appl., 6.12.19.

SEE J., 1918, 193 A.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Albumins and antitoxins; Behaviour of — towards the electric current and isolation of pure antitoxic albumin from diphtheric serum by the electro-osmotic method. W. G. Rappel. *Ber. deuts. pharm. Ges.*, 1920, 30, 314—328.

PURE antitoxic albumin can be obtained from diphtheric serum by subjecting the latter in a divided cell (the central portion of which contains the serum and is separated from the outer portions which contain the electrodes and pure water by suitable semi-permeable membranes) to the action of an electric current. Electrolytic impurities are thereby first removed, after which euglobulin is precipitated; from the filtered solution the paraglobulin and albumin can be further separated by utilising the fact that the latter is more highly charged than the former, and, in consequence, has a greater rate of migration. The antitoxin is mainly combined with the paraglobulin. (*Cf.* J.C.S., Aug.)—H. W.

Digitalis leaves; Valuation of —. G. Joachimoglu. *Arch. Exp. Path. Pharm.*, 1920, 86, 307—312.

THE potency of extracts from digitalis leaves prepared in various ways was studied. Extraction with absolute alcohol in a Soxhlet apparatus yielded the most potent preparation. By extracting the leaves first with water and then with 50% alcohol the extract had only 60% of the potency of the absolute alcohol extract. Tinctures prepared according to the formula of the German pharmacopœia contained only 75% of the active principle, while tinctures prepared by percolation according to the American pharmacopœia were more active. Chloroform extracted only one-third of the glucoside. Extraction with benzene and subsequent extraction with absolute alcohol yielded 70% of the active principle. Acetone extracted the glucoside extremely slowly. The minimum lethal dose per gram of frog was used as the unit in this investigation.—S. S. Z.

Dulcin (p-phenetoleurea); Alteration in the taste of the sweetening agent — as a result of chemical change. H. Thoms and K. Nettesheim. *Ber. deuts. pharm. Ges.*, 1920, 30, 227—250.

p-PHENETOLEUREA loses its sweetening power when substituents are introduced into the benzene nucleus. (*Cf.* J.C.S., Aug.)—H. W.

p-Hydroxyphenylurea; Sweetening power of derivatives of —. F. Boedecker and R. Rosenbusch. *Ber. deuts. pharm. Ges.*, 1920, 30, 251—258.

ATTEMPTS to obtain a sweetening agent analogous to dulcin (p-phenetoleurea) but possessing greater sweetening power and greater solubility in water were unsuccessful. Hydroxy-p-ethoxyphenylurea, $\text{HO}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{O}\cdot\text{C}_6\text{H}_4\cdot\text{NH}\cdot\text{CONH}_2$, m.p. 160°C ., is more soluble in water than dulcin, but is less sweet and has also a bitter after-taste; dihydroxy-p-propoxyphenylurea, $\text{CH}_2(\text{OH})\cdot\text{CH}(\text{OH})\cdot\text{CH}_2\cdot\text{O}\cdot\text{C}_6\text{H}_4\cdot\text{NH}\cdot\text{CONH}_2$, m.p. 156°C .— 157°C ., has no sweet taste. (*Cf.* J.C.S., Aug.)—H. W.

Binary solutions; Influence of substitution in the components on the equilibrium in —. AXIII. Binary system antipyrine-benzoic acid. R. Kremann and H. Markl. *Monatsh. Chem.*, 1920, 41, 1—1.

ANTIPYRINE and benzoic acid when melted together form a single equimolecular compound, m.p. 66°C . This compound forms eutectics with antipyrine containing 63.5% of antipyrine at 59.5°C ., and with benzoic acid containing 50.5% of antipyrine at 59.5° .—J. F. S.

Oxalic acid and lactic acid; Detection of — especially their differentiation from tartaric acid. K. Brauer. *Chem.-Zeit.*, 1920, 44, 491.

OXALIC acid gives a bright violet coloration when heated with concentrated sulphuric acid and resorcinol; under similar conditions lactic acid and tartaric acid give red colorations. Heating with resorcinol and dilute sulphuric acid (1:1, or even weaker concentration) serves to distinguish lactic acid from tartaric and oxalic acids; the colorations obtained with this test are:—lactic acid, red; oxalic acid, green; tartaric acid, yellow.—W. P. S.

Nitroform; Estimation of — by potassium permanganate. P. V. McKie. *Chem. Soc. Trans.*, 1920, 117, 646—648.

NITROFORM may be accurately estimated, even in presence of nitric and nitrous acids, by titration with permanganate in acid solution according to the equation: $\text{CH}(\text{NO}_2)_3 + 2\text{O}_2 + \text{H}_2\text{O} = \text{CO}_2 + 3\text{HNO}_3$. The nitroform solution is added to a known excess of N/10 permanganate acidified with sulphuric acid, the mixture is heated to 30°C ., and after remaining 1 min. at that temperature potassium iodide is added and the liberated iodine titrated with thiosulphate. In nitration products the nitrous acid must be destroyed by boiling for about 1 min. with ammonium nitrate, the solution being then cooled to 30°C . and titrated as above. As an alternative the nitroform may be distilled in a current of steam from the product after treatment with ammonium nitrate (30 mins. distillation required) and the distillate titrated.—G. F. M.

Charcoal; Heat of adsorption of vapours on —. A. B. Lamb and A. S. Coolidge. *J. Amer. Chem. Soc.*, 1920, 42, 1146—1170.

THE heat of adsorption of carbon tetrachloride, ether, chloroform, ethyl formate, carbon bisulphide, methyl alcohol, ethyl chloride, ethyl bromide, ethyl iodide, benzene, and ethyl alcohol by charcoal at 0°C . is represented by the equation $h = m\epsilon^n$, where h is the heat of adsorption per normal c.c., ϵ the number of c.c. adsorbed, and m and n characteristic constants. The molecular heats of adsorption are nearly alike in all cases, the extremes being 12.0 cal. (ethyl chloride) and 15.5 cal. (carbon tetrachloride), and the values are the same with charcoals of different adsorptive activity. The net heats of adsorption per c.c. of liquid are practically identical in all cases. Vapours containing halogens appear to react with the charcoal when warmed, so as to decrease the heat evolved by any subsequent adsorption.—J. F. S.

Methyl alcohol; Determination of —. A. Heiduschka and L. Wolf. Pharm. Zentralh., 1920, 61, 361–366.

A SUITABLE quantity of the alcohol is placed in a cylindrical glass bulb of about 150 c.c. capacity, provided with a sealed-in tube reaching to the bottom, and an exit tube; 30% sulphuric acid and an excess of potassium bichromate solution are added, the ends of the two tubes are drawn out to capillaries and sealed, and the bulb is immersed in boiling water for one hour. After cooling the exit tube is connected with a calcium chloride tube and a weighed potash bulb, the capillary point is broken, and the carbon dioxide formed absorbed in the potash bulb. The capillary point on the other tube is broken after connexion with an apparatus supplying a current of air free from carbon dioxide. When all the carbon dioxide has been collected the excess of bichromate is titrated. The amount of methyl alcohol present is calculated either from the weight of carbon dioxide found or the amount of oxygen required for the oxidation. The method may be used for the determination of methyl and ethyl alcohols in mixtures of the same. The acetic acid resulting from the oxidation of the ethyl alcohol is retained by the solution and by the calcium chloride tube during the absorption of the carbon dioxide, and does not enter the potash bulb. Since 0.8% of the ethyl alcohol present is invariably oxidised to carbon dioxide, a corresponding allowance must be made in calculating the quantity of methyl alcohol. The methyl alcohol produced by the hydrolysis of cocaine may be determined by the method.—W. P. S.

See also pages (A) 543, *Adsorption by charcoal* (Pickles). 550, *Cod-liver oil* (De Kadt). 557, *Dichloroethyl sulphide* (Mayer and others). 558, *Conductometric titrations* (Kolthoff). 559, *Benzoic acid etc.* (Guerbet).

PATENT

Dimethylbutadiene; Manufacture of —. Farbenfabr. vorm. F. Bayer und Co. G.P. 319,505, 2.9.16.

PINACONE chlorhydrin is treated with compounds which will combine with hydrochloric acid but do not contain hydroxyl groups, e.g., ammonia or organic bases. A mixture of water and dimethylbutadiene is obtained by distilling a mixture of pinacone chlorhydrin and dimethylaniline between 60° and 80° C.—D. W.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Colloidal silver; Formation of — in photographic developers and means of avoiding it. L. Lobel. Bull. Soc. Franc. Phot., 1920, 7, 21–22.

IN using developing solutions containing sodium sulphite small quantities of silver bromide pass into solution. This occurs, in general, more easily with papers than with plates, since the bromide is in a finer state of division, and more easily with developers having a low content of alkali carbonate in proportion to the sulphite. The dissolved silver salt is reduced by the developer, and part of the reduced silver is deposited on the dishes, etc., and part remains in colloidal solution. Continued use of the developer tends to the production of dichroic fog by deposition of the colloidal silver in the body of the film, and solutions still having considerable developing power may be rendered useless on this account. Ordinary filtering methods will not hold back the colloidal silver. If, however, about 20% of sodium sulphate is added the colloidal silver is coagulated and deposited; its formation is also pre-

vented by the addition of the same salt to a new developer.—B. V. S.

Cyanine dyes. Mills and Wishart. See IV.

XXII.—EXPLOSIVES; MATCHES.

Nitric esters [cordite]; Decomposition of — by lime. T. M. Lowry, K. C. Browning, and J. W. Farmery. Chem. Soc. Trans., 1920, 117, 552–561.

HYDROLYSIS or saponification of the normal type is not the predominant action when cordite is decomposed by lime in the presence of pyridine. Two partly denitrated nitrocelluloses, of approximate formulae $C_{12}H_{18}O_8(NO_3)_2$ and $C_{12}H_{17}O_7(NO_3)_3$, were obtained as by-products of incomplete decomposition, but no cellulose was obtained when the decomposition was carried to completion. Traces of glycerol were detected only when a very large excess of pyridine was used, and these were likewise the only conditions in which a clean crystalline calcium nitrate was obtained. After calcium oxalate, nitrate, and nitrite, the calcium salts of hydroxypropionic acid and dihydroxybutyric acid appeared to be the most important products of decomposition, the former being present in every case where the products were reported to have a toxic action on plants. It is suggested that the main action is a decomposition of the nitric esters into ketones or aldehydes and a nitrite, instead of hydrolysis into an alcohol and nitric acid.

—G. F. M.

Cellulose solutions. Gibson and others. See V.

Pyroxylin solutions. Higgins and Pitman. See V.

XXIII.—ANALYSIS.

Viscosimeter; Falling sphere —. W. H. Gibson and L. M. Jacobs. Chem. Soc. Trans., 1920, 117, 473–478.

A TUBE 29 cm. long and 2 cm. internal diam. is arranged vertically in a bath provided with a stirrer and thermometer. The tube is divided into three 5 cm. lengths for measurement of the time of fall, and a further 5 cm. above for the sphere to acquire uniform velocity. The sphere is a steel ball, 0.15 cm. diam., as used for ball-bearings, and it is introduced into the viscosimeter tube by means of a short length of glass tubing, 3 mm. diam., fixed by means of a rubber stopper and adjusted to dip 3 cm. below the surface of the liquid. The theory of the falling sphere viscosimeter is briefly discussed, and the following equation expressing viscosity is developed:

$$9\eta S(1+2.4x)(1+3.3r/h)=2gr^2(s-\sigma)T,$$

where T is the time of fall through a length, S, r is the radius and s the density of the sphere, σ the density of the liquid, x the ratio of the radius of the sphere to that of the viscosimeter tube, and h the height of the liquid. This equation reduces itself to $\eta=K(s-\sigma)T$, where K is the numerical constant involving all the corrections for the particular tube. This constant having been found, either by the use of the first equation above, or by experiment with a liquid of known viscosity, determinations for other substances simply resolve themselves into determinations of s, σ and T. For castor oil at 20° C. the value $\eta=9.888$ was found by this method, a figure in close agreement with Kahlbaum and Raber's value.—G. F. M.

Conductometric titrations in neutralisation analysis. III. Titration of combined weak acids or bases in salts. [Displacement curves.] I. M. Kolthoff. Z. anorg. Chem., 1920, 111, 97–108.

The quantity of a weak acid or base combined as a

salt may be estimated by titration with a strong base or acid respectively, using the electrical conductivity as indicator. On adding a strong acid, say hydrochloric acid, to the salt of a weak acid, the conductivity of the solution changes very little until all the weak acid has been liberated, but the first drop of strong acid in excess causes a great increase in the conductivity. On plotting the conductivity against the number of c.c. of standard acid a sharp break will be found, which represents the point of equivalence. The method is found to work well in all cases where after displacement of 50% of the weak acid the conductivity has not increased more than 1%. Examples are given of the titration of sodium acetate and sodium formate, and for weak bases ammonium chloride. In the case of the dibasic acids in sodium oxalate, sodium tartrate, and sodium citrate equally good results may be obtained if in the two last-named cases an equal volume of alcohol is added to the solution before titration.—J. F. S.

Benzoic acid; Reaction of — based on its diazotisation. Application to the toxicological detection of atropine, cocaine and stovaine. M. Guerbet. *Comptes rend.*, 1920, 171, 40—41.

The method consists in nitrating the benzoic acid, reducing the mixed nitrobenzoic acids, diazotising the aminobenzoic acids, and coupling the diazo compounds with β -naphthol to form an orange-red mixture of azo dyes. It is equally applicable to substances such as cocaine or stovaine, which contain a benzoyl group, or atropine, which on oxidation yields benzoic acid. The test may be carried out in a few minutes on a watch-glass with 0.0001 g. of material without isolating or purifying the intermediate products. For toxicological work the test may be applied to extracts of organs prepared by Stas' method. (*Cf.* J.C.S., Aug.)—W. G.

See also pages (A) 538, *Coal analysis* (Sinnatt); *Volatile matter in coal* (Sinnatt and Grounds). 541, *Pyroxylin solutions* (Higgins and Pitman). 542, *Sulphuric acid* (Scheringa). 543, *Mercuric salts* (Jolibois and Bouvier); *Helium and hydrogen* (McLennan and Elworthy); *Helium in gases* (Murray). 546, *Optical glass* (von Biehowsky). 548, *Phosphorus in bronzes* (Bertiaux). 550, *Cod-liver oil* (De Kadt). 553, *Calcium cyanamide* (Marqueyrol and others). 554, *Invert sugar* (Boyersdorfer); *Sucrose* (Schoorl). 555, *Wine* (Fresenius and Grünhut). 556, *Milk serum* (Grossfeld); *Hydrocyanic acid* (Czapski); *Carbon dioxide* (Rödt). 557, *Digitalis leaves* (Joachimoglu); *Oxalic, lactic, and tartaric acids* (Brauer); *Nitroform* (McKie). 558, *Methyl alcohol* (Heiduschka and Wolf).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Adam. Apparatus for catalytic etc. reactions. 21,258. July 14.
Adler and Knöpfmacher. Purification etc. of gases. 21,847—8. July 21. (Austria, 12.4 and 8.8.19.)
Aymard. Method of filtering. 21,405. July 15.
Babcock. Evaporators. 20,513—4. July 10. (U.S., 14.4.17.)
Berkerson, Ltd., and Wade. Recovery or separation of colloidal matter from liquids. 21,363. July 15.

Borzykowski. 21,460. *See V.*
Chenard. Separation of liquids from gases etc. 20,647. July 10. (Fr., 24.12.18.)
Danhardt. Filters for gases. 20,626 and 20,631. July 10. (Ger., 26.10 and 25.2.18.)
Deutsche Maschinenfabrik A.-G. Apparatus for separating solids from gases etc. 20,633 and 20,985. July 10 and 12. (Ger., 2.12 and 12.5.18.)
Deutsche Maschinenfabrik A.-G. Removing dust from filters in dry gas purifying plants. 20,630. July 10. (Ger., 2.12.18.)
Head, Wrightson and Co., and others. 21,797. *See X.*
Heylandt Ges. Liquefaction of gases. 20,986—7, 20,989. July 12. (Ger., 27.8.17, 8.5.19, 1.12.16.)
Herman. Compressed gases, and compositions containing same. 21,015. July 12.
Heylandt Ges. Developing gas under pressure from liquid gases. 20,990. July 12. (Ger., 27.8.17.)
Lecesse. Calcining-furnaces. 20,458. July 10. (Fr., 3.4.19.)
Lloyd. Lubricants for internal-combustion engines. 21,220. July 14.
Metallatom Ges. Atomising fusible materials. 20,599. July 10. (Ger., 5.2.18.)
Metcalfe. Continuous tunnel kilns. 20,612. July 10.
Pfeiffer. Air-heater for drying plants. 20,689. July 10. (Ger., 7.2.19.)
Ransford (Aluminium Welding Works). Rectifying columns for stills. 21,039. July 12.
Robson. Drying apparatus. 21,703. July 19.
Rohn. Liquefaction of gases. 20,988. July 12. (Ger., 27.8.16.)
Rohn. Vessels for conveying and storing liquefied gases. 20,991. July 12. (Ger., 27.8.16.)
Rummel. Preventing incrustation in boilers etc. 20,602. July 10. (Ger., 24.4.18.)
Seaman. Refrigerants and refrigeration. 20,690—1, 20,693—4. July 10. (U.S., 5.2 and 27.5.18.)
Soc. Anon. Fours Spéciaux. Distilling mineral and organic substances. 20,596. July 10. (Belg., 30.6.19.)
Wade (Sprengluft Ges.). Producing vacuum by means of charcoal. 21,408. July 15.
Wüster. Filters. 20,888. July 12. (Ger., 16.3.18.)

COMPLETE SPECIFICATIONS ACCEPTED.

8424 (1919). Duveusart. Exchanging the heat of liquids. (146,579.) July 21.
8557 (1919). Soc. Chim. Usines du Rhône. Carrying out chemical reactions by catalysis. (126,279.) July 21.
9379 (1919). Simon and Simon. Rotary-cylinder drying machines. (146,635.) July 21.
10,064 (1919). Pedersen. Separating liquid mixtures or emulsions. (125,981.) July 28.
18,718 (1919). Jackson (Grimson-Russell Co.). Evaporators. (146,730.) July 21.
21,553 (1919). Krause. Evaporating liquids. (132,256.) July 28.
21,694 (1919). Allsop and Sibson. Drying machines. (146,744.) July 21.
28,071 (1919). Bigot. Tunnel furnaces. (147,391.) July 28.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Armstrong. Manufacture of briquettes. 20,887. July 12.
Ashcroft. Synthetic manufacture of waxes, oils, and/or spirits of the petroleum and coal tar series. 21,728. July 20.
Benson. Removing reaction products from hydrocarbon substances. 20,646. July 10.

Bergius. Manufacture of liquid or soluble organic compounds from coal. 20,383. July 10. (Ger., 22.8.16.)

Bibby. Drying coal etc. 21,887. July 21.
Birkholz. Production of illuminating gas from coal. 20,658. July 10. (Switz., 22.10.15.)

Colmer. Coal substitute. 21,002. July 12.
Dessy. Furnaces for distilling fuels. 20,622. July 10.

Deutsche Erdöl A.-G. Converting hydrocarbons into fatty acids. 20,715. July 10. (Ger., 12.7.19.)

Deutsche Erdöl A.-G. Separating solid and liquid hydrocarbons. 21,906. July 21. (Ger., 31.7.19.)

Franke. Manufacture of dry peat. 21,266. July 14.

Garrow and Nielsen. Apparatus for destructive distillation of carbonaceous material etc. 21,558. July 17.

Head, Wrightson and Co., and others. 21,797. See X.

Hennebutte. Vertical retort. 20,777. July 10. (Fr., 12.3.18.)

Lloyd. Manufacture of coal etc. briquettes. 20,533. July 10.

Lloyd. 21,220. See I.
Maclaurin, and Scottish Bye-Products, Ltd. Manufacture of lubricating greases. 22,136. July 24.

Morgan, and Thermal Industrial and Chemical Research Co. Distillation of oils. 21,903. July 21.

Pintsch A.-G. Gas-producers. 20,577. July 10. (Ger., 18.5.18.)

Riedel. Recovery of nitrogen compounds from coal etc. 20,603. July 10. (Ger., 8.11.15.)

Riedel. Manufacture of coke for blast furnaces. 20,605. July 10. (Ger., 3.1.16.)

Riedel. Recovery of ammonium chloride from fuel. 20,606. July 10. (Ger., 29.2.16.)

Riedel. Recovery of nitrogen from fuel. 20,607-8. July 10. (Ger., 29.2.16, 16.7.17.)

Riedel. 20,922. See XVI.
Scherk. Fractional distillation of poor fuels. 20,570. July 10. (Ger., 26.10.18.)

Soc. Anon. Fours Spéciaux. 20,596. See I.
Sudfeldt and Co. 20,584. See III.

COMPLETE SPECIFICATIONS ACCEPTED.

15,568 (1918). Leamon. Treating gases containing hydrogen sulphide. (120,554.) July 21.

5814 (1919). Macdonald and Macdonald. Making hydrocarbon gas. (146,560.) July 21.

8708 (1919). Naito. See XXIII.
9756 (1919). Olsson. Treatment of peat. (147,286.) July 28.

9768 (1919). Smith. Furnace retorts. (132,487.) July 28.

10,883 (1919). Beswick and Rambush. Destructive distillation of solid fuels. (147,311.) July 28.

18,162 (1919). Wade (Standard Oil Co.). Manufacture of liquid fuel from petroleum. (147,353.) July 28.

20,157 (1919). Skinningrove Iron Co., and Bury. Recovery of products from gases evolved in the destructive distillation of coal, shale, etc. (147,360.) July 28.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

Asheroft. 21,728. See II.
Benson. 20,646. See II.

Falk. Manufacture of tar. 20,609. July 10. (Ger., 10.12.17.)

Morgan and others. 21,903. See II.
Seidenschmur. Extraction of paraffin from tar etc. 22,143. July 24.

Sudfeldt u. Co. Obtaining salts of sulpho-acids and alkyl-sulphuric acids from acid resins. 20,584. July 10. (Ger., 14.3.19.)

Sudfeldt u. Co. Obtaining sulpho-acids from brown coal-tar oils. 20,585. July 10. (Ger., 14.3.19.)

Tetralin Ges. 20,353, 20,366, and 20,751. See XX.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

Arnot. Azo dyes and process of manufacture. 22,015-6. July 22.

Davies, Thomas, and Scottish Dyes, Ltd. Production of dyestuff intermediates. 22,073. July 23.

Davies, and Scottish Dyes, Ltd. Manufacture of dyestuffs. 22,074. July 23.

Imray (Soc. Chem. Ind. in Basle). Manufacture of azo dyestuffs. 22,090-1, 22,163. July 23 and 24.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Borzykowski. Production of threads. 21,459. July 16. (U.S., 31.4.17.)

Borzykowski. Production of masses or solutions free from air. 21,460. July 16. (U.S., 13.8.17.)

Bouillon and Worms. Filters for producing cellulose threads. 21,633. July 19.

Bouillon and Worms. Treatment of cellulose filaments, threads, or films. 21,634. July 19.

Bouillon and Worms. Apparatus for producing threads from viscose. 21,635. July 19.

Coghlan. Treatment of wool. 21,902. July 21.

Duvinae and Muth. 20,905. See VII.

COMPLETE SPECIFICATIONS ACCEPTED.

8518 (1918). Deuts. Gasglühlicht A.-G. Producing artificial silk. (116,103.) July 28.

14,798 (1919). Portadown Weaving Co., and Greeves. Retting and drying flax and other fibres. (146,702.) July 21.

24,119 (1919). Laan. See XXII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Farbw. vorm. Meister, Lucius, u. Brüning. Printing with dyestuffs. 21,295. July 14. (Ger., 23.8.16.)

Fish, Gass, Hammond, and Jackson and Bro. Bleaching etc. apparatus. 20,390. July 10.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Azote Française Soc. Anon. Absorbing nitrous vapours. 21,583. July 17. (Switz., 18.7.19.)

Casale. Preparing nitrogen or nitrogen and hydrogen mixtures. 20,706. July 10. (Ital., 12.7.19.)

Chambers, Hammond, and Sowden. Treatment of ferrous chloride liquors resulting from cleansing wire etc. 21,322. July 15.

Chem. Fabr. vorm. Goldenberg, Geromont u. Co., and Wolfenstein. Production of formates of alumina. 21,779. July 20.

Davis and Levy. Absorbent for carbon monoxide. 21,786. July 20.

Duvinae and Muth. Preparing compounds of alumina for sizing paper etc. 20,905. July 12. (Ger., 13.5.19.)

Fiévet. Manufacture of zinc oxide. 21,576. July 17. (Fr., 17.7.19.)

Gepp, Webb, and Williams. Purification of zinc-bearing solutions. 21,499. July 16. (Australia, 17.7.19.)

Griggs. Production of hydrogen. 20,746. July 10.

Janbert. Purification of hydrogen or oxygen. 20,566. July 10. (Fr., 12.10.18.)

Naef. Manufacture of sodium compounds. 21,592. July 10.

Ossa. Cuprous iodide processes. 21,991. July 22.

- Patterson. Substitute for table salt. 21,608. July 19.
 Riedel. Manufacture of compounds of nitrogen. 20,601. July 10. (Ger., 20.11.15.)
 Riedel. 20,603, 20,606-8. *See* II.

COMPLETE SPECIFICATIONS ACCEPTED.

- 310 (1918). Bailey, Denny, Norris, and Adams. Production of ammonium sulphate. (146,516.) July 21.
 5594 (1918). Harger. Manufacture of hydrogen. (147,235.) July 28.
 8776 (1919). Davies. Extraction of arsenic and other impurities from sulphuric acid. (146,598.) July 21.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

- Beatty, Reeves, and Tucker. Obtaining viscous charges of glass from a viscous mass thereof. 20,556. July 10.
 Hampshire. Glass furnace. 20,917. July 12.
 Llewellyn, Spence, and Spence and Sons. Drying and calcining siliceous substances. 21,111. July 13.
 Rietz. Manufacture of white clouded glasses, enamels, or glazes. 20,654. July 10. (Ger., 19.7.18.)
 Soc. Anon. Verreries de Fauquez. Glass furnaces. 20,451. July 10. (Belg., 9.2.18.)

IX.—BUILDING MATERIALS.

APPLICATION.

- Collyer. Vulcanised wood. 21,376. July 15.

COMPLETE SPECIFICATION ACCEPTED.

- 18,843 (1918). Atkins and Colquhoun. Manufacture of bricks, tiles, clinker, etc. (147,239.) July 28.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

- Amsler. Annealing furnace. 21,018. July 12.
 Bansen, and Façonseisen Walzwerk Mannstedt u. Co. Hearth smelting or annealing furnace. 21,079. July 12. (Ger., 2.12.18.)
 Bardeen and Harris. Restoring strength of steel. 22,066. July 23.
 Chambers, and others. 21,322. *See* VII.
 Coad. Appliances for extracting tin etc. from metalliferous slimes. 21,742. July 20.
 Diehl. Preparation and smelting of ores etc. 20,885. July 12.
 Head, Wrightson and Co., Ringquist, and Wrightson. Means for separating dust from blast furnace gases. 21,797. July 20.
 Heraeus Ges., and Stahlwerke Lindenberg. Manufacture of iron and steel. 20,543. July 10. (Ger., 15.12.17.)
 Huth Ges. Expelling gases from metal bodies etc. 20,996. July 12. (Ger., 21.2.17.)
 Isabellen Hütte Ges. Alloys. 20,473. July 10. (Ger., 4.2.17.)
 Kosugi. Alloys. 21,387. July 15.
 Krupp A.-G. Producing low-carbon ferrochromium. 20,413. July 10. (Ger., 27.7.16.)
 Leiser. Production of alloys. 20,512. July 10. (Ger., 17.8.14.)
 Metallatom Ges. 20,599. *See* I.
 Phoenix A.-G. Smelting iron ores. 20,747. July 10. (Ger., 10.3.16.)
 Simon. Chloridising roasting of burnt pyrites. 21,033. July 12. (Ger., 2.8.17.)

- Wade (Byers Co.). Manufacture of wrought iron. 21,911 and 22,005. July 21 and 22.

COMPLETE SPECIFICATIONS ACCEPTED.

- 2997 (1919). Waring and Waring. Steel-melting plant. (147,244.) July 28.
 11,499 and 15,475 (1919). August. Rotary muffle furnaces. (146,673.) July 21.
 12,186 (1919). Allison (Naigurai Kagaku Seihin Kabushiki Kwaisha). Composition for preventing corrosion of metal surfaces. (146,676.) July 21.
 15,686 (1919). Odani. Production of metallic powders. (129,631.) July 21.
 15,823 (1919). Jacobs. Electrodeposition of metals. (147,338.) July 28.
 3061 (1920). Clarke and Clarke. Composition for preserving metallic surfaces. (146,796.) July 21.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

- Aymard. Continuously renewable cathode or anode surface. 20,755. July 10.
 Aymard. Electrolytic cell. 20,756. July 10.
 Cherry. Apparatus for electro-chemical treatment of vapours. 21,756. July 20.
 Dynamobürsten-Fabr. Nürnberg-Reichelsdorf, and Muth. Production of electric carbons. 20,688. July 10. (Ger., 20.3.18.)
 Grundhofer. Electric induction furnaces. 20,583. July 10. (Ger., 21.1.18.)
 Nolle. Insulating material. 21,655. July 19. (Holland, 19.7.19.)
 Pilkington. Electric furnaces. 20,865. July 12.
 Sieurin. Manufacture of carbon electrodes. 21,051. July 12.
 Sieurin. Burning carbon electrodes in electric furnaces. 21,676. July 19.

COMPLETE SPECIFICATIONS ACCEPTED.

- 5761 (1919). Thompson. Manufacture of carbon electrodes. (147,247.) July 28.
 7179 (1919). Austin and Adkins. Electric cells or batteries. (147,249.) July 28.
 15,823 (1919). Jacobs. *See* X.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

- Byrnes. Separating aldehyde fatty acids from congeneric substances, and manufacture of soaps from these acids. 21,291. July 14.
 Farley. Soap. 21,794. July 20.
 Farley. Grease solvents. 21,795. July 20.
 Mont. 21,949. *See* XIX.

COMPLETE SPECIFICATIONS ACCEPTED.

- 6733 (1919). Sorensen. *See* XIX.
 8757 (1919). Clayton and Nodder. *See* XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

- Badische Anilin u. Soda-Fabrik. Manufacture of artificial resins. 21,151. July 13.
 Damard Lacquer Co., Fleet, and Potter. Phenol aldehyde condensation products. 22,078. July 23.
 Wade (Barrett Co.). Manufacture of resin. 21,409. July 15.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

- Feldenheimer, Plowman, and Schidrowitz. Manufacture of rubber. 22,087. July 23.
 Greengate and Irwell Rubber Co. Vulcanising rubber goods. 21,122. July 13.

COMPLETE SPECIFICATION ACCEPTED.

19,132 (1919). Peachey. Process for vulcanising dissolved caoutchouc. (146,734.) July 21.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Chem. Fabr. Worms A.-G. Manufacture of tanning agents. 20,720-1. July 10. (Ger., 17 and 24.8.16.)

Howroyd and Turnbull. Manufacture and use of solutions for tanning. 21,775. July 20.

Stoeckly. Hardening surface of patent leather. 20,469 and 21,704. July 10 and 19. (Ger., 13.4.18, 8.8.19.)

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Benbough. Manufacture of manure. 20,557. July 10. (Ger., 6.3.18.)

British Glues and Chemicals, Ltd., and Kernot. Production of fertilisers. 21,006. July 12.

Riedel. Utilising impure gases containing carbonic acid. 20,922. July 12. (Ger., 24.11.17.)

Riedel. Enriching the atmosphere that envelops plants. 20,923. July 12.

COMPLETE SPECIFICATION ACCEPTED.

12,397 (1919). Hawker, and Hawker and Botwood. Manufacture of a preparation for applying to soil or plants as a fungicide, insecticide, and soil steriliser. (146,678.) July 21.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

Martin, Roche, and Tavroges. Manufacture of lactose or milk sugar. 22,019. July 22.

Martin, Roche, and Tavroges. Purification of crude lactose or milk sugar. 22,019. July 22.

COMPLETE SPECIFICATION ACCEPTED.

8741 (1919). Reckitt and Sons, and Hardy. Manufacture of rice starch. (147,255.) July 28.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Bensley. Portable dry yeast. 22,102. July 24.

Plesch. Manufacture of beer. 20,737. July 10. (Ger., 23.8.16.)

Stouffs. Preserving by-products of brewing, distilling, etc. for use as animal foods. 21,673. July 19.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Barrett Co. Manufacture of baking powder. 22,004. July 22. (U.S., 26.4.20.)

Claes. Alimentary substances. 20,449. July 10. (Belg., 15.10.17.)

Martin, Roche and Tavroges. Treatment of whey produced in cheese manufacture. 22,020. July 22.

Maschinenbau A.-G. Balcke. Preventing precipitation from water. 20,542. July 10. (Ger., 25.6.18.)

Mout. Crystallisation of margarine emulsions etc. 21,949. July 22.

Piekee. Manufacture of foodstuffs with calcium chloride as therapeutic mixture. 21,890. July 21.

Stouffs. 21,673. See XVIII.

Strong. Drying fruit, meats, etc. 21,682. July 19.

Thompson. Margarine. 20,779. July 10.

COMPLETE SPECIFICATIONS ACCEPTED.

6733 (1919). Sorensen. Manufacture of margarine. (146,567.) July 21.

8757 (1919). Clayton and Nodder. Manufacture of butter substitutes, edible fats, etc. (147,257.) July 28.

12,397 (1919). Hawker and others. See XVI.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Adam and Legg. Production of organic acids. 21,777. July 20.

Byrnes. 21,291. See XII.

Deuts. Erdöl A.-G. 20,715. See II.

Etabl. Poulenc Frères, and Oechsli. Dichlorides of monoarylsarsines. 20,398. July 10.

Henning. Non-inflammable volatile liquid. 21,371. July 15.

Tetralin Ges. Production of tetrahydro- β -naphthol and tetrahydro- β -thionaphthol. 20,353. July 10. (Ger., 18.5.16.)

Tetralin Ges. Production of ar-1-tetrahydro-naphthalenesulphonic acid. 20,336. July 10. (Ger., 25.2.19.)

Tetralin Ges. Production of nitro compounds of tetrahydronaphthalene etc. 20,751. July 10. (Ger., 17.3.16.)

COMPLETE SPECIFICATION ACCEPTED.

15,774 (1919). Marks (U.S. Industrial Alcohol Co.). Methods of forming esters. (147,337.) July 28.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Baker. Photographie films. 21,219. July 14.

Faulstich. Manufacture of multicolour screens for natural-colour photography. 20,910. July 12. (Ger., 27.3.17.)

Ratier. Photographic processes. 20,419. July 10. (Fr., 16.6.19.)

COMPLETE SPECIFICATION ACCEPTED.

25,208 (1919). Natural Color Pictures Co. Colour photography. (143,180.) July 21.

XXII.—EXPLOSIVES; MATCHES.

APPLICATIONS.

Bash. Matches. 21,425. July 16.

Imray (Soc. Chem. Ind. in Basle). Manufacture of an explosive. 21,293. July 14.

Sprengluft Ges. Blasting-charges. 20,523. July 10. (Ger., 5.1.14.)

COMPLETE SPECIFICATION ACCEPTED.

24,119 (1919). Laan. Stabilisation of nitrocellulose. (147,378.) July 28.

XXIII.—ANALYSIS.

APPLICATIONS.

Arndt. Gas-analysing apparatus. 20,902. July 12. (Ger., 30.11.15.)

Davis. Apparatus for detection and estimation of carbon monoxide. 21,785. July 20.

Davis. 21,786. See VII.

Ubbelohde. Determination of specific weight of gases. 20,579. July 10. (Ger., 7.7.14.)

Union Apparatebau Ges. Testing gas mixtures. 20,586. July 10. (Ger., 24.2.19.)

COMPLETE SPECIFICATION ACCEPTED.

8708 (1919). Naito. Analysing or estimating volatile constituents of coal. (147,254.) July 28.

I.—GENERAL; PLANT; MACHINERY.

PATENTS.

Filter. E. J. Sweetland, Assr. to United Filters Corp. U.S.P. 1,344,215, 26.2.20. Appl., 2.7.13.

AN inclined pipe is interposed between filter leaves contained in a casing, from which a uniform stream of fluid is projected against the face of the leaves and toward the bottom of the casing.—W. H. C.

Filtration membrane; Treating — J. F. Waite. E.P. 141,961, 2.12.19.

IN order to render a colloidal membrane capable of filtering a liquid which is not miscible with the liquid already contained in the membrane, the latter liquid is displaced by a liquid (or liquids used successively) which is miscible with both of the immiscible liquids. For example, a collodion membrane which has been used for filtering an aqueous liquid is treated with 95% alcohol before using it for filtering a coal-tar or petroleum derivative.

—W. H. C.

Filtering apparatus. R. G. y Gonzalez. U.S.P. 1,292,758, 28.1.19. Appl., 12.4.18.

AN outer stationary casing is provided with bearings, which support a hollow shaft carrying a frame composed of discs and rods. A filtering fabric is held in position between the discs, and a flexible fabric of cloth or wire netting is also clamped close to but slightly spaced from the material. The liquid is supplied to the hollow shaft, and thence through openings into the filter body, where it is filtered by centrifugal force. By displacing the filter body along the shaft the fabric becomes folded or collapsed, and the adhering solid matter is detached into a compartment at the bottom of the casing.—W. F. F.

Drying drum with sectorial compartments. J. Janka. G.P. 320,526, 8.9.18.

DRUMS arranged horizontally in a drying chamber and divided into sectorial compartments for containing the material, are filled and emptied at the top, and the speed of rotation of the drums is regulated by the difference in weight between the freshly added and the dried material, so that the material is dried in a single revolution.—L. A. C.

Drying plant. P. Barducci. U.S.P. 1,311,163, 22.6.20. Appl., 22.3.19.

A CASING containing a ventilator is reciprocated between the longitudinal shelves on which the material to be dried is supported within a chamber, so as to produce a draught and a blast of air simultaneously between the supports.—W. H. C.

Dryer; Rotary — W. M. Schwartz, Assr. to The Philadelphia Textile Machinery Co. U.S.P. 1,345,620, 29.6.20. Appl., 22.1.20.

A ROTARY drying cylinder is lined with two series of longitudinal trough-shaped blades, one set having inwardly and the other outwardly projecting edges which overlap.—B. M. V.

Solvents; Apparatus for reclaiming — P. Gerli and O. A. Ross. U.S.P. 1,315,083, 29.6.20. Appl., 4.5.18.

A MATERIAL in the form of a long strip which is to be treated with solvent is passed over rollers in a hermetically sealed chamber and is wound from one roller on to another. The speed of the winding roller is varied in accordance with its diameter, so that all parts of the material are exposed to the solvent for the same time. The solvent is evaporated from the strip into the chamber, and the vapour is exhausted and then condensed.—W. F. F.

Reverberatory furnace for use with liquid fuel. L. C. Strub. G.P. 319,379, 6.10.18.

THE flue in which combustion takes place is arranged longitudinally below the middle of the hearth and opens out into a space behind it, from which the hot gases are drawn to both sides of and over the hearth towards the front, whence they pass through openings at each side into flues which lie on either side of the combustion flue and open into the common stack.—A. R. P.

Heating furnace. (A, B) W. C. Buell, Assr. to Tate-Jones and Co., Inc. U.S.P. 1,311,136-7, 22.6.20. Appl., 21.3.18, 8.4.18. (C) W. C. Buell and J. W. Griswold, Assrs. to H. L. Doherty and Co. U.S.P. 1,311,438, 22.6.20. Appl., 21.4.18.

THE waste gases are led through the side walls of the furnace near the bottom, then upwards. Recuperators are provided surrounding the upward conduits (A, B) or elsewhere (C), and may be simple counter-current jackets (A) or of the multiple-pass type (B), and in (C) mechanical means may be provided for forcing the air to burners and exhausting the waste gases.—B. M. V.

Purification of air or other gases. W. J. Mellersh-Jackson. From B. F. Everitt. E.P. 145,344, 20.11.19.

CLEAN air is drawn sideways or backwards from a stream of dirty air which has been propelled against the convex face of a concavo-convex deflector (or other means of producing a Venturi effect), whereby the speed of the air and dirt is increased to such an extent that the dirt is carried past the opening for clean air.—B. M. V.

Gases; Apparatus for cleaning — G. W. Hewitt and A. Steinbart. U.S.P. 1,344,585, 22.6.20. Appl., 8.9.19.

GAS passes into a vertical cylindrical chamber at the top through a tangential inlet, and the solid impurities are separated centrifugally during its downward passage to a central outlet conduit. The solid matter separates into a number of collecting receptacles at the bottom formed by concentric cylindrical partitions, each receptacle being closed at the bottom and provided with a controllable outlet. The partitions extend upwards to different heights above the lower end of the outlet conduit.

—W. F. F.

Orifice-precipitator. L. Bradley, Assr. to Research Corp. U.S.P. 1,344,330, 22.6.20. Appl., 31.10.18.

A NUMBER of plates are fixed vertically, parallel to one another, within the precipitating chamber. Each plate has an orifice which is offset laterally with respect to the orifice in the adjacent plates and through which a rod attached to the opposite plate projects.—W. H. C.

Cooling and crystallising hot solutions; Apparatus for — Maschinenbau-A.-G. Balcke. G.P. 319,968, 10.8.18.

A SERIES of endless bands circulates through the solution, so that, while the lower part of each band is immersed, the upper part, to which the liquid adheres, or by which it is taken up by means of special apparatus attached, is subjected to a current of cold air.—A. R. P.

Solutions; Making and transporting — F. W. Thorold. U.S.P. 1,344,102, 22.6.20. Appl., 29.5.18.

A SOLVENT is forced under pressure through a measured quantity of the material to be dissolved, and the pressure is utilised to force the solution into a receptacle. The flow of solvent is so adjusted that the whole of the material is dissolved when the receiver is filled to a predetermined level.

—W. H. C.

Mixing and/or agitating machines. Brinjes and Goodwin, Ltd., and H. P. Harris. E.P. 145,230, 23.5.19.

A HEMISPHERICAL pan supported on trunnions is provided with a double stirrer that enters the pan at an angle so that the pan can be tilted for pouring without disturbing the stirring gear. The stirrer consists of two elements revolving in opposite directions; a sleeve shaft rotates a circular hoop which almost scrapes the interior of the pan, and a concentric solid shaft carries stirring blades rotating in the opposite direction to and within the hoop.—B. M. V.

Grinding or pulverising ore or other material; Mills or machines for —. R. Hush. E.P. 145,159, 26.3.19.

A disc grinder is fitted with various mechanical improvements, among which are a ball and socket joint connecting the door to the fixed disc, adjustable bearings for the shaft, and an unequal number of corrugations on each disc.—B. M. V.

Pulveriser mill. J. W. Fuller, Assignor to Fuller-Lehigh Co. U.S.P. 1,345,082, 29.6.20. Appl., 1.6.18.

MATERIAL is ground in a horizontal ball mill and then carried upward by a current of air into a cylindrical settling chamber. Another chamber having the same diameter is arranged above the settling chamber and contains a fan for maintaining the upward draught of air. The pulverised material is finally carried outwards from the superposed chamber to the discharge.—W. F. F.

Deflocculating solid materials and agents therefor. E. G. Acheson. U.S.P. 1,345,305, 29.6.20. Appl., 22.1.20.

A DEFLOCCULATING compound is made by the reaction of hexamethylenetetramine upon the heat-conversion products of starchy materials.—B. M. V.

Deflocculating solid substances [graphite]; Method of —. E. G. Acheson. U.S.P. 1,345,306, 29.6.20. Appl., 1.5.20.

GRAPHITE is mixed to a paste with a deflocculating agent and moisture (up to 32% of the latter) and subjected to attrition.—B. M. V.

Drying goods in layers; Apparatus for —. A. Hofmann. U.S.P. 1,344,893, 29.6.20. Appl., 31.10.18.

SEE E.P. 123,923 of 1918; J., 1919, 275 A.

Refrigerating apparatus; Ammonia-dissolving composition for use in —. C. Delaygue. U.S.P. 1,345,144, 29.6.20. Appl., 16.2.20.

SEE E.P. 140,353 of 1913; J., 1920, 355 A.

Straining or filtering apparatus. F. C. Fulcher. E.P. 145,339, 13.11.19.

Decanting and agitating apparatus. E. S. Pettis. E.P. 145,389, 13.3.20.

Furnaces; Recuperators or regenerators for —. A. B. Chantaine. E.P. 18,472, 8.8.14. Conv., 29.8.13.

Heat-transference between gases, vapours, or liquids; Appliances for effecting —. W. Y. Lewis. E.P. 145,109, 12.3.19.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Hydrocarbon motor fuels. G. Baume, P. Niclardot, P. E. Erckmann, and H. Vigneron. Chim. et Ind., 1920, 3, 761—768.

THE minimum temperature at which a definite homogeneous mixture of hydrocarbon, alcohol, and benzene (or ether) can exist rises very rapidly with

increase in water content of the alcohol. The solubility of polymethylene hydrocarbons in alcohol (95 and 90%) at a given temperature is greater than that of the aliphatic hydrocarbons, with the exception of hexamethylene, which has a high m.p. (+5° C.). This exception is eliminated by the addition of benzene to the solvent. The solubility of an alcohol of a given strength at a definite temperature in a hydrocarbon decreases with the rise in the position of the hydrocarbon in its series, whilst the lighter a hydrocarbon the greater is its solubility in alcohol. At 0° C. heptane and absolute alcohol are miscible in all proportions; 95% alcohol at that temperature dissolves up to 30% and 90% alcohol up to 17% of heptane. All mixtures of benzene and alcohol containing over 50% of the former solidify above 0° C. The volume of benzene to be added to a system of hydrocarbon and alcohol to obtain a homogeneous mixture increases with decrease in the volatility of the hydrocarbon; a mixture of 33% of ordinary petrol with 67% of 95% alcohol required the addition of 15% of benzene at -5° C., whereas when the petrol was replaced by a very light petroleum spirit only 1.5% of benzene was required.—C. A. M.

Thiocyanates; Determination of — in ammoniacal liquor and waste liquor from ammonia stills in the by-product coking industry. J. A. Shaw. J. Ind. Eng. Chem., 1920, 12, 676—677.

To 500 c.c. of the liquor are added 5 g. of ferrous sulphate and 100 c.c. of 10% sodium hydroxide solution; after 18 hrs. the mixture is filtered, the insoluble portion washed with 3% sodium hydroxide solution, the filtrate heated to 60° C., acidified with sulphuric acid, and treated with 10% ferric chloride solution. The precipitate (Prussian blue, sludge, etc.) is filtered off, the filtrate diluted to a definite volume, and an aliquot portion heated to 70° C. with the successive addition of dilute sulphuric acid, an excess of sodium bisulphite, and hot 10% copper sulphate solution. The cuprous thiocyanate is collected, oxidised with bromine in alkaline solution, the solution then acidified with hydrochloric acid, evaporated, the residue dissolved in hot dilute hydrochloric acid, the solution filtered to remove silica, and the sulphuric acid in the filtrate precipitated as barium sulphate. This quantity of sulphuric acid is equivalent to the amount of thiocyanate in the original liquor.—W. P. S.

Petroleum; Origin of —. Formation of petroleum from free fatty acids or soaps. Formation from animal hydrocarbons. A. Grün and T. Wirth. Ber., 1920, 53, 1301—1312.

The experiments of Pictet and Potok (J., 1919, 939 A) have been repeated with very different results. The main products of the distillation of sodium stearate *in vacuo* are unsaturated liquid hydrocarbons and pentatriacontanone, the latter of which is practically completely decomposed when the distillates are fractionated at the ordinary pressure. The recent isolation of hydrocarbons by Mastbaum, Tsujimoto, and Chapman from the liver oils of certain fish suggests another possible origin of petroleum (cf. J.C.S., Sept.).—H. W.

Oil emulsions; Separation and examination of —. K. Glaser. Feuerungstech., 1920, 18, 149—153.

A TYPICAL sample of brine from a Boryslaw crude petroleum emulsion was found to contain 13% of sodium chloride with smaller amounts of calcium, magnesium, and bromine. The separation of emulsions can be effected more or less readily by heating to about 80° C., by standing, or by increasing the difference in specific gravity of the two components, e.g., by adding some water-soluble substance or by diluting the oil with benzene. Heating under a pressure of 2—3 atm. has been employed with satisfactory results. Filtration alone is of little value, but

centrifuging is effective, as also is the Cottrell method of agglomerating the water droplets in a high-tension electrostatic field. On a commercial scale it is customary to allow as much water as possible to separate spontaneously and to pass the oil through preheaters heated by flue gases or hot distillate, whereby the water and light petroleum oil are vaporised. This water contains fatty acids, phenols, and sulphurous acid. Emulsified lubricating oil from a steam engine is best dehydrated by heating, when part of the water separates, and filtering the hot oil through a filtering medium previously wetted with oil. For the estimation of the water content of emulsions the oil may be diluted with petroleum spirit in a graduated tube and the volume of separated water read off directly, or the oil may be heated in a current of dried air and the water trapped in a cooled receiver and finally in a calcium chloride tube. Direct measurement of the water thrown out by centrifuging is sometimes adopted, or the oil may be mixed with xylene and distilled, the water in the distillate being collected and measured. Among chemical methods of estimation the best consists in exposing the wet oil to calcium carbide and measuring the acetylene evolved. A similar method based on the use of metallic sodium does not appear to be very satisfactory.—A. E. D.

Oil shale; Utilisation of —. G. Grube. *Z. angew. Chem.*, 1920, 33, 181—182.

WÜRTEMBERG oil shales yield on distillation 6—7% of oil (or 10—11% if a special process is used), with about 1% of sulphur as a by-product. The shales yield about 30% of gas in the ordinary retorts at 1100°—1300° C., and the residual slag forms a heat-insulating and weather-resisting building material. The carbon dioxide in the gas derived from the calcium carbonate is converted into carbon monoxide by passing the gas over red-hot coke. One kg. of shale yields 0.128 cb. m. of gas of 4200 cals. per cb. m.—C. A. M.

Petroleum spirit; Estimation of aromatic hydrocarbons in —. W. Hess. *Z. angew. Chem.*, 1920, 33, 176.

To avoid loss by evaporation in measuring the petroleum spirit (*cf.* J., 1920, 509 A) a 60 c.c. pipette is employed having a long tip which is allowed to touch the bottom of the flask. Nitric acid (sp. gr. 1.42) intended for use in the nitration acid is freed from nitrous oxides by air-blowing at 40°—45° C. In estimating the un-nitrated hydrocarbons a temperature correction for the volume of the petroleum spirit is necessary if the reading is made immediately after nitration.—W. J. W.

Pyrogenic acetylene condensations. I. I. R. Meyer and K. Taeger. *Ber.*, 1920, 53, 1261—1265.

THE mixture of picrates obtained from the fractions of higher boiling point derived from the acetylene tar described previously (J., 1919, 35 A) is shown to contain fluorene, acenaphthene, anthracene, phenanthrene, pyrene, small quantities of fluoranthene, and a hydrocarbon, $C_{17}H_{14}$, m.p. 186° C., which yields a picrate, m.p. 231° C.—H. W.

Tar from coal. Fischer and Schrader. *See* III.

Sulphur in fuel. Vita. *See* X.

PATENTS.

[*Fuel.*] *Drying the pressed residues obtained from the hydrolysis of starchy mosses; Process for* —. V. H. Frestadius. G.P. 320,375, 12.2.18.

RESIDUAL sugar is removed from the material by fermentation or repeated washing with water so that it may be readily dried for use as fuel.

—L. A. C.

Fuel for explosion motors. Hlasehkowerko Eberhard und Jacob. G.P. 319,893, 10.4.18.

THE fuel consists of a mixture of 80 parts or less of coal-tar hydrocarbons of sp. gr. 0.8—0.9 with 20 parts or more of fatty (acetic) acid esters.—L. A. C.

Peat, crude cellulose, oil seeds, grain, or the like; Process for disintegrating and extracting liquid from moist —. B. Melzer. G.P. 320,856, 29.12.17.

THE material is disintegrated between revolving compressors, and the liquid content is taken up by a layer of absorbent material from which it is subsequently expressed.—L. A. C.

Gas; Process for making —. D. E. Campbell. E.P. 141,868, 29.5.19.

LIQUID hydrocarbon or mineral oil such as a petroleum distillate of about 48° B. (sp. gr. 0.786) is converted into a permanent gas by subjecting a jet to violent concussion against a hard surface. The jet is ejected from a nozzle of about 0.01 sq. in. in area under a pressure of 9 to 10 atm. against a small transverse metal plate placed in a conduit through which a current of air is flowing. The oil is thereby converted into a stable gas and mixed with air, and the mixture is suitable for heating, lighting, or power purposes.—W. F. F.

Producer gas generated within retort setting or retort; Process for enrichment of —. C. Griffin. E.P. 141,989, 15.4.20.

TAR is injected by means of steam into one or more retorts in a setting, and the resulting gas is mixed with the producer gas in the ascension pipe or hydraulic main.—W. F. F.

Furnace retorts. C. H. Smith. E.P. 126,614, 17.4.19. *Conv.*, 9.5.18.

A HORIZONTAL furnace retort for the partial carbonisation of coal is of inverted heart shape in cross section, and is heated by two sets of combustion flues separated by longitudinal flues and controlled independently. The two sets of flues are symmetrical with respect to the separating partition in the longitudinal vertical axis of the retort. Each heating flue is independently supplied with gas, and with air from a recuperator, the gas and air supply valves being regulated from a longitudinal tunnel below the retort bounded on either side by the recuperators. The waste gas from both sets of flues passes through a common collecting flue to the recuperators. The retort is provided with a pair of parallel rotary conveyors, one in each side of the heart-shaped section. The blades of the two conveyors rotate in paths which intersect, and the shafts of the conveyors are adjustable both horizontally and vertically to vary the position of the blades.—W. F. F.

Illuminating gas and tar; Manufacture of —. H. Geyer. G.P. 319,521, 6.5.19. *Addn.* to 317,977.

FINELY-GROUND coke dust is mixed with the coal dust before it is charged into the retort and treated as described in the chief patent (J., 1920, 395 A) to prevent the tar adhering to the balls and to ensure better contact with the gas stream.—L. A. C.

Gas producers. W. Antrobus. E.P. 145,161, 29.3.19.

LOW-GRADE fuel such as wood, peat, Kimmeridge and oil shales, and inferior coal is fed into a storage chamber at the top of the producer, where it is dried and preheated. The fuel then passes downwards through a number of vertical cast-iron tubes of 12 to 24 in. diameter to the lower part of the producer, where it is heated by the hot gas rising from the combustion zone below. The rich gas generated in this zone is drawn off through an

outlet immediately below the vertical tubes. The producer gas generated in the combustion zone passes upwards through small vertical tubes depending from the larger tubes into the combustion zone, and then upwards through these tubes to an outlet at the top of the producer. The central portion of the producer tapers outwards and the lower part tapers inwards and is provided with two constricted sections. The producer is suitable for the production of by-products such as ammonia and tar.—W. F. F.

Gas producers. J. Harger. E.P. 145,136. 22.3.19.

COAL is introduced into the top of a vertical producer which is provided with two side openings opposite to one another near the bottom, in each of which a regenerator is fitted. The regenerators are connected with a heat exchanger in such a way that either may be connected with the tubes or with the space surrounding the tubes. When a mixture of air and steam or carbon dioxide is blown through the heat interchanger and one regenerator into the producer, producer gas is generated and removed through the other regenerator and the recuperator to heat the incoming air. Water-gas may be generated by first blowing air through in one direction to heat the fuel and then blowing steam through in the opposite direction. During the next cycle the air and steam are blown through in the opposite directions. Some coal gas may be extracted at the top, and the distillation is effected without cracking the gas.—W. F. F.

Hydrocarbons; Apparatus for cracking and distilling —. J. W. Coast, jun., Assr. to The Process Co. U.S.P. 1,345,132-4, 29.6.20. Appl., 22.3.17 and 29.9.17.

(A) A LIQUID-PHASE horizontal cracking still is arranged so that the vapours are continuously drawn off through a condenser and the uncondensed gases returned through a compressor to the bottom of the still in order to effect agitation and prevent coke formation at the heating surface. (B) A false bottom is fitted to the still in (A) to serve as a coke trap. The contents of the still are continuously circulated by the injected gases so that they pass through this coke trap and deposit suspended coke. (C) The still is heated by a bath of fusible metal which is also circulated by a pump into the oil and back into the main bath of metal.

—A. E. D.

Wax from petroleum distillates; Apparatus for expressing —. W. C. Wells and F. E. Wells. U.S.P. 1,344,745, 29.6.20. Appl., 12.4.19.

A FILTERING pad comprises a frame of coarse woven wire, a covering of finer woven wire imposed thereon, and a woollen fabric strainer mounted on this covering in such a way as to be adequately supported.—A. E. D.

Mineral oils; Process for converting — into higher boiling products. W. North. G.P. 319,123, 2.3.17.

PARAFFIN oil or the like from which solid paraffin has been removed is heated with finely-divided gas-coke for 1 hr. at 300° C. under a pressure of about 400 atmos. Copper oxide may be added to remove the liberated hydrogen. The product contains 20% of soft paraffin, m.p. 38.5° C.—L. A. C.

Low-boiling hydrocarbons; Process for obtaining — from high-boiling hydrocarbons, tar, or the like. P. Schwarz. G.P. 319,049, 3.10.14.

THE hydrocarbon material is heated under pressure in a still which is completely filled with the material, and is forced by the pressure through an outlet tube at the bottom into a second, larger vessel for distillation.—L. A. C.

Hydrocarbons; Process for obtaining light — from heavy hydrocarbons. F. Bergius, Assr. to The Chemical Foundation, Inc. U.S.P. 1,344,671, 29.6.20. Appl., 12.2.14.

SEE G.P. 304,348 of 1913; J., 1920, 222 A.

Coke ovens; Air valves for —. Valves for coke ovens. G. B. Ellis. From Foundation Oven Corp. E.P. 145,372 and 145,374, 29 and 30.1.20.

Gas-producer plants for vehicles, locomotives, and aircraft or the like. F. J. Wilshire. E.P. 145,201, 22.4.19.

Spent oxide furnace. E.P. 145,366. See VII.

Carbon black. U.S.P. 1,344,060. See XIII.

Extracting montan wax. G.P. 320,543. See XIII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

PATENTS.

Distilling carbonaceous material; Apparatus for —. W. P. Perry. E.P. (A) 145,101 and (B) 145,102, 20.1.19.

(A) PEAT, shale, or the like is fed through a gas-tight valve into one chamber of a producer divided by a vertical partition, and is subjected to a gradually increasing temperature and distilled during its downward passage. The partition extends downwards to a level at which all the carbon dioxide has been converted into monoxide. The hot gas passes upwards into the other chamber, and thence into the fuel chamber through openings inclined downwards to prevent passage of solid material in the opposite direction. The distillation is effected by the hot gases. The gas passes out through openings in the wall of the fuel chamber to a main and thence to a condenser. (B) The apparatus is of similar construction to that described in (A) except that the partition extends the full length of the producer. A fire is maintained in the bottom of the empty chamber to generate gas, which then passes into the producer chamber to distil the material.—W. F. F.

Heat; Method of producing —. C. E. Lucke. U.S.P. 1,344,049, 22.6.20. Appl., 11.9.15.

AN explosive gaseous mixture is projected, with an initial velocity greater than that of flame propagation, in a rapidly expanding free jet. The mixture is burned at the point at which its velocity is reduced to that of flame propagation.—W. F. F.

Mercury arc lamp. W. C. Heraeus G.m.b.H. G.P. 318,836, 31.12.18.

A MERCURY arc lamp has a tungsten anode placed at such a distance from the mercury as to maintain permanency of the arc even at high pressures and with great resistance. In this lamp a five- to tenfold increase in brightness corresponds to a ten- to twenty-fold increase in the ultra-violet radiation in the neighbourhood of 366 μ , and there is smaller consumption of energy and a more permanent length of arc than with carbon arc lamps.

—W. J. W.

Heating furnace. U.S.P. 1,344,436-7. See I.

III.—TAR AND TAR PRODUCTS.

Tar from coal; Determination of the yield of —. F. Fischer and H. Schrader. Z. angew. Chem., 1920, 33, 172-174.

THE apparatus consists of an aluminium retort, the interior walls of which slope towards the base to facilitate removal of residue, and having a close-

fitting lid. On one side of the retort is a nose-shaped extension with central bore, into which is screwed a brass tube through which the tar distils into a glass receiver immersed in water. A thermometer is inserted into a semi-circular expansion of the retort wall. For the estimation 20 g. of coal is heated to 500°–520° C. within 30 mins., the temperature being maintained for a further 15 mins. The distillate is then weighed, after which the water constituent is distilled off with xylol or centrifuged, the residual tar being again weighed. Results obtained with several varieties of coal are tabulated.—W. J. W.

Oxylation of benzene. [Preparation of nitrophenols.] L. Vignon. Bull. Soc. Chim., 1920, 27, 547–550.

To obtain the best yield of nitrophenols by the action of nitric acid and mercuric nitrate on benzene it is advisable to use an excess of benzene; the proportions recommended are 1000 g. of nitric acid (sp. gr. 1.3–1.33), 25 g. of mercuric nitrate, and 500 g. of benzene, the mixture being heated in a water bath for 5–7 hrs. For each 100 g. of benzene utilised in the process 230–250 g. of a mixture of nitrophenols of m.p. 75°–80° C. is obtained; the nitrogen content indicates that it is a mixture of about 52% of dinitrophenol and 48% of picric acid.—W. G.

Phenol and certain of its homologues; Diazometric determination of —. R. M. Chapin. J. Ind. Eng. Chem., 1920, 12, 568–571.

The phenol is titrated with a diazonium salt solution. The latter is prepared by dissolving 14 g. of *p*-nitraniline in 400 c.c. of water and 70 c.c. of concentrated nitric acid, filtering the solution after 18 hrs., and diluting it to 1 litre; 5 mins. before use a portion of this solution is mixed with an equal volume of 1% sodium nitrite solution. It is standardised against *N*/10 β -naphthol solution (cf. Bucherer, J., 1907, 818). For the determination 20 c.c. of approximately *N*/10 phenol solution is mixed with 50 c.c. of 10% neutral sodium acetate solution and 10 c.c. of 30% basic lead acetate solution, and the diazo solution is run in from a burette, with constant stirring; as each 10 c.c. of diazo solution is added a further quantity of 10 c.c. of basic lead acetate solution is introduced. When the end of the titration is near the stirring is interrupted for 20 mins. and two small filtered portions of the mixture are then tested, one with a drop of the diazo solution and the other with a drop of phenol solution. If no colour develops in either within a few seconds, a drop of 25% sodium hydroxide solution is added to each; the distinction is sharp when the end-point is at some distance on either side, but if it is near a coloration may develop in both tests. The limits coarsely defined in this first titration may be narrowed down by subsequent titrations. Trustworthy results are obtained in the case of phenol, cresols, and xlenols, but the figures for thymol are much too high. (Cf. Fox and Gauge, J., 1920, 260 r.)—W. P. S.

Furfuraldehyde and phenols. Beckmann and Dehn. See XIII.

Nitro compounds. Van Duin. See XXIII.

Nitro-groups. Desvergues. See XXIII.

PATENTS.

Tar; Apparatus for the continuous distillation of —. G. Romberg. G.P. 321,293, 5.7.17.

A VESSEL for the continuous distillation of tar is provided with inclined, zig-zag, or spiral-shaped plates attached to the bottom so that the tar is in constant motion during the process. Local overheating of the tar is thus prevented, better frac-

tation is attained, and the residue is readily removed from the apparatus.—L. A. C.

Benzol and its homologues; Process of purifying —. Phoenix A.-G. für Bergbau und Hüttenbetrieb. G.P. 299,073, 14.12.15.

SUFFICIENT alkali or alkaline-earth, solid or in concentrated solution, is added to neutralise the acid in the mixture obtained after treating crude benzol or the like with sulphuric acid, so that after distillation of both layers together a water-soluble residue, suitable for use in keeping machine tools cool, is obtained without production of waste water. The two layers may be distilled separately, in which case a material suitable for use as a substitute for resin is obtained from the lower layer.—L. A. C.

Carbon bisulphide; Extraction and recovery of —. H. G. Colman and E. W. Yeoman. E.P. 145,099, 20.12.18.

CARBON bisulphide may be readily extracted from liquids containing it (e.g., benzol) by agitation with a cold aqueous solution of an alkali or alkaline-earth bisulphide, whereby a salt of perthiocarbonic acid, H_2CS_3 , is formed. The perthiocarbonate may be used as a bactericide or decomposed by acids to produce carbon bisulphide, hydrogen sulphide, and sulphur; the first of these is steamed off and condensed, the second absorbed in hydrated ferric oxide, and the sulphur recovered by filtration and used in the preparation of more bisulphide solution. If iron salts or ferric hydroxide are employed instead of acid a precipitate of ferrous sulphide and sulphur is obtained which on exposure to the air oxidises to ferric oxide and sulphur. Simple distillation of the perthiocarbonate solution gives a distillate of carbon bisulphide and hydrogen sulphide and a residue of polysulphide and hydroxide which can be converted into bisulphide again. The escaping gases which contain carbon bisulphide may be stripped by a preliminary purification with hydrated ferric oxide and lime and subsequent passage through alkali-cellulose, thus forming cellulose xanthate.—A. E. D.

Gas and tar. G.P. 319,524. See II A.

Varnish. G.P. 319,010. See XIII.

Resin solutions. G.P. 319,011. See XIII.

Eliminating carboxyl groups. E.P. 114,897. See XX.

IV.—COLOURING MATTERS AND DYES.

Dyes; Fission of organic — by hydrogenation. R. Meyer. Ber., 1920, 53, 1265–1276.

ALIZARIN Direct Violet R and Alizarin Direct Green G (M. L. and B.) are decomposed by hydriodic acid with the formation of leucoquinizarin and *p*-toluidine-2-sulphonic acid and are thus isomeric with Alizarin Irisol and Alizarin Cyanine Green G (Bayer), which give leucoquinizarin and *p*-toluidine-3-sulphonic acid. The utility of hydriodic acid in effecting the fission of azo-dyes has been investigated; in general, an aqueous or alcoholic solution of the dye is heated with an excess of concentrated hydriodic acid until action appears to be complete. The liberated iodine is removed by sulphurous acid and the hydriodic acid by evaporation with hydrochloric acid. The method has the advantage over other reduction processes that it does not introduce any inorganic salts into the solution. Satisfactory results have been obtained with Methyl Orange, Diphenylamine Orange, Metanil Yellow, Congo Red, and Naphthylene Red.—H. W.

3.6-Tetramethyldiaminoselenopyronine. M. Battagay and G. Hugel. Bull. Soc. Chim., 1920, 27, 557–560.

A MIXTURE of sodium selenite and sulphuric acid

monohydrate is added gradually to sulphuric acid containing 25% of sulphur trioxide, and at the same time tetramethyldiaminodiphenylmethane is added in small portions so that it is always present in slight excess, the temperature not being allowed to rise above 35°. After 1½ hrs. the mixture is poured on to ice, filtered, and zinc chloride solution (sp. gr. 1.6) added to the filtrate. A crystalline precipitate of tetramethyldiaminoselenopyrroline zinc chloride, $C_{12}H_{12}N_2SeCl_2 \cdot ZnCl_2$, is obtained. It dissolves in sulphuric acid to a red solution, which turns blue on dilution, and on the addition of sodium hydroxide a reddish-blue base is precipitated, which is soluble in ether. It dyes tannin-mordanted cotton deep violet-red shades from aqueous solution.—W. G.

Colouring matters of flowers. Kögel. See XXI.

PATENT.

Dyestuffs and their intermediates; Process for purifying organic —. Elektro-Osmose A.-G. (Graf Schwerin Ges.). G.P. 320,149, 31.7.14.

EITHER basic or acid electrolytes, which act respectively as electro-negative and electro-positive colloids, are added to dyes (or their intermediates), whereby the dyes are converted to the colloidal state, and impurities are then removed by an electric current. An example is given of the purification of an anthracene dyestuff of unknown composition, and insoluble in water, acids, and alkalis, by means of sodium silicate solution.—L. A. C.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Silk; Relation between the internal complex metallic salts and the solubility of — in ammoniacal nickel solutions. The biuret reaction (Cu, Ni, Co) of silk and wool. M. Battagay and T. Voltz. Bull. Soc. Chim., 1920, 27, 536—540.

THE colour changes and the ultimate solution of silk when it is immersed in an ammoniacal nickel solution are explained on the basis of the formation of a complex nickel salt with a cyclic structure. The action is thought to be similar to the biuret reaction, which is found to be obtained under suitable conditions with alkaline copper and cobalt solutions, the former giving a violet and the latter a pink tint on silk. Allowing for the sulphur present in wool, similar results are obtained with it or with the products of its partial hydrolysis by acid.—W. G.

PATENTS.

Typhacæ; Process for treating — to obtain fibrous material and a by-product. P. Höring. G.P. 303,933, 2.11.17.

REEDS such as *Typhacæ*, before treatment with liquor to separate the fibrous matter, are boiled exhaustively with water, and, after removal of the undissolved residue, the aqueous solution is evaporated to dryness under reduced pressure. A yellowish brown, hygroscopic product is obtained which contains 20% of ash, chiefly calcium and phosphoric acid, nitrogenous matter, and hexoses and pentoses, and possesses nutrient and therapeutic properties.—L. A. C.

Scouring fibres of vegetable origin; Process for — with the object of accelerating and facilitating subsequent bleaching. E. T. J. Watremez. E.P. 135,516, 21.11.19. Conv., 8.11.18.

THE scouring bath contains soluble alkali-metal compounds obtained by treating a metallic oxide or

preferably a powdered metal with an alkali, an alkali hypochlorite, and a reducing agent, and may be prepared thus: 5 kg. of zinc dust is stirred into a solution of 25 kg. of sodium "hyposulphite" in 50 l. of commercial sodium hypochlorite solution, allowed to stand 12 hrs., 150 l. of caustic soda of 30° B. (sp. gr. 1.262) is added, and the liquor decanted after standing 24 hrs. Vegetable fibres are boiled in this solution (suitably diluted) for 4—6 hrs., 15 kg. of slaked lime is added, the fibres are further boiled for 3—4 hrs., immersed for 2—3 hrs. in a solution of ammonium sulphate (5—7% by volume), rinsed, immersed for 2—4 hrs. in a cold acid bath containing 2% by volume of sulphuric acid of 60° B. (sp. gr. 1.712), and rinsed; they are then ready for bleaching by ordinary methods. The treatment may also be carried out in the cold.—A. J. H.

Washing and cleaning; Process of —. M. Buchner. G.P. 319,933, 24.8.16. Addn. to 312,220 (J., 1919, 714 A).

COLLOIDAL magnesium hydroxide, prepared by treating a solution of a magnesium salt, of which the acid radicle does not form an insoluble calcium salt, e.g., magnesium chloride, with milk of lime at 70°—100° C., is used for washing and cleaning all kinds of materials.—L. A. C.

Wood-pulp liquor; Method of refining crude —. P. Hein, Assr. to The Chemical Foundation. U.S.P. 1,344,691, 29.6.20. Appl., 24.10.16.

CRUDE wood-pulp liquor is refined by mixing it with wood ashes.—A. J. H.

Sulphite waste liquor; Obtaining [resinous] products from —. W. Oehl. G.P. 320,508, 30.11.16.

SILICATES or compounds of heavy metals are added to sulphite waste liquor, whereby the calcium bisulphite is converted into calcium silicate or into an insoluble bisulphite, which is then separated from the resin solution.—L. A. C.

Plastic, gummy material from cellulose esters; Manufacture of —. F. Clouth. G.P. 319,723, 6.12.17.

A PLASTIC, gummy material contains a cellulose ester, such as cellulose acetate and anthracene oil, with or without the admixture of a substance which forms a homogeneous mixture with anthracene oil, such as triphenyl phosphate, sulphanilide, triacetin, or ethyl lactate. Anthracene oil increases the elasticity and waterproof qualities of the product.—L. A. C.

Celluloid, vulcanite or the like; Production of substitutes for —. W. T. Robinson-Bindley, A. W. Weller, and E. Dulcken. E.P. 145,128, 21.3.19. Addn. to E.P. 134,564 (J., 1920, 14 A).

IN the production of synthetic resins from cresol and formaldehyde, the sodium sulphite used as catalyst may be replaced by an inorganic fixed base as sodium or potassium hydroxide, or by an alkali salt as sodium carbonate, acetate, or formate.—A. J. H.

Retting fibrous substances; Process of —. E. L. Rinman. U.S.P. 1,344,719, 29.6.20. Appl., 2.10.18. See E.P. 128,464 of 1918; J., 1919, 678 A.

Ramie, flax, hemp, and like plants; [Mechanical] treatment of — for the production of fibre for spinning. W. P. Govaerts and G. A. M. Dreypondt. E.P. 8559, 4.4.14.

Reclaiming solvents. U.S.P. 1,345,083. See I.

Cellulose. G.P. 320,856. See IIA.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyes fast to chlorine; Method for rendering —. [Printing.] J. Brandt. Sealed note No. 1421. Bull. Soc. Ind. Mulhouse, 1920, 86, 28—30. Report by H. Bourry, *ibid.*, 30—31.

COLOURED effects obtained with printing pastes containing chrome mordant dyes having phenolic or acidic and basic groups are fast to chlorine if tannic acid be present. A blue reserve, fast to chlorine, on Para Red is obtained by the use of a printing paste consisting of 1 kg. of acid starch paste, 200 g. of Modern Violet 40% paste, 400 g. of chromium acetate 11° B. (sp. gr. 1.082), 400 g. of gum tragacanth (66 g. per l.), and 200 g. of a 50% tannic acid solution, with the usual subsequent development. A black reserve is obtained by the use of a paste composed of 1 kg. of starch paste, 50 g. of Brown salt G., 200 g. of Modern Violet, 400 g. of chromium acetate 11° B., and 360 g. of gum tragacanth (60 g. per l.), with the addition of 60 g. of sodium chlorate immediately before use. Bourry reports that the two printing processes are satisfactory, but that the tannin alters the shade of the dyes without much increasing their fastness to chlorine.

—A. J. H.

Aniline Black; Resists and conversions under —. P. W. Pluzanski. Sealed note No. 1468. Bull. Soc. Ind. Mulhouse, 1920, 86, 31—32. Report by M. Battegay. *Ibid.*, 32—33.

Blue or yellow resists are obtained by printing a paste containing caustic soda and 40 g. of stannous chloride per kg. on cloth prepared for Aniline Black with the usual padding liquor, but containing 50 g. of Indanthrene or Flavanthrene per kg., drying, rapidly ageing to develop the black, and then steaming in the absence of air for 6 mins. The alkaline resist paste may be suitably coloured, and if the cloth be first prepared with β -naphthol the resists can be developed with diazo compounds. Indigo may be used in place of Indanthrene if sodium hydrosulphite NF be present in the resist paste. Battegay reports the process to be satisfactory.—A. J. H.

PATENTS.

Bleaching treatment of open fabrics; Apparatus for —. E. W. Hunt. E.P. 145,113, 19.3.19.

AN ordinary bleaching kier contains a rectangular fabric chamber, two opposite sides of which are movable. The fabric enters the kier, is plaited down in open width by a suspended pair of electrically-driven nip rollers, compressed by the two movable sides, and bleached by the usual methods of boiling.—A. J. H.

Dyeing machine. F. L. Bickel. U.S.P. 1,344,788, 29.6.20. Appl., 31.5.19.

A DIAPHRAGM oscillates in the liquid chamber of the machine, whereby the fabric is compressed and periodically removed from the dye liquor.—A. J. H.

Printing cotton fabrics and yarns. The Calico Printers' Assoc., Ltd., W. Rouse, and G. Nelson. E.P. 145,240, 5.6.19.

THE alkaline discharge of alumina, iron, and chrome mordants is satisfactory in the presence of tannin. Cotton, mordanted with the tannates of aluminium, iron, or chromium, is printed with an alkaline mordant such as sodium aluminate or an alkaline lead solution, or a mixture comprising a caustic alkali, a reducing agent, and a vat or sulphur dye, aged or steamed, washed, and dyed by the usual methods.—A. J. H.

Glue substitute. G.P. 318,957. See XV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric acid; Concentration of — [by the Strzoda tube system]. K. Mattenklodt. Chem.-Zeit., 1920, 44, 529 (cf. J., 1918, 688A).

By means of the Strzoda plant, used in conjunction with a Petersen pre-concentration tower, a strength of 97—98% may be reached from initial 50% acid. Working to 93—94% concentration, with an output of 10 tons per 24 hrs., a reduction of 1.34 tons of weak acid treated is effected as compared with other processes, such as the Gaillard, Kessler-Duron, Hartmann and Benker, and Pauling. Loss in the Strzoda process is negligible; about 1% of very weak acid (1° B.; sp. gr. 1.007) is obtained, whereas other plants show about 10% containing an appreciable amount of sulphuric acid which has to be recovered by re-concentration. The increased fuel consumption in the Strzoda plant, 1.8 ton per 10 tons 94% acid, as against 1.2—1.5 ton in other plants, is counterbalanced by the higher yield of concentrated acid, and smaller amount of weak acid obtained. By the employment of an iron-silicon alloy the wear and tear of the plant is reduced to a minimum.—W. J. W.

Ammonia-water; The system —. S. Postma. Rec. Trav. Chim., 1920, 39, 515—536.

THE author has constructed the melting-point, boiling-point, and vapour pressure curves of mixtures of ammonia and water and the sublimation curve of ammonia, and has deduced a formula for the variation of the vapour pressure of ammonia with temperature.—W. G.

Ferrous compounds; Catalytic action of copper salts on the oxidation of — by air. L. Maquenne and E. Demoussy. Comptes rend., 1920, 171, 65—69.

COPPER salts exert a marked catalytic action on the oxidation of ferrous salts by air even at extreme dilutions. It is most noticeable with ferrous salts of weak acids.—W. G.

Tin; Precipitation of — by iron. N. Bouman. Rec. Trav. Chim., 1920, 39, 537—541.

AN explanation of the fact that iron will not precipitate tin from a solution of stannous chloride is given, based on potential measurements. At the same time, it is shown that when reduced iron is boiled with a dilute acid solution of stannous chloride some tin is precipitated.—W. G.

Ammonium hydrogen fluoride; Titration of —. W. S. Chase. J. Ind. Eng. Chem., 1920, 12, 567—568.

TO avoid the interfering action of hydrofluoric acid on ordinary indicators in determining the acidity of ammonium hydrogen fluoride, the solution of the sample is treated with an excess of calcium chloride; an equivalent quantity of hydrochloric acid is liberated, and this is titrated without removal of the precipitated calcium fluoride. Methyl orange is used as the indicator.—W. P. S.

Thiocyanates in ammoniacal liquor etc. Shaw. See IIa.

Pickling solutions. Boyle. See X.

Acetic acid in acetates. Pickett. See XXIII.

PATENTS.

Sulphuric acid; Process and apparatus for removal of arsenic from —. G. E. Clark. E.P. 144,869, 29.5.19.

AN apparatus for treating sulphuric acid with a de-arsenicating gas, such as hydrogen sulphide,

without the use of a steam injector, comprises a tower with suitable baffles, through which the acid gravitates in the form of spray into a sealed container, also provided with baffles which cause the acid to flow in a zig-zag path. The reacting gas is blown by a fan into a holder, from which it is conveyed under pressure through a series of pipes, terminating in nozzles, and entering the container between its baffles. After passing through the acid in this vessel and effecting its de-arsenication, the gas is passed upwards through the tower and thus brings about a preliminary treatment of the descending acid.—W. J. W.

Sulphurous gases; Production of concentrated —. Farbenfabr. vorm. F. Bayer und Co. G.P. 304,262, 24.11.16.

GASES containing a low percentage of sulphur are passed over porous charcoal moving in the opposite direction, and the sulphurous acid absorbed by the charcoal is then recovered by careful heating. By keeping the charcoal in motion during the heat treatment the formation of carbon oxysulphide is prevented, and the sulphur dioxide content of the gases may be raised from 0.3% to 70%.—W. J. W.

Sulphur dioxide; Method of producing —. F. Laist and F. F. Frick. U.S.P. 1,344,905, 29.6.20. Appl., 9.6.19.

A MIXTURE of sulphur dioxide and oxygen is made to react with a substance containing sulphur. —W. J. W.

Oxides of sulphur; Manufacture of — from sulphates, specially calcium sulphate. Badische Anilin- und Soda-Fabrik. G.P. 306,313, 6.11.15.

THE sulphates, which may be mixed with combustible substances, are introduced in a finely-divided condition into oxidising flames, the air admitted being previously heated.—W. J. W.

Gases containing sulphur; Producing — suitable for the manufacture of sulphur and a magnesia sludge from magnesium chloride liquor. Harburger Chem. Werke Schön und Co., and W. Daitz. G.P. 306,441, 15.2.17.

IN the process for decomposing magnesium chloride liquor by means of calcium sulphide, a mixture of calcium sulphide and calcium oxide, prepared by roasting calcium sulphate together with lime or dolomite under the action of reducing fire-gases and steam, is employed instead of calcium sulphide alone. A small quantity of iron oxide may be added to the charge in the roasting process to regulate the action of the gases, provided that a product free from iron is not required.—L. A. C.

Sulphur; Process for obtaining — from calcium sulphate. Badische Anilin- und Soda-Fabrik. G.P. (A) 301,682, 4.11.16; (B) 302,471, 18.11.16; (C) 306, 312, 24.12.16.

(A) THE gases containing sulphur dioxide derived from the decomposition of calcium sulphate are mixed with carbon monoxide, or gases containing carbon monoxide, in presence of red-hot coal or coke. (B) Instead of, or in addition to, carbon monoxide, oxygen or air may be introduced in such excess that the gases, after leaving the combustion zone and on entering the reduction chamber, contain an appreciable proportion of oxygen. (C) When gypsum is employed for the production of the gases, it may be first dehydrated, the air introduced into the process being also completely dried. The yield of sulphur is practically doubled by this means.—W. J. W.

Nitrogen products [cyanides]; Apparatus for the production of —. C. T. Thorssell and H. L. R. Lundén. E.P. 145,299, 14.8.19.

IN the manufacture of cyanide by interaction of

nitrogen with coal and oxygen-containing compounds of alkali or alkaline-earth metals, at a high temperature, and under high pressure, the energy of the gas mixture at the end of the reaction may be utilised both for introducing the nitrogen into the reaction chamber and for other purposes. The gases leaving the reaction chamber at high temperature, and consisting largely of combustible gas (carbon monoxide), are led through a branch pipe on the nitrogen inlet pipe to an expansion engine, which operates a pump for driving the nitrogen under pressure to the reaction chamber. The gases leaving the expansion engine are then conveyed to a combustion engine or gas burner for further utilisation. The expansion engine and pump may be connected to two or more reaction chambers adapted to work alternately so that gas from one is used for compressing nitrogen for supplying another.—W. J. W.

By-products, particularly nitrogen compounds, from blast furnaces; Method of obtaining —. W. Ostwald, Assr. to The Chemical Foundation, Inc. U.S.P. 1,344,770, 29.6.20. Appl., 10.10.16. Renewed 25.11.19.

IN the treatment of ores in blast furnaces a hydrated chloride is added to the charge. The water of hydration, liberated as steam, acting in the ammonia-forming temperature zone, converts the nitrogen of the fuel into ammonium chloride, which is then separated from the blast-furnace gases. —W. J. W.

Aluminium nitride and the like; Production of —. M. Shoeld, Assr. to Armour Fertilizer Works. U.S.P. 1,344,153, 22.6.20. Appl., 10.7.19.

BRIQUETTES of aluminous and carbonaceous material are mixed with practically infusible resistor elements of substantial size which do not enter into the chemical reaction, and which are of greater electrical conductivity than, and prevent fusion of, the briquettes. Aluminium nitride is produced by heating the mixture by means of an electric current in an atmosphere of gas containing nitrogen. —A. R. P.

Aluminium nitride; Furnace for manufacture of —. G. Herman. G.P. 319,046, 23.10.14.

A FURNACE for treatment of bauxite with excess of nitrogen containing methane comprises chambers heated by gas (e.g., natural gas), divided into a number of smaller, vertical reaction chambers, in which the material is treated at a high temperature with reaction gases of high concentration, the process being carried out continuously by increasing the reaction velocity.—W. J. W.

Aluminium silicates; Treatment of natural —. W. Borchers. G.P. 300,684, 14.4.16. Addn. to 300,092 (J., 1920, 64A).

CARBONATES or hydroxides of the alkaline-earths may be substituted for the sulphates referred to in the original patent. During the roasting the silicic acid will combine with the alkaline-earth oxide, leaving the aluminium oxide in the finished product in a finely-divided condition, and readily susceptible to suitable solvents.—W. J. W.

Spent iron oxide; Furnace or kiln for burning —. W. Crowther. E.P. 145,366, 9.1.20.

A NUMBER of superposed burning compartments are arranged one above the other in a setting, each being provided with an arched roof leaving a space between the roof and the floor next above. The burnt gas passes through the roof into the space above and thence through side openings into a common vertical flue. The material on each floor is thus preheated by the gas from the chamber below. The burnt oxide is pushed out of the burning cham-

bers into a receiver at the back, and the light dust is deposited in the outlet flues.—W. F. F.

Sodium sulphite; Manufacture of —. E. O. Barstow, Assr. to The Dow Chemical Co. U.S.P. 1,343,897, 22.6.20. Appl., 25.6.18.

NITRE-CAKE is caused to interact with calcium sulphite, the acid sodium sulphite produced being then treated with milk of lime.—W. J. W.

Potassium; Treatment of minerals containing —. A. Messerschmitt, Assr. to The Chemical Foundation, Inc. U.S.P. 1,341,705, 29.6.20. Appl., 11.12.14.

SILICATES containing potassium are finely ground, mixed with sodium nitrate, and heated to a high temperature, under pressure, in presence of water. —W. J. W.

Potash [from silicates]; Recovery of —. A. C. Spencer. U.S.P. 1,344,830, 29.6.20. Appl., 17.11.15.

AMORPHOUS silicates containing potassium are heated under pressure with a neutral salt of a strong base, in presence of a solvent, strong bases or acids being absent. The salt equivalent should be about 15 times that of the potassium present. —W. J. W.

Sodium silicofluoride; Preparation of — and hydraulic cement. W. Esch. G.P. 319,559, 27.3.19.

SILICIC acid, fluorspar, and magnesium sulphate are roasted together in a rotary furnace, and the silicon tetrafluoride evolved is passed into a solution of sodium chloride. The residual product in the furnace, on account of its content of magnesium oxide and calcium sulphate, may be employed as a hydraulic cement.—W. J. W.

Salts of heavy metals; Manufacture of —. H. Kühl. G.P. 308,396, 23.6.17.

SALTS of heavy metals are prepared by heating a mixture of an oxide, hydroxide, or carbonate of a heavy metal and an ammonium salt of a strong acid; e.g., by heating a mixture of iron oxide and ammonium sulphate in a stream of air, ammonia is liberated and iron sulphate is formed at a little above 100° C.—L. A. C.

Magnesium arsenate; Method of making —. E. O. Barstow, Assr. to The Dow Chemical Co. U.S.P. 1,344,018, 22.6.20. Appl., 16.9.18.

AN aqueous suspension of magnesium hydroxide is mixed with a solution of arsenic acid.—A. R. P.

Lead arsenate; Manufacture of —. O. F. Hedenburg and D. S. Pratt, Assrs. to The Toledo Red Spray Co. U.S.P. 1,344,035, 22.6.20. Appl., 1.7.19.

FINELY-DIVIDED lead oxide is suspended in a liquid medium, free from electrolytes other than those resulting from partial hydrolysis of the oxide, and arsenic acid or oxide is added, whereby lead arsenate is produced in a fine, crystalline form suitable for use as an insecticide.—W. J. W.

Potassium perchlorate; Cyclic process for the manufacture of —. Oberschlesische A.-G. für Fabr. von Lignose. G.P. 298,991, 25.12.15.

A SOLUTION of sodium perchlorate obtained by electrolysis is treated with potassium chlorate, and the solution of sodium chlorate formed, after addition of sodium chloride to replace the small loss of Na ions, is again electrolysed.—L. A. C.

Potassium chlorate; Electrolysis of solutions of — to prepare potassium perchlorate. Oberschlesische Sprengstoff A.-G. G.P. 300,021, 7.12.15.

A SOLUTION of potassium chlorate is electrolysed at

27° C., using nickel cathodes and a current density of about 0.15 amp. per sq. cm. of cathode surface.

—L. A. C.

Bromates; Manufacture of —. C. W. Jones, Assr. to The Dow Chemical Co. U.S.P. 1,343,918, 22.6.20. Appl., 25.6.18.

A SOLUBLE bromate is separated from admixture with other salts by converting it into barium bromate, which is relatively insoluble.—A. R. P.

Hydrogen; Manufacturing of —. The British Oxygen Co., Ltd., S. W. Bray, and I. H. Balfour. E.P. 111,751, 11.2.19.

THE spent gases issuing from a unit in which iron oxide has been reduced by producer gas to metallic iron to be used in the manufacture of hydrogen are treated by known methods, e.g., condensation or absorption, to remove the greater part of the water vapour, carbon dioxide, and sulphur compounds. The purified gas is then used to reduce a further charge of oxide.—A. R. P.

[Oxidising] catalyst, and process of making it. J. C. W. Frazer and C. C. Scalone. U.S.P. 1,345,323, 29.6.20. Appl., 4.2.19.

A HYDRATED metal oxide is precipitated in an extremely finely divided condition, washed free from soluble matter, and compressed into a cake which is dried at a low temperature in a current of gas containing free oxygen.—W. J. W.

Flue dust. U.S.P. 1,345,034. See X.

Lime-potash fertiliser. U.S.P. 1,345,077. See XVI.

VIII.—GLASS; CERAMICS.

"Soda composition" [as substitute for sodium carbonate in glass manufacture]. L. Springer. Sprechsaal, 1920, 53, 310—313.

THE term "soda composition" or "glass composition" is applied to a number of materials used as substitutes for sodium carbonate in glass manufacture. One of these compositions was found to be completely soluble in water and to consist of sodium carbonate 81.0, barium peroxide 2.0, iron oxide 0.013, and water 16.4%. Other compositions consisted of powdered bluish-green soda-lime glass (cullet) with 20% of soda; soda 18, chalk 17, rock salt 60, and fluorspar 5%; and impure rock salt. The presence of calcium sulphate in such compositions is objectionable, as it forms scum on or "seeds" in the glass. Barium carbonate (with or without barium peroxide) is a popular substitute for soda and potash. If all the lime in a batch mixture is replaced by twice its weight of barium carbonate, the glass melts more readily and fines rapidly. If part of the alkali is omitted, the glass still melts readily, but is liable to be stony. If part of the alkali is replaced by baryta a readily fusible and rapidly fining glass is produced. When the lime is only partially replaced by baryta the glass melts well, and the fining is much improved. A saving in potash can only be effected by forming a true barium glaze without lime, or when part of the lime and a small part of the alkali are replaced by barium carbonate. When fluorspar is used as a substitute for alkali, the glass appears to melt readily; this is not due to the direct fluxing action of the spar, but to the volatilisation of some of the sand whereby the silica-content of the mixture is reduced; at the same time the quality of the glass is impaired. Attempts have been made recently to use a specially purified sodium sulphate free from iron as a substitute for soda in crystal glass, but it is doubtful whether so highly purified a sulphate is as cheap as soda, and whether a wholly white crystal glass can be prepared from it. Sodium

silicofluoride and other fluorine compounds are also used as substitutes for soda and as opacifiers, especially in association with kaolin and felspar. Sodium silicofluoride has the same disadvantages as fluorspar and, in addition, it tends to make the glass turbid, especially if alumina is present. Sodium fluoride is also used as a substitute for boron and arsenic compounds, and as a fining accelerator.—A. B. S.

Glass batches containing lime; The choice of —. W. E. S. Turner. J. Amer. Ceram. Soc., 1920, 3, 379–383.

In the manufacture of glass bottles, batch mixtures containing about 300 parts of limestone to each 1000 parts of sand are used for hand-worked glass, but not in works employing automatic machines. In the latter the use of a glass with so high a lime content would be attended with difficulty, and a "lime-flint" glass with a low proportion of lime and a higher proportion of alkali (soda ash and Chile saltpetre) is preferable, as it is more fusible, remains plastic for a longer time and at a lower temperature, has a lower thermal conductivity, and a higher coefficient of expansion, and requires a lower annealing temperature. The durability and resistance of the lime-flint glasses towards air and liquids is reduced as the proportion of alkali is increased and, for this reason, glasses containing less than 7% of lime or less than 166 parts of limestone or more than 412 parts of soda ash per 1000 parts of sand should be avoided.—A. B. S.

Optical glass; Twenty-three types of —. R. J. Montgomery. J. Amer. Ceram. Soc., 1920, 3, 401–410.

On plotting the dispersion and refractive index of various optical glasses as abscissæ and ordinates, respectively, it was found that there are 23 ordinary types of optical glass, composed of:—(a) Fundamental oxides (SiO_2 , K_2O , Na_2O , CaO , PbO , ZnO , B_2O_3) affecting the optical properties, and (b) control chemicals affecting the fusibility of the glasses, i.e., the "proportioning" of carbonates, nitrates, chlorides, and sulphates or the use of arsenic and antimony. Sometimes a fundamental oxide may be used as a control chemical. A diagram shows that very large changes in composition are needed to change the optical properties appreciably, yet if the refractive index and dispersion are to be kept constant only very small variations in composition can be permitted, a variation of 0.1% in the fundamental oxides affecting the third decimal place of the refractive index and the first decimal place of the dispersion. The diagram also shows that there is a natural field in which each oxide has its normal use, though it may be employed in other fields for secondary purposes. Thus, the long, narrow lead zone extends from the "telescope flint" to the densest silica flint, the lead from 10–80% of the batch, and the refractive index from 1.50 to 1.96, and the dispersion from 57.0 to 19.7. To the right of the lead zone barium and zinc are introduced, ranging from dense baryta flint (with 10% barium and 5% zinc) through baryta flint to baryta light flint with 15% barium and 10% zinc. The zinc is highest (15%) in a baryta light flint and dense barium crown; it decreases as the dispersion increases to about 2% in the soft silicate crown and ordinary crown. The soda-lime field includes the ordinary crown, whilst the telescope crown is a soda-lime glass with boric and barium oxides. The "light silicate crown" field overlaps some of the others; it contains 5–10% of boric oxide. The borosilicate crown of high refractive index contains about 10% of boric oxide, and is rather high in alkali, whilst the ordinary borosilicate crown contains 10–15% of boric oxide and may contain 2–4% of barium oxide or alumina. When a new glass is required of any given refractive index and disper-

sion its composition may be roughly determined by extrapolation on the diagram.—A. B. S.

Glass; Measurements on the thermal dilatation of — at high temperatures. C. G. Peters and C. H. Cragoe. J. Opt. Soc. Amer., 1920, 4, 105–144.

THE dilatation of 32 different kinds of glass was measured by means of the Fizeau-Pulfrich interferometer at temperatures between 20° and 650° C. The curves drawn from the figures so obtained show that the glass passes through a critical expansion region in which the rate of expansion increases 2–7 times; this region, which does not exceed 40° C. for any glass examined, was found as low as 400° C. with a barium crown and as high as 575° C. with a lead glass. About 75° C. above the critical region the glass softens and contracts. The results of such dilatation measurements give information as to the temperature for most careful and thorough annealing, the upper limit for rapid annealing, and the region where careful cooling is essential.

Kaolins; Elutriation tests on American —. H. G. Schurecht. J. Amer. Ceram. Soc., 1920, 3, 355–378.

A NUMBER of American kaolins were elutriated in a modified Schulze's apparatus, consisting of a series of four cans, the upper parts of which were cylindrical and the lower parts conical. The diameter of the cans and the speed of liquid passing through them were adjusted to secure the velocities specified by Seger for separating silt and clay substance, and for dividing the latter into three fractions. In carrying out the process the clay is crushed to pass completely through a No. 4 sieve, after which 500 g. is thoroughly blunged with water in a 2-quart churn, sifted, and the slip which has passed through a 200-mesh sieve is deflocculated by the addition of a little caustic soda and admitted to the elutriator drop by drop. The rate of flow must not exceed two-thirds of the maximum velocity until all the slip has been added. To avoid flocculation as the caustic soda is washed out of the clay, a little dilute ammonia is added periodically to the water passing through the elutriator. The elutriation requires three days. The residue in each can is then collected, dried, and weighed, and the size of grains measured under a microscope as a check. Distilled water should be used throughout, as the soluble salts in tap-water cause partial coagulation. The observed diameters of the particles agree well with those calculated by Schöne's formula. When tested in this manner, kaolins from Georgia, South Carolina, and Pennsylvania resembled English china clay in containing a large percentage of particles below 0.010 mm. diameter (Seger's "clay substance"), but those from North Carolina contain only a low percentage of these minute particles. As English ball clays contain 98.55% of very fine grains carried away by a current with velocity of 0.0197 mm. per sec. whereas china clay contains only 40%, it is suggested that the greater plasticity of ball clays is due to the large proportion of these minute grains.—A. B. S.

Terra-cotta bodies; Effect of variation of the size of grog in —. R. L. Clare and D. F. Albery. J. Amer. Ceram. Soc., 1920, 3, 384–387.

VARIATIONS in the character, quality, and size of particles of the grog used in the manufacture of terra-cotta bodies cause corresponding variations in the bodies. Coarse grog (33% on 14-mesh, 12.3% of 20-mesh, 33% on 40-mesh, and 21% through 60-mesh) reduces the drying shrinkage and increases the difficulties in pressing and finishing and in preventing warping. It makes the burned ware weak and the glaze covering poor. Fine grog (1.5%

on 20-mesh, 28.9% on 40-mesh, 20.3% on 60-mesh, 15.1% on 100-mesh, and 31.2% through 100-mesh) facilitates production, but the goods tend to crack when drying and to warp when burned. Grog of medium size (e.g., 11.5% on 14-mesh, 8.2% on 20-mesh, 34.3% on 40-mesh, 13.4% on 60-mesh, 5.7% on 80-mesh, 4.4% on 100-mesh, and 22.5% through 100-mesh) gives the best results. It is, therefore, necessary to select the raw material and to burn and grind it under carefully controlled conditions.

—A. B. S.

PATENTS.

Ruby-glass; Manufacturing — M. Meth. Assr. to The Lancaster Lens Co. U.S.P. 1,311,131, 22.6.20. Appl., 22.11.18.

SUITABLE quantities of antimony sulphate and crude tartar are mixed with the ordinary transparent glass ingredients.—H. S. H.

Furnaces for melting glass and other purposes. J. S., S. H., and A. E. Shaw. E.P. 111,744, 21.12.18.

Cold air is heated by passing through longitudinal flues lying between the bottom of the tank or bath and longitudinal flues conveying the waste gases, in the opposite direction to the air, to the chimney. After further heating by passing through longitudinal flues above the combustion chamber, the air mixes with the gas and enters the combustion chamber.—H. S. H.

Refractory product and method of making the same. C. W. Thomas. U.S.P. 1,311,461, 22.6.20. Appl., 7.8.18.

UNBURNED raw quartzite is mixed with Portland cement, a hardener, and water. After moulding and allowing to set, the mixture is heated sufficiently to calcine the cement and sinter the refractory material.—H. S. H.

Hardened material. U.S.P. 1,343,976—7. See X.

IX.—BUILDING MATERIALS.

Blast-furnace slag; Experiments with — H. Burchartz. Stahl u. Eisen, 1920, 40, 814—819.

THE final results of a series of tests (extending over 5 years) made on blast-furnace slags to ascertain their suitability for use in making concrete, are given and collated with the results obtained after 1, 2, and 3 years. The slags showed no alteration after storage in the open for 5 years, and concrete containing the slags gave no indication of having altered during the period 3 to 5 years. The results in general confirm the conclusions which had been drawn from the shorter period tests, viz., that it is possible to obtain equally good concrete with blast-furnace slags as with gravel.—T. St.

Concrete; The use of boiler-clinker for — Nitzsche. Zement, 1920, 9, 146—148, 152—156, 158—160, 195—197. Chem. Zentr., 1920, 91, IV., 83.

BOILER clinker to be suitable as an aggregate for concrete should have a volume-weight, when loosely filled, of 0.72, sp. gr. 2.15. A concrete made of porous aggregate has a greater volume-weight and strength than one in which a denser aggregate is used, as in the former the grains can be compressed more closely together. Sand-cement concretes are stronger than those devoid of sand. In slag concretes with slag and cement in the ratio of 1:4.5 or 1:7 the volume-weight is between 1.1 and 1.6 and the porosity between 21 and 33%; the absorption is less than that of bricks. It is necessary to control the proportion of sulphates and ferrous oxide to avoid unburnt material.

—A. B. S.

Anhydrite; Use of — for mortar. F. Hartner. Z. angew. Chem., 1920, 33, 175—176.

THE slowness with which anhydrite sets has so far militated against its use for mortar. If, however, it is ground to pass a sieve of 5000 meshes per sq. cm., hardening takes place in the course of a few hours. With a coarser material the same result may be obtained by the addition of 1—3% of lime or Portland cement, the product differing little from flooring plaster. A sample mixed with 20—25% of water to a stiff paste began to set in 2 hrs. and hardened in 4—7 hrs.; the tensile strength of anhydrite mixed with 15% of water was 20—25 kg. per sq. cm. after 21 hrs. The tensile strengths of mixtures of anhydrite and lime in the proportions of 1:1; 1:2; 1:3; with 7, 6, and 5% of water added, after 3, 7, and 28 days, were respectively 15, 20, 50; 12, 14, 25; and 8, 10, 16 kg. per sq. cm. Resistance tests on the same samples gave 200, 300, 500; 180, 240, 320; and 80, 110, 160 kg. per sq. cm. Comparisons of the effect of the fineness of the material on its tensile strength were made on samples to which 1% Portland cement was added after periods of 1, 2, 3, 7, and 28 days. The results were 10, 13, 14, 19, 30; 18, 20.5, 21.5, 25.5, 35.5; 22, 24, 25, 34, 39.5 kg. per sq. cm. Fuel consumption in the case of anhydrite-mortar amounts to about 6—10%, as against 20—30% with lime, 15—30% with gypsum, and 40—60% with Portland cement. In addition no loss results in manufacture, whereas the losses with lime, gypsum, and Portland cement are approximately 45%, 20%, and 35%.—W. J. W.

PATENTS.

Cement; Process for producing — of liquid slags in electric furnaces. K. G. Wennerström. U.S.P. 1,313,948, 22.6.20. Appl., 29.3.20.

LIME or limestone in broken pieces is delivered upon the surface of a bath of liquid slag, and an electric current is passed through the slag bath to melt the lime and cause it to combine with the slag to form the cement.—H. S. H.

Wood; Method of preserving — M. Rüping. U.S.P. 1,315,253, 29.6.20. Appl., 14.12.16.

WOOD is subjected in a closed retort to the action of heated air under pressure, followed by heated creosote oil, injected at a higher pressure. When the walls of the pores and cells have become coated with creosote, the excess of oil is drawn off, the pressure relieved, and the air imprisoned in the wood allowed to escape.—A. B. S.

Lagging material; Manufacture of — “Lipsia” Chem. Fabr. G.P. 318,885, 25.11.17.

BASIC magnesium carbonate, obtained in a flocculent condition by heating ammonium magnesium carbonate with water, is mixed with a fibrous material, either during or after the boiling process. The filtered and dried product is very light and has a very low heat-conductivity.—W. J. W.

Cement. G.P. 319,559. See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron ore; Electric smelting of — with coke. G. Stig. Chem. and Met. Eng., 1920, 23, 29—31.

IN the electrothermic smelting of iron ore the best results, technically as well as economically, are obtained with furnaces capable of utilising a large amount of power. A consideration of the present types of furnaces, both single and with several shafts, points to the Electrometal type having the greatest possibility of development. The largest furnace of this type built is for 7200 kw., but it is

possible to construct furnaces for 12,000 to 15,000 kw. provided electrodes of high quality are available. In working it is shown from actual operations that the most important factors are the rate of melting; the dimensions of the furnace hearth; the shape and height of the furnace; the voltage, current, current density, and arrangement of the leads; the composition of the charge; and the gas circulation. A slow melting rate causes bridging of the charge and the production of cold iron; an improperly dimensioned furnace tends to bring about graphitisation of the coke. A certain relation is necessary between the voltage, current, and current density to obtain uniform heating, and an adequate gas circulation both to promote uniform heating and to increase reduction by the gases. The efficiency of an Electrometal furnace operating with coke for four weeks was about 67% and the average production per day 23 metric tons.

—J. W. D.

Steels; Effect of the time of adding silicon during the preparation of open-hearth —. E. Piwowarsky. *Stahl u. Eisen*, 1920, 40, 773—781.

A SERIES of experiments was carried out on open-hearth steel charges to determine the effect of the time of adding silicon (as ferrosilicon) to the bath on the properties and gas content of the resulting steel, with the following general results. With too early addition of ferrosilicon the high temperature of the bath favours the formation of silicates, consequently the ferrosilicon acts as a deoxidiser and a large proportion of the silicon passes into the slag. The amount of silicon lost in this manner varies from 18 to 35% using 45% ferrosilicon. If, however, the addition is made just previous to finishing, only 3.2—6.7% of the silicon forms deoxidation products and a steel of higher silicon content is obtained. Addition of ferrosilicon as late as possible also reduces the gas content of the steel in its fluid and solid, as well as in its rolled condition.—J. W. D.

Iron; The determination of oxygen in —. II. P. Oberhoffer and O. von Keil. *Stahl u. Eisen*, 1920, 40, 812—814.

THE apparatus and method originally described (see this J., 1919, 256 A) have been simplified. By the modified method each determination requires 52 minutes for completion, but in continuous work results are obtained at intervals of 32 minutes.

—T. St.

Sulphur in pig-iron, steel, ores, slags and fuels; Determination of — by combustion in oxygen.

A. Vita. *Stahl u. Eisen*, 1920, 28, 933—938.

THE gases produced by combustion of pig iron or steel at about 1200° C. in oxygen are passed into a solution of potassium iodide and potassium iodate, the liberated iodine colouring the solution, which is then compared with a standard scale of solutions coloured with potassium bichromate corresponding to steps of 0.01% S. A determination requires 5—10 mins. This process is also applicable to ores, slags, and fuels, the temperature being raised to 1350° C. to ensure complete combustion of the sulphur compounds, but instead of colorimetric comparison the liberated iodine is titrated with N/10 or N/4 sodium thiosulphate. In cases where the sulphur is sublimed on heating, a platinum spiral is introduced which ensures the formation of sulphur trioxide.—T. H. Bu.

High-speed steel; Analysis of —. H. O. Ward. *Chem. and Met. Eng.*, 1920, 23, 28.

A METHOD of analysing high-speed steel is described in which the original feature is the solution of the tungsten and the determination of the manganese, chromium, and vanadium, using a single sample and without filtering during the operation. The

steel is dissolved in a solution of sulphuric acid and silver nitrate, and when solution is complete a nitric-phosphoric acid solution is added and the liquid boiled until the tungsten disappears. The chromium, vanadium, and manganese are next oxidised in order with ammonium persulphate and the manganese titrated with standard sodium arsenite solution. The silver nitrate is then precipitated with hydrochloric acid, excess of ammonium persulphate boiled off, and the chromium titrated with standard ferrous ammonium sulphate, standard potassium permanganate, and standard sodium arsenite solutions. The vanadium is determined immediately by adding standard ferrous ammonium sulphate, destroying the excess with ammonium persulphate, running in standard potassium permanganate solution, and titrating back the excess with standard sodium arsenite solution as before.—J. W. D.

Steel; Determination of zirconium [and titanium] in —. G. E. F. Lundell and H. B. Knowles. *J. Ind. Eng. Chem.*, 1920, 12, 562—567.

FIVE g. of the steel is dissolved in hydrochloric acid with the addition of a small quantity of nitric acid, the silica is separated, the acid solution evaporated to a syrup, 40 c.c. of hydrochloric acid (sp. gr. 1.1) added, and the greater part of the iron and molybdenum is extracted with ether. The acid solution is heated to expel dissolved ether (matter remaining after treating the silica with hydrofluoric acid and dissolved by fusion with pyrosulphate is added at this stage), and an excess of 20% sodium hydroxide solution is added. The precipitate formed is collected, dissolved in hydrochloric acid, and re-precipitated with sodium hydroxide. The precipitate is once more dissolved in hydrochloric acid, the solution diluted to 250 c.c., partially neutralised with ammonia, 2 g. of tartaric acid is added, and the whole treated with hydrogen sulphide until all iron has been reduced. A slight excess of ammonia is then added, and the precipitate formed collected, and washed with dilute ammonium sulphide-ammonium chloride solution. The filtrate is neutralised with sulphuric acid, diluted, heated to expel hydrogen sulphide, filtered, and the filtrate, measuring 400 c.c. and containing 10% of sulphuric acid, is cooled to 0° C., and treated with 6% "cupferron" solution. The precipitate is collected after 10 mins., washed with cold 10% hydrochloric acid, ignited, and weighed. This ignited residue consists of zirconium and titanium oxides; it is fused with potassium pyrosulphate, the melt dissolved in 50 c.c. of 10% (by vol.) sulphuric acid, and the titanium determined colorimetrically or volumetrically. The weight of titanium oxide found is deducted from that of the mixed oxides to obtain the quantity of zirconium oxide. The combined filtrates from the sodium hydroxide precipitates obtained as above may be used for the determination of the aluminium present, and directions are given for this determination when the steel also contains chromium, uranium, and vanadium.—W. P. S.

Oxygen in cyanide solutions [; Determination of —]. E. M. Hamilton. *Eng. and Min. J.*, 1920, 110, 116.

WHITE'S method for the estimation of oxygen in cyanide solutions (J., 1918, 547 A) depends on the degree of coloration imparted to a cyanide solution by the addition of either methyl orange, acid, and potassium chromate, or a solution of pyrogallol and caustic soda oxidised to saturation and then diluted to the shade required. By the first of these methods it is difficult to obtain a match, while in the second the colour produced fades in daylight, thus preventing its use as a standard for more than a brief period. A perfect colour match is obtained by the use of a solution of caramel; the

ting appears to be permanent as far as could be observed in a period of three to four weeks, a slight fungoid growth produced after ten or twelve days being prevented by the addition of formaldehyde. Determinations by this modified method can be made to 1 mg. of oxygen per litre.—J. W. D.

Pickling solutions; Indicators used for determining free acid in —. C. L. Boyle. *J. Ind. Eng. Chem.*, 1920, 12, 571–572.

The presence of a large quantity of ferrous sulphate in pickling solution interferes with the observation of the end-point of the titration of the acidity when ordinary indicators are used, but sodium or potassium thiocyanate solution may be used for the purpose. Ten c.c. of the pickling solution is treated with a few drops of the thiocyanate solution and titrated with standardised alkali solution until the red colour of the ferric thiocyanate disappears. The pickling solution should not be diluted before titration.—W. P. S.

Zinc and copper; The constituents formed by reciprocal penetration of — at a temperature at which these two metals and all their alloys are in the solid state. H. Weiss. *Comptes rend.*, 1920, 171, 108–111.

CONTACT was obtained either by rapid cooling, after immersion of copper in zinc melted under zinc chloride, or by driving a cylinder of zinc into a hole made in a cylinder of copper. When these metals were heated under such close contact at 400° C. for 2 days the constituents detected were pure zinc or its solid solution η ; the constituent ϵ , thickness 0.2 mm.; the γ constituent, thickness about 1 mm.; a very thin zone of the β constituent, and copper or its solid solution α . If the time of heating was increased to 10 days the thickness of the zones ϵ and γ increased without appreciable alteration of the zone β .—W. G.

Blast-furnace slags. Burchartz. See IX.

PATENTS.

Iron; Manufacture of grey —. G. J. Stock. E.P. 143,596, 19.2.19.

WHITE iron is first produced and desulphurised in a smelting furnace at 1200°–1300° C., then transferred to an electrical or other refining furnace and heated in the presence of a desulphurising slag and sufficient carbon and silicon to 1500° C.—J. W. D.

Steel; Process of manufacturing —. R. H. Irons, Assr. to Central Iron and Steel Co. U.S.P. 1,345,192, 29.6.20. Appl., 11.8.19.

IS the "scrap and carbon" process of manufacturing steel a material containing manganese is supplied to a charge of scrap steel and carbonaceous material, in a basic open-hearth furnace, in such quantity as to provide residual manganese throughout the refining operation.—J. W. D.

Iron; Hardening — by means of a mixture containing titanium. R. Eichler. G.P. 319,971, 10.6.17.

IRON hardened by a mixture containing titanous acid or other titanium compound, potassium carbonate, carbon, and leather charcoal or other compound rich in nitrogen does not lose its hardness when made red hot.—L. A. C.

Steel, iron, and similar articles; Treatment of —. H. C. Barnes, Assr. to The Cleveland Metal Products Co. U.S.P. 1,344,372, 22.6.20. Appl., 6.9.16.

THE articles are treated with a solution of a phosphorus compound capable of depositing with or forming a protective coating in the presence of an agent evolving nascent hydrogen in the solution. —J. W. D.

Rust-preventing process for needles, knitting pins, hat pins, and other articles. The Redditch Electro-Plating Co., Ltd., and J. Guise. E.P. 145,116, 19.9.19.

THE articles are first immersed in a solution of caustic potash in water to which bismuth and plum-bago have been added, then electro-plated with a suitable metal (e.g. nickel), and finally immersed and agitated in a solution of water, soap, soda, and lime before drying.—J. W. D.

Electric welding of iron, steel, or alloys thereof; Means [flux] to be employed in —. A. Hyde. E.P. 141,842, 1.5.19.

THE flux (e.g. asbestos and sodium silicate) described in E.P. 128,463 (J., 1919, 610 A) applied to wires etc. for welding is not vitrified but heated only to 700°–800° C., so that only the sodium silicate becomes fused, in which condition the flux adheres well to the metal, but when completely fused under the electric arc is easily detached when required.—B. M. V.

Metallic oxides; Reducing —. A. E. Bourcoud. U.S.P. 1,341,977, 29.6.20. Appl., 17.6.18.

THE oxides are brought into contact at high pressure with a reducing gas formed by mixing a non-combustible gas with atomised fuel at a high temperature.—J. W. D.

Metallurgical process. Process for treating liquids with gases. W. E. Greenawalt. U.S.P. (A) 1,344,030 and (B) 1,344,031, 22.6.20. Appl., 11.8.17 and 5.1.17.

(A) A gas is confined over a liquid, which is mixed with the gas (e.g. by spraying) and at the same time electrolysed, so that additional gas is supplied to make up for that absorbed by combination with the constituents of the liquid. In (B) the gas is sulphur dioxide and the liquid dilute sulphuric acid.

—B. M. V.

Furnace; Electrically heated — [for metals, ores, etc.]. Morgan Crucible Co., Ltd., and C. W. Speirs. E.P. 144,802, 17.3.19.

AN electrical furnace for melting or heating metals or ores comprises a boat-shaped trough with spout, having end extensions on which may be clamped suitably-shaped terminals for the supply of single-phase current, these terminals being hollow to allow cooling water to circulate through them. The trough container is surrounded by a metal casing, extending above its top, to form a receptacle for gradually charging bulky scrap into the trough, the space between the trough and casing being filled with refractory insulating material. Flues are arranged for the removal of fumes during the operations of heating or pouring. The apparatus admits of the use of heavier charges of metal than heretofore, and, by avoiding the necessity for very thick walls for the melting vessel, enables current of higher voltage and lower amperage to be employed. (cf. J., 1913, 1019; 1919, 686 A.)

—W. J. W.

Roasting-furnace; Gas-heated —. Donnersmarckhütte, Oberschlesische Eisen- und Kohlenwerke A.-G. G.P. 319,716, 20.7.18. Addn. to 310,283 (J., 1919, 641 A).

MECHANICAL rabbles are arranged in the zone of the furnace which is at the critical temperature of the combined water, so as to move the ore over the less steeply inclined plates (cf. J., 1920, 69 A, 457 A).

—A. R. P.

Metals; Process of extracting —. C. J. A. Dalziel. U.S.P. 1,344,681, 29.6.20. Appl., 17.1.20.

FINELY divided metalliferous ores are agitated in a leaching solution at atmospheric temperature by

a blast of air of sufficient intensity to form a readily flowing thin pulp, and the pulp, still agitated by the air blast to prevent sedimentation, is passed through a series of electrolytic cells.—J. W. D.

Flue-dust; recovery of solubles from —. R. B. Stringfield, Assr. to International Precipitation Co. U.S.P. 1,345,034, 29.6.20. Appl., 7.12.17.

FLUE-DUST containing calcium, potassium, and sulphate radicles is treated with water to produce a solution containing calcium sulphate and potassium sulphate and an insoluble double sulphate of calcium and potassium. The concentrated solution is then separated from the insoluble salt, which is further treated with water to extract potassium sulphate.—J. W. D.

Hardening material for use as substitutes for diamonds, etc. A. J. Liebmann and C. A. Laise, Assrs. to Independent Lamp and Wire Co. U.S.P. (A) 1,343,976 and (B) 1,343,977, 22.6.20. Appl., 5.5.17 and 23.11.18.

(A) HARD materials suitable for use as wire drawing dies or as substitutes for jewels are produced by pressing substantially pure refractory metal powder into suitable shapes, and hardening these at a white heat between suitable electrodes embedded in a powdered mixture of carbon and a rare-earth oxide. (B) A mixture of refractory metal and powdered carbon is pressed into shapes, baked, and sintered at a white heat.—J. W. D.

Hard lead; Substitute for — for acid-resisting ware. W. Stockmeyer and H. Hanemann. G.P. 305,611. Addn. to 301,721 (J., 1920, 161 A).

A LEAD-SODIUM alloy is employed containing 0.5—1% Na.—L. A. C.

Aluminium solder. J. Segura. U.S.P. 1,344,566, 22.6.20. Appl., 18.5.20.

A SOLDER consisting of Zn 49, Sn 40, Pb 10, white metal 1%, the composition of the white metal being Zn 80—90, Sb 5—15, Cu 3—10%.—J. W. D.

Case-hardening. S. Whyte. U.S.P. 1,345,050, 29.6.20. Appl., 10.10.17.

SEE E.P. 125,666 of 1916; J., 1919, 421 A.

Iron and steel; Manufacture of —. S. Whyte. U.S.P. 1,345,051, 29.6.20. Appl., 24.7.17. Renewed, 1.5.20.

SEE E.P. 124,816 of 1916; J., 1919, 371 A.

Smelting-furnace and the like. J. Gaunt and D. Brookfield. U.S.P. 1,344,878, 29.6.20. Appl., 26.12.17.

SEE E.P. 114,684 of 1917; J., 1918, 308 A.

Metals; Process for increasing the hardness and tenacity of — and the case of their working with cutting tools. A. G. Bloxam, From Soc. Anon. des Usines Giulini. E.P. 145,129, 21.3.19.

SEE U.S.P. 1,326,775 of 1919; J., 1920, 161 A. The treatment of aluminium or its alloys may be effected at 160°—200° C.

Classifying and concentrating apparatus for minerals and the like. W. W. Richardson. E.P. 145,297, 11.8.19.

Furnaces for the heat treatment of metallic or other bodies. J. S. Atkinson, and Stein and Atkinson, Ltd. E.P. 145,284, 17.7.19.

Plates or sheets; Production of — by electro-deposition. S. O. Cowper-Coles. E.P. 145,250, 12.6.19.

Nitrogen compounds. U.S.P. 1,344,770. See VII.

XI.—ELECTRO-CHEMISTRY.

PATENTS.

Furnace for roasting electrodes and pulverulent materials in crucibles or moulds. F. K. Meiser. G.P. 319,481, 10.2.18.

A NUMBER of openings are provided in the lower parts of the furnace, through which the powder flows by gravity when the furnace is being emptied. The openings may be closed when desired.

—J. S. G. T.

Baking artificial carbon [electrodes]; Process for —. F. K. Meiser. G.P. 320,247, 19.6.17.

THE electrodes are surrounded by an adhesive, coherent mass of incombustible material which has no deleterious effect upon the electrodes in subsequent use, and are baked in unlined crucibles or moulds, or may be subjected to direct heat of the furnace without the use of such crucibles etc. For the production of anodes used in the manufacture of aluminium, alumina may be so employed, and iron oxide in the case of electrodes to be used in the manufacture of carbide.—J. S. G. T.

Supports for the active material employed in lead accumulators; Hard lead alloy for use as —. Accumulatoren-Fabr. A.-G. G.P. 320,096, 28.6.17.

LOSS by burning during the melting process is reduced by the addition of 0.1—1% of zinc to alloys of lead with 3—10% of cadmium.—J. S. G. T.

Depolariser for galvanic cells. B. Harte. G.P. 319,222, 25.5.16.

By incorporating finely-divided mercurous chloride with a manganese dioxide-graphite mixture, to the extent of about 1% of the graphite, the fall of potential in battery cells is considerably retarded.

—W. J. W.

Potassium perchlorate. G.P. 298,991 and 300,021. See VII.

Metallurgical process. U.S.P. 1,344,030—1. See X.

Extracting metals. U.S.P. 1,344,681. See X.

Electric furnace. E.P. 144,802. See X.

XII.—FATS; OILS; WAXES.

Origin of petroleum. Grün and Wirth. See IIa.

PATENTS.

Metacholesterol; Preparation of —. J. Lifschütz. G.P. (A) 318,900, 17.2.17, (B) 318,901, 2.3.17.

(A) CHOLESTEROL or substances containing cholesterol are treated with mild oxidising agents until the formation of oxidation products of cholesterol is just detectable by the acetic-sulphuric acid reaction. Metacholesterol, m.p. 140°—141° C., crystallises in glittering leaflets from alcohol, in which it is considerably more soluble than is cholesterol. It gives no colour reaction with the acetic-sulphuric acid reagent, nor with ferric chloride. The alcoholic solution gives after addition of water and evaporation of the alcohol an opalescent colloidal solution which can retain in suspension large quantities of fats or higher alcohols. Fats or oils or vaseline melted with 2% of metacholesterol can take up over 500% of water. (B) Metacholesterol is prepared from animal substances containing cholesterol by treating them with solvents in the usual way, so that the cholesterol is first thrown out as a solid, and then, on concentrating the filtrate, it separates

out, leaving mainly oxysterol in the solution. Or the material containing metacholesterol, *e.g.*, ox brain or blood fat, is treated with volatile solvents and water, or with water alone, the liquid is dialysed or filtered, and the dissolved substance subjected to the above fractional separation by means of solvents in order to obtain a more concentrated preparation of metacholesterol.—G. F. M.

Oil seeds. *ti.* P. 320,856. See IIa.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Ink in writing; Determination of the age of —.
C. A. Mitchell. Analyst, 1920, 45, 217–256.

THE evolution of writing inks, artificial ageing of inks, microscopical differences in old and recent inks, colour of old and new inks in writing, methods of recording the colour, influence of blotting on the colour, sequence of strokes as proof of prior writing, copying methods, chemical tests for age, and chemical changes in the drying of ink are dealt with, and each of these points is shown to be of value as supplying possibly important information regarding the age of writing. The most useful chemical reagent is a 5% oxalic acid solution; a drop is applied to the writing and the effect noticed under a lens. In old blue-black ink writing the Aniline Blue, if still present, is prevented from diffusing by the insoluble tannate which has formed. Many instances of the value of one or another of the tests are given.—W. P. S.

Furfuraldehyde; Action of — on phenols [artificial resins]. E. Beckmann and E. Dehn. Sitzungsber. Preuss. Akad. Wiss., 1918, 1201–1221.

THE prospect of large quantities of furfuraldehyde becoming industrially available induced an inquiry as to how far it possesses the technically valuable properties of formaldehyde. The condensation products from furfuraldehyde and phenols are black in colour but otherwise have all the essential characteristics of bakelites. Thus, a mixture of phenol, furfuraldehyde, and 39% hydrochloric acid (most suitably in the proportions 5:5:1–2) becomes warm and gradually solidifies, contracting away from glass or porcelain, but not from metal surfaces, and after 24 hours is sufficiently hard to be only faintly scratched by gypsum, still retaining a certain elasticity. It is the complete analogue of Bakelite C in its insolubility in acids or alkalis, even at the boiling point, or in organic solvents. Its fracture is similar to that of vulcanite, and it can be worked on the lathe, any tendency to attack the metal being easily removed by warming, which renders it somewhat harder and more brittle. Of the 24 other compounds of which details are given, crude cresol, guaiacol, carvacrol, and β -naphthol gave the best results, the proportions employed in each case being 10:10:1. Alcoholic hydrochloric acid can also be used to promote reaction, any excess being removed at a temperature not exceeding 60° C.; and the products have the advantage of complete homogeneity, without the variations in hardness observed when aqueous acid is employed. By the addition to the original mixture of certain substances, of which the most efficient were “laneps,” coal tar, kieselguhr, sawdust, cellulose, and asbestos, the product was much improved in plasticity and not otherwise impaired. The influence of hydrobromic and hydriodic acids, or the chlorides of weak bases, *e.g.*, zinc, aluminium, and cuprous chlorides, was similar to that of hydrochloric acid, and solid products were also obtained by the aid of sulphuric and phosphoric acids. Organic acids do not induce condensa-

tion. Ammonium chloride and hydroxylamine hydrochloride gave liquid and solid products respectively, whilst that from aniline was partly liquid and partly solid unless subjected to prolonged heating. In presence of basic reagents, *e.g.*, sodium hydroxide or carbonate, or ammonia, condensation did not proceed beyond the formation of black or brownish black plastic masses, *m.p.* ca. 150° C., completely soluble in organic solvents, from which they separated as a varnish. The preliminary stages of condensation also occurred at high temperatures in the absence of catalysts. The liquid condensation products, which may be prepared *in situ*, can be used to render paper, or textiles, waterproof and electrical insulators. The solid products adhere firmly to metallic surfaces, but they are only imperfect insulators, although aniline hydrochloride under certain conditions furnishes a product satisfactory in this respect. The material also proved more durable than leather for footwear. Unsatisfactory results were obtained by the use of mixtures of furfuraldehyde and formaldehyde or benzaldehyde, or of benzaldehyde or salicylaldehyde alone, in conjunction with phenols. Furfuraldehyde has not the same hardening and destructive effect on micro-organisms or albuminoid substances as formaldehyde, and is therefore of little value as a steriliser, or for hardening photographic plates or for tanning purposes.—J. K.

Paint pigments. Gardner. See XVI.

Turpentine. Simonsen. See XX.

PATENTS.

Carbon black; Apparatus for producing —. F. E. Paterson. U.S.P. 1,314,060, 22.6.20. Appl., 21.11.19.

NATURAL gas is burnt in a series of burners, the flames from which impinge on outwardly and upwardly branching plates supported over the burners. The carbon black deposited on the plates is detached by moving them against scrapers, which discharge it into a hopper with inclined sides and a trough at the lower end, through which a conveyor passes.—A. R. P.

Soluble constituents of materials [e.g., resin, montan wax]; Process for extracting —. H. Bollmann. G.P. 320,543, 10.8.18. Addn. to 303,846.

THE process described in the chief patent (J., 1920, 459 A) is suitable for extracting other materials, *e.g.*, resins from vegetable matter or montan wax from lignite.—L. A. C.

Resin solutions; Manufacture of clear —. Deutsch-Luxemburgische Bergwerks- und Hütten A.-G., and S. Hilpert. G.P. 319,011, 12.5.16.

RESIN obtained by heating with water, without carbonising, the waste sulphuric acid from benzol hydrocarbon purification, is extracted with benzene hydrocarbons. The solution is separated from the dilute sulphuric acid, and may be added direct to resin varnishes, *e.g.*, coumarone varnish, or the solvent may be removed, in which case the product is obtained as a bright yellow powder.—L. A. C.

Varnish; Manufacture of a quick-drying — from tar products. Deutsch-Luxemburgische Bergwerks- und Hütten A.-G., and S. Hilpert. G.P. 319,010, 28.4.16.

A VARNISH contains coumarone resin, the organic matter separated without charring from sulphuric acid used in the purification of benzol hydrocarbons, and a drying fatty oil, with or without a solvent.—L. A. C.

Sheet material for use in making printing plates and matrices. J. S. Stokes. E.P. 145,123 and 145,124, 20.3.19.

MATRICES for the production of printing plates con-

sist of a fibrous backing faced with a non-fibrous material as metallic foil or a phenolic condensation product (from phenol and formaldehyde) hardened by heat. The backing is impregnated just short of saturation with a phenol condensation product capable of becoming hard and set under heat and pressure. It is capable of absorbing moisture, thereby becoming flexible.—A. J. H.

Celluloid or vulcanite substitutes. E.P. 145,128. See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; Permeability of — to gases. J. D. Edwards and S. F. Pickering. Chem. and Met. Eng., 1920, 23, 17—21, 71—75.

THE experimental procedure was that of the U.S. Bureau of Standards; pieces of balloon fabric were exposed at 25° C., on one side to air at a pressure of 760 mm. of mercury and on the other to the gas under examination at an excess pressure of 30 mm. of water; the amount of transfused gas was usually estimated by the interferometer. A decrease in permeability is observed with the ageing of the rubber film and with an increased degree of vulcanisation, the effect in the former case possibly being due to the lower permeability of resinous oxidation products. With different pressures the permeability is proportional to the partial pressure of the gas, and it is possible to correct all results to a standard partial pressure of 760 mm. of mercury. The resistance of a rubber film to the passage of a gas increases with its thickness, and the specific permeability of rubber can be calculated; the greater resistance of a given weight of rubber when spread on cloth is to be attributed to the greater effective thickness of the rubber layer. Using the Shakespear catharometer it is found that the actual time required for penetration is very small. There is no obvious simple relation between the permeability and the temperature for any gas, but the relative increase with rise of temperature is approximately the same in all cases. The specific permeability of vulcanised rubber, representing the volume of gas at a partial pressure of 760 mm., which would pass across a centimetre cube in one minute at 25° C., is found to approximate to 20.4×10^{-6} for hydrogen (cf. Kayser, Wied. Ann., 1891, 43, 544). Taking hydrogen as a standard and making comparisons with it at the same partial pressure, the following values are obtained for the relative permeability of the respective gases and vapours:—Oxygen, 0.45; nitrogen, 0.16; air, 0.23; carbon dioxide, 2.91; helium, 0.65; ammonia, 8.0; methyl chloride, 18.5; ethyl chloride, approx. 200; water vapour, approx. 50. For water the value is almost twice as great if liquid water is in contact with the rubber.

—D. F. T.

Rubber; Direct method for the determination of rubber hydrocarbon in raw and vulcanised —. W. K. Lewis and W. H. McAdams. J. Ind. Eng. Chem., 1920, 12, 673—676.

UNVULCANISED rubber is extracted overnight with acetone, dried, dissolved in carbon tetrachloride, and the solution filtered. An aliquot portion of this solution, containing about 0.2 g. of rubber hydrocarbon, is treated with an excess (130%) of bromine dissolved in carbon tetrachloride and, after the mixture has stood for 3 hrs. in the dark, the excess of bromine is determined iodometrically. Ten c.c. of 5% potassium iodate solution is then added and the iodine liberated due to the hydrogen bromide formed by substitution is titrated. Twice the number of c.c. of thiosulphate solution used in the second titration is deducted from the number used in the first, and the result is subtracted from the volume of thiosulphate required for the titra-

tion of the bromine solution without rubber. The equivalent of rubber hydrocarbon ($C_{10}H_{16}$)_x, is 34. In the case of vulcanised rubber, 2 g. of the sample is extracted with acetone for 6 hrs., dried, extracted under a reflux condenser with 100 c.c. of tetrachloroethane for 4 hrs., the mixture cooled, diluted to 250 c.c. with carbon tetrachloride, and a portion of this solution treated as described. It is shown that the so-called "combined sulphur" is actually combined with the double bonds of the rubber hydrocarbon.—W. P. S.

PATENTS.

Rubber and rubber-like materials; Processes for reclaiming — from fabrics coated therewith. E.C. R. Marks. From Acushnet Process Co. E.P. 145,254, 16.6.19.

SEE U.S.P. 1,321,201 of 1919; J., 1920, 35 A.

Vulcanite substitutes. E.P. 145,128. See V.

XV.—LEATHER; BONE; HORN; GLUE.

PATENTS.

Pelts; Removing lime from and tanning —. Chem. Fabr. Rosenberg und Co. G.P. 320,301, 9.11.18.

THE pelts are treated with a solution, diluted with water, of condensed lime-free neutral or acid sulphite-cellulose waste lye in glycerin still-residues. —L. A. C.

Glue; Manufacture of a substitute for —. Farbenfabr. vorm. F. Bayer und Co. G.P. 318,957, 8.7.13.

SOLUBLE starch produced by the action of acids on starch is heated with formaldehyde in the presence of water. A colourless solution equal in adhesive power to a solution of animal glue of the same concentration is obtained; on drying the solution a colourless residue is formed which first swells and then goes gradually into solution when added to water. The product may be used as a substitute for animal glue, or as a size or thickener in printing textiles.—L. A. C.

XVI.—SOILS; FERTILISERS.

Plant growth and pigmentation; Effect of coloured light upon —. Precipitation of paint pigments under coloured light. H. A. Gardner. U.S. Paint Manufs.' Assoc., Circ. 98, 5 pp.

PRELIMINARY experiments indicate that the colour of the light under which plants are grown has some influence on the plant growth and chlorophyll formation. A purple-blue tone seemed to be most injurious. There is some indication that pigments precipitated under differently coloured lights may exhibit slight differences in tone.—W. G.

Soils; Acidity and acidimetry of —. III. Comparison of methods for determining lime requirements of soils with hydrogen electrode. IV. Proposed method for determination of lime requirements of soils. H. G. Knight. J. Ind. Eng. Chem., 1920, 12, 559—562.

THE vacuum method (heating the soil with finely divided calcium carbonate under reduced pressure) and the hydrogen electrode method are the most trustworthy for determining the lime requirements of soils, but in the latter method the soil must be shaken with lime for more than 3 hrs. For measuring the reduction of acidity in limed soils the Hopkins (cf. Hopkins and Pettit, J. 1920, 461 A) and the hydrogen electrode methods yield the most concordant results. A method proposed for deter-

mining the power of soil to decompose calcium carbonate consists in boiling 10 g. of the soil and an excess of calcium carbonate with 25 c.c. of neutral sodium or potassium chloride solution for 10 mins. and measuring the volume of the liberated carbon dioxide.—W. P. S.

Soil extracts; Solid phases obtained by the evaporation of certain — M. S. Anderson and W. H. Fry. *J. Ind. Eng. Chem.*, 1920, 12, 663—669.

THE salts obtained by the evaporation of aqueous soil extracts have a complex character, and resemble those produced by the natural or artificial evaporation of sea water. No soil, however, furnishes all the salts occurring in natural deposits of saline substances, since the latter represent crystallisation from a composite soil extract. Complex salts begin to separate from a water extract at a point not far from the concentration of air-dry soil, and hence the roots of growing plants may be in contact with solutions of these salts. It is thus probable that the complex salts may be present in the soils only in solution in soil moisture and never in the solid phase.—W. P. S.

Potassium of greensand; Effect of manure-sulphur composts upon the availability of the — A. G. McCall and A. M. Smith. *J. Agric. Res.*, 1920, 19, 239—256.

THE results obtained with two greensands containing 5.88% and 1.42% K respectively indicate that the composting of greensand with sulphur and manure may prove to be a practical and efficient method for obtaining available potassium from its comparatively insoluble minerals. The composts containing the largest proportion of manure developed the highest degree of acidity, oxidised the greatest amount of sulphur, and produced the largest amount of water-soluble potassium. In such composts the total amount of potassium recovered in the water extracts after twenty-three weeks varied from 9.1% to 41.3% of the total amount initially present. The addition of small amounts of ferrous sulphate or aluminium sulphate did not stimulate sulphofication. Calcium carbonate, though acting as a stimulant in the early stages, did not increase the soluble potassium obtained.—W. G.

Nitrate-nitrogen and nitrates in fertilisers; Determination of — H. C. Moore. *J. Ind. Eng. Chem.*, 1920, 12, 669—673.

TO determine total nitrogen in fertilisers containing nitrates, 0.8—1.7 g. of the sample is shaken for 15 mins. with 35 c.c. of sulphuric acid containing 1 g. of salicylic acid, 5 g. of sodium thiosulphate is then added and the mixture heated until frothing ceases. After the addition of 5 g. of potassium or sodium sulphate and 0.5 g. of mercury, the mixture is heated until a clear solution is obtained, and the heating is then continued for a further 90 mins. Sodium sulphide is added in the subsequent distillation of the ammonia formed.—W. P. S.

Field heterogeneity; Practical universality of — as a factor influencing plot yields. J. A. Harris. *J. Agric. Res.*, 1920, 19, 279—314.

PATENTS.

Fertilisers; Manufacture of — containing phosphoric acid and alkali nitrates. E. Reinau. G.P. 301,703, 17.2.16. Addn. to G.P. 299,001 and 299,007 (J., 1920, 449 A).

THE mother liquors, wash waters, or distillation residues obtained by the treatment described in the original patents are allowed to interact with tertiary phosphorite, the product being then dried if desired (cf. J., 1920, 461 A).—W. J. W.

Lime-polash fertiliser; Process of producing — from polash-bearing minerals and lime. R. Do Luce. U.S.P. 1,315,077, 29.6.20. Appl., 9.4.20.

A MINERAL containing potassium is mixed with more than its own weight of powdered calcium oxide, and the mixture is passed into a pressure tank by means of superheated steam.—J. H. J.

Crude calcium cyanamide; Continuous manufacture of — A. Lang. G.P. 319,798, 21.11.17.

NITROGEN, previously heated to the reaction temperature, is passed into a nozzle containing finely divided calcium carbide, and the mixture is then blown under pressure into a reaction chamber.—W. J. W.

XVII.—SUGARS; STARCHES; GUMS.

Two-masseuite system of boiling used in Hawaii. R. C. Pitcairn. *Facts about Sugar*, 1920, 10, 230—231.

A SYSTEM of boiling has been adopted in Hawaii according to which two masseuites are produced in order finally to obtain an unrefined sugar polarising about 96°, and exhausted molasses. During the boiling of the first or high-grade, the purity of the syrup coming from the evaporators is lowered to about 72°—73° by the addition of unexhausted molasses, and on centrifuging this masseuite a sugar having the polarisation stated and a molasses having a purity of 48°—50° are obtained. The second or low-grade masseuite is made up with syrup, molasses of 48°—50° purity, and some re-melted low-grade sugar, these being added in proportions to give a purity of about 50°. A temperature of 140° F. (60° C.) is used in boiling this low-grade pan, the contents of which are cooled slowly to 130° F. (54° C.) before discharging. On centrifuging the resulting masseuite, a molasses having a purity about 30° (which is exhausted) and a low-grade sugar are obtained, the latter being either re-melted for the low-grade pan, or used for "seed" in boiling the first masseuite.—J. P. O.

Chemical control figures for the 1919 Java sugar crop. H. C. Prinsen Geerligs. *Intern. Sugar J.*, 1920, 22, 318—325.

DURING 1919 the cane harvested was 38.10 tons per acre, and the sugar extracted was 10.06%, which values are smaller than any recorded since 1915, owing chiefly to the dry weather prevailing and to the lack of fertilisers. Chemical control figures for 1919 and 1918 were: sucrose in the cane, 12.38, 13.63; fibre in the cane, 13.01, 12.99; sucrose in the bagasse, 4.01, 4.34; water in the bagasse, 47.01, 47.22; mill extraction, 92.0, 92.1; sucrose in the press-cake, 3.7, 4.36; and calculated available sugar, 10.60, 12.17%. On 100 parts of sucrose originally present in the cane, the sucrose lost during manufacture was due to that remaining in the bagasse, 7.99; that left in the press-cake, 0.49; that unextracted from the molasses, 7.27; and that unaccounted for, 2.59, a total of 18.34%. Other values for 1919 and 1918 respectively are: purity of the raw juice, 83.7, 86.5; and the purity of the final molasses, 32.1, 33.1. From figures for a number of different factories it appears that there is no relationship between the purity of the juice and that of the final molasses, nor does the method of clarification (defecation, sulphitation, or carbonatation) seem to exert any marked influence on the desaccharification. This is substantiated further by the values for the sugar actually obtained per 100 of the calculated available amount, which averaged 97.4, but was 97.6 for the defecation process, 97.1 for sulphitation, and 97.4 for carbonatation. Of the total output of sugar in Java in 1919 the plantation white was 51.8, refining assortments (11—20 D.S.) 46.4%, and the rest

"sack" and molasses sugars. About 96,500 tons of solidified molasses (which is chiefly exported to India for alcohol production) was also made during the period under review.—J. P. O.

Reducing sugars; Determination of — by the volumetric permanganate method. T. van der Linden. *Archief Suikerind. Nederl.-Indië*, 1917, 25, 325—327. *Intern. Sugar J.*, 1920, 22, 413.

In order to avoid any error that may arise owing to atmospheric oxidation in Bertrand's method (J., 1907, 60; 1919, 381 A), it is recommended that the cuprous oxide should be dissolved in an unacidified solution of the ferric salt used, the sulphuric acid being added immediately before proceeding to the titration with standard permanganate solution.

—J. P. O.

Reducing sugars; Iodometric determination of —. H. van Nouhuys. *Archief Suikerind. Nederl.-Indië*, 1920, 28, 591—595. *Intern. Sugar J.*, 1920, 22, 411—412.

SCHOORL'S modification of the iodometric method should be operated as follows, the quantity of sulphuric acid stated in the description given by Geerlings and Tervooren in their "Handboek" (3rd ed., p. 95-96) being too small: 30 g. of the product is clarified with 25 c.c. of normal lead acetate (30%), made up to 500 c.c., and filtered; 100 c.c. of the filtrate is treated in a 500 c.c. flask with sufficient sodium phosphate solution to precipitate all the lead and calcium present, after which the liquid is made up to 500 c.c. and filtered. Following the procedure described by Herzfeld, 50 c.c. of this filtrate and 50 c.c. of Fehling's solution are boiled together; at the end of the 2 min. specified the reaction is checked by the addition of 100 c.c. of cold water (recently boiled), and the whole cooled; 7.5 g. of potassium iodide and 25 c.c. of 25% sulphuric acid are successively added, the iodine liberated being titrated with N/10 thiosulphate. Compared with Bertrand's permanganate method (J., 1907, 60; 1919, 381 A), this iodometric modification gives slightly lower results. It is recommended as a rapid procedure, convenient for routine practice.

—J. P. O.

Sugar; Delicacy of the α -naphthol test for the presence of traces of —. M. Kauffman. *Archief Suikerind. Nederl.-Indië*, 1920, 28, 515—519. *Intern. Sugar J.*, 1920, 22, 412.

In testing for the presence of traces of sugar in the waters condensed from evaporators and pans by means of α -naphthol, the procedure involving underlaying with sulphuric acid is generally followed in the sugar factory. A more delicate and certain indication is stated to be obtained by the Skärblom modification, in which one or two drops only of the water under examination are mixed with about 0.2 c.c. of a 4% alcoholic solution of α -naphthol and about 1 c.c. of concentrated sulphuric acid, and the whole shaken together. Whereas this modification was found to be capable of detecting 0.0005 g. of sugar per litre, the method generally followed failed to show the presence of 0.015 g. It is said the delicacy of the underlaying method depends much upon the manner in which the sulphuric acid is added to the sugar solution. Thus, if the acid is added very slowly so as to minimise mixing, the ring may be hardly visible; whereas in the case of a solution of lower sugar content, if the test-tube be shaken a little after the addition of the acid a distinct colour will be obtained.—J. P. O.

Tint photometer for sugars. Meade and Harris. See XXIII.

PATENT.

Drying residues of starchy mosses. G. P. 320,375. See IIA.

XVIII.—FERMENTATION INDUSTRIES.

Yeast fermentation; Sulphurous acid and —. E. Hägglund. *Biochem. Zeits.*, 1920, 103, 299—306.

SULPHUROUS acid at 0.007-N concentration completely inhibited the fermentation of 1 g. of yeast in 25 c.c. of solution; 0.005-N acid did not completely inhibit the fermentation. The toxic effect of sulphurous acid on yeast fermentation is attributed to the undissociated acid. Sodium sulphite showed an inhibitive action as compared with potassium sulphate. A 0.2-N potassium sulphate solution increased the fermentation by about 25%.

—S. S. Z.

Diastatic power; Volumetric method for the determination of —. J. T. Flohil. *J. Ind. Eng. Chem.*, 1920, 12, 677.

ONE c.c. of a 5% malt solution (prepared according to Lintner's method) is added to 100 c.c. of 2% soluble starch solution, the mixture is kept at 20° C. for 1 hr., 10 c.c. of N/10 sodium hydroxide solution added, and the whole diluted to 200 c.c.; 25 c.c. of this solution, 20 c.c. of Fehling's solution, and 5 c.c. of water are boiled together for 2 mins., cooled to 25° C., 3 g. of potassium iodide and 10 c.c. of 25% sulphuric acid are added, and the liberated iodine is titrated with thiosulphate solution. The amount of cupric salt reduced by the hydrolysed starch is calculated into Lintner degrees.

—W. P. S.

Alcohol-testing device. S. Waldbott. *J. Ind. Eng. Chem.*, 1920, 12, 690.

FIFTY c.c. of the alcoholic liquid is boiled in a flask provided with a column filled with glass beads and a short bent tube at the top of the column, and the alcohol vapour escaping from the open end of the bent tube is ignited. The duration of the flame indicates the alcoholic strength of the liquid.

—W. P. S.

PATENTS.

Distillers' slop; Process of treating —. H. P. Bassett. U.S.P. (A) 1,344,850 and (B) 1,344,851, 29.6.20. Appl., 12.7.19.

(A) DISTILLERS' slop is treated with the hydroxide of an alkaline-earth metal, then acidified, and the volatile fatty acids precipitated by the addition of a salt of a metal which forms insoluble basic salts with them. The liquid is then neutralised and the glycerin separated. (B) Formic and acetic acids are removed before precipitation.—J. H. J.

XIXA.—FOODS.

Frozen fish in storage for short and long periods; Chemical study of —. E. D. Clark and L. H. Almy. *J. Ind. Eng. Chem.*, 1920, 12, 656—663.

FROZEN fish remained palatable and wholesome for 13—16 months when kept at 15° F. (−9° C.) provided that they were glazed or coated with ice; paper wrappings afforded but little protection against loss of moisture. After 2 years' storage all the fish became unpalatable. Generally there was a decrease in the water-soluble and coagulable nitrogen, whilst the amino-acid nitrogen also increased slightly; in some cases the proteose nitrogen decreased. Ammonia and amino nitrogen increased considerably in certain species of fish stored in a glazed condition. The acid value of the extracted fat increased whilst the iodine value decreased.

—W. P. S.

Copra meal; Biochemical study of —. F. O. S. y Alvarez. *Philippine J. Sci.*, 1920, 16, 181—189.

A SAMPLE of Manila copra meal from which most of the oil had been extracted contained: Moisture,

11.3; oil, 12.2; crude proteins, 20.1; ash, 5.5; crude fibre, 13.2; and carbohydrates 37.0%. Compared with cotton seed meal it was richer in amides (15.35% of the N) and cystine (3.93%), poorer in arginine, lysine, and non-amino N, whilst the humin N (2.90%) and mono-amino N (25.04%) were about the same. Alkali-soluble protein is the principal protein in copra meal; 47.55% of the protein nitrogen is soluble in hot water. In admixture with green leaves copra meal is an excellent feeding stuff.—C. A. M.

Jelly. C. H. Campbell. J. Ind. Eng. Chem., 1920, 12, 558—559.

In making apple jelly the pectin content of the apple juice should be 1—1.25% and the acidity (expressed as H_2SO_4) 0.3%; 5—6 lb. of sugar is required per gall. of juice. Excess of sugar produces a soft jelly, whilst an insufficient amount gives a tough product. The pectin content is determined by adding 10 c.c. of the filtered juice drop by drop to 180 c.c. of alcohol, collecting the precipitate at once, and drying it for 2 hrs. at 70° C. *in vacuo*. After being weighed, the precipitate is incinerated and allowance made for the amount of ash present. (Cf. Barker, J., 1918, 213 T.)—W. P. S.

Rice. Hofmeister. See XX.

PATENTS.

Atomising and drying milk or other liquids; Apparatus for —. Chemische Verwertungs-Ges. m.b.H. G.P. 320,200, 17.6.19. Addn. to G.P. 311,471 (J., 1919, 694 A).

The milk inlet pipe is surrounded by another pipe, conveying dry air, which is discharged horizontally so as to mix intimately with the atomised material. A second pipe conveying dry air is fixed below the atomising device.—J. S. G. T.

Oranges; Art of treating —. M. Henius, Assr. to F. Mendelsohn. U.S.P. 1,343,915, 22.6.20. Appl., 15.5.18.

Juice expressed from oranges is treated with sulphurous acid and then pasteurised at 50°—60° C.; about 80 parts of actual SO_2 per million parts of juice should be added.—W. P. S.

XIXb.—WATER PURIFICATION; SANITATION.

Water; Aluminium hydroxide in mechanically filtered —. N. J. Howard and F. Hannan. Canadian Eng., 13.5.20.

In Toronto the water supply is mechanically filtered by the drifting sand method, and alumina is found in the filtered water. Laboratory experiments showed that water treated with one or two grains of alum per gallon, allowed to settle, and filtered through a rapid sand filter always gave a reaction for alumina with the haematoxylin test, even if an equivalent amount of calcium hydroxide had been added. When the treated water was filtered through a layer of six fine filter papers alumina was removed at first, but later passed through. This is attributed to the adsorption of the positively charged hydrosol of alumina by the negatively charged filter paper until the latter charge was neutralised. Treated water which had stood for four months still gave the alumina reaction, but it appeared as if the hydroxide had been converted into an aluminate. In Toronto water the residual alumina has been found to have no corrosive action and to be harmless from a health point of view.—J. H. J.

Carbon dioxide; Determination of solvent — in water. H. Noll. Z. angew. Chem., 1920, 33, 182—184.

In Tillmans and Heublein's method of determining the carbon dioxide having solvent action upon

calcium carbonate (cf. J., 1920, 556 A), any carbon dioxide present in the water as iron bicarbonate is titrated with the free carbon dioxide. Hence 1.1 mg. of CO_2 must be deducted for each mg. of Fe_2O_3 present. In the case of waters free from iron the results for solvent dioxide are most accurate when magnesium and sodium bicarbonates are present in addition to calcium bicarbonate. Iron bicarbonate appears to reduce this solvent action the most, and then calcium, magnesium, and sodium bicarbonates in the order given.—C. A. M.

PATENTS

Sludge and scum; Separation of — from sewage and other liquids. S. H. Adams. E.P. 145,291, 1.8.19.

A SEDIMENTATION tank is provided with an apron roof, sloping longitudinally from near one wall to near the bottom of the opposite wall, and suspended on pivots. The floor of the tank slopes in the opposite direction to the roof, and at its lower side terminates in a sludge channel. Sewage enters at one end of the tank and flows over the roof towards the exit pipe at the opposite end. Solids settling out of the sewage slide down the roof into the lower portion of the tank to the sludge channel. Scum rising from the floor of the tank is directed by the under side of the roof into a scum space at the surface between the top of the roof and the side wall of the tank, from which it is removed over a weir into a scum channel. When used for the activated sludge process the tank is made preferably of circular form, the air inlets being so placed as to impart a revolving motion to the liquid. In this case the exit pipe is placed axially and receives the liquid through perforations along its length. (Reference is directed to E.P. 11,073 of 1903, 10,981 of 1907, and 111,401.)—J. H. J.

Oxygen compounds of sulphur for use as disinfectants, fungicides, and insecticides; Apparatus for manufacture of —. T. A. Clayton. E.P. 141,661, 1.10.19. Conv., 11.4.19.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Digitalis leaf extracts; Stability of —. The infusion. II. C. A. Pomeroy and F. W. Heyl. Amer. J. Pharm., 1920, 92, 391—401. (Cf. J., 1919, 654 A.)

The aqueous infusion of digitalis is slightly less active than the tincture of equal concentration. The infusions deteriorate on keeping at the rate of about 20% in 3—4 days in warm weather, and in 7 days in cold weather (10° C.), and should accordingly be discarded at the expiration of these periods. The addition of alcohol does not increase the stability.—G. F. M.

Rice polishings; Alkaloid constituents of —. F. Hofmeister. Biochem. Zeits., 1920, 103, 218—225.

AN alkaloid substance, termed "oridine," was extracted from rice, which showed antineuritic activity and which on further purification became inactive. It is isomeric with betaine and valine, and is supposed to be related to the pyridine and piperidine groups. (Cf. J.C.S., i., 586.)—S. S. Z.

Turpentine from Pinus longifolia Roxb.; Constituents of Indian —. J. L. Simonsen. Chem. Soc. Trans., 1920, 117, 570—578.

By repeated fractional distillation of Indian turpentine three main fractions were isolated. The lowest fraction, b.p. 155°—167° C. at 706 mm., consisted mainly of α - and β -pinene. The second fraction eventually gave an oil boiling constantly at 165°—169° C. at 705 mm. consisting of a new

bicyclic terpene, sp. gr. 0.8586 at 30° C., $n_D^{20} = 1.469$, $\alpha_D = +7.69^\circ$, to which the name *d*-carene is given. It is readily oxidised by air, gives a crystalline nitrosate, and is converted by hydrogen chloride in dry ether into a mixture of the hydrochlorides of *d*-sylvestrene and dipentene. From the products of its oxidation by Baeyer and Ipatiev's method (Ber., 1896, 29, 2796) *trans*-caronic acid was isolated, and it is considered that the terpene contains a carane ring, and that it is either Δ^3 - or Δ^4 -carene. From the highest fraction of the distillation of the original turpentine the sesquiterpene *d*-longifolene was isolated as a colourless, somewhat viscous oil, b.p. 254°–256° C. at 706 mm., sp. gr. 0.9284 at 30° C., $n_D^{20} = 1.495$, $\alpha_D = +42.73^\circ$. It yields no solid nitrosate, but gives a beautifully crystalline hydrochloride, hydrobromide, and hydriodide.—G. F. M.

Phytin in plant extracts; Determination of —. A. Rippel. Biochem. Zeits., 1920, 103, 163–172.

PHYTIN can be precipitated more or less quantitatively in the presence of inorganic phosphates by copper acetate in acetic acid solution. Favourable results can only be obtained with definite concentrations of copper acetate and acetic acid. An extract from barley yielded 63.3% of the phytin P_2O_5 by precipitation with copper acetate in 1% acetic acid. The same method accounted for 92.8% of the phytin in standard solutions of that substance. The phytin in an acetic acid extract from peas could not be precipitated by copper acetate at all.—S. S. Z.

Anemonine; Synthesis and constitution of —. Y. Asahina and A. Fujita. Yakugakuzasshi, 1920, 455. J. Pharm. Chim., 1920, 22, 22–26.

RANUNCULUS oil, on reduction with sodium amalgam and acetic acid in methyl alcoholic solution, gave only an insignificant amount of dihydroanemonine, the main product being the lactone of angelic acid, which was identified by conversion into the semicarbazone of levulinic acid. The chief constituent of the oil is therefore not anemonine but a substance which is converted into angelic lactone by reduction, and into anemonine by the spontaneous condensation of two molecules, and to which the name protoanemonine is given. Formulæ are suggested for protoanemonine, and for anemonine, which were confirmed by the synthesis of anemonine from β -bromolevulinic acid, and also from the dibromide of angelic acid lactone.—G. F. M.

Furfuraldehyde and phenols. Beckmann and Dehn. See XIII.

Alcohol. Waldbott. See XVIII.

Mercury. François. See XXIII.

PATENTS.

Lecithin; Process of recovering — from animal and vegetable substances. C. Baumann and J. Grossfeld. E.P. 144,895, 11.7.19.

LECITHIN is recovered from animal and vegetable substances, such as egg yolk or fish roe, or from raw commercial lecithin before the separation of the fat, cholesterol, etc., by dissolving the substances in an organic solvent such as methyl alcohol, and treating the solution with hydrogen in presence of a hydrogenating catalyst, for example, palladium or nickel, until little or no lecithin can be extracted from the mixture by cold ethyl acetate. By this means the lecithin is saturated first, and the hydrogenation is stopped at this stage before the fats are attacked. The residue after the evaporation of the solvent, is dissolved in hot ethyl acetate, and on cooling the solution the lecithin is almost quantitatively precipitated in a technically pure condition.—G. F. M.

Acetaldehyde from acetylene as primary material; Catalyst for the manufacture of —, and process of regeneration of the catalyst. Soc. des Acières et Forges de Firminy. E.P. 124,194, 18.2.19. Conv., 13.3.18.

AN improved catalyst for the hydration of acetylene consists of a solution of sulphuric acid containing mercury, ferric, and vanadium salts, which after becoming exhausted can be regenerated by electrolytic oxidation either intermittently or continuously, using a cell with a magnetite or platinum anode and a current density of 3–5 amps. per sq. dm., the temperature being maintained at 30°–40° C. As an example, the catalyst may consist of a solution containing 10–20% of sulphuric acid, 2% of mercury, 0.3–0.5% of iron, and 0.025–0.1% of vanadium, the metals being present as sulphates. This bath is maintained at about 65° C., and up to 1 ton of aldehyde can be produced with 2 cb. m. of catalyst before the reaction slows down. For the electrolytic regeneration it is not necessary to interrupt the process and transfer at once the whole of the catalyst to the cell, but a fraction of the catalytic liquid in circulation may be withdrawn by means of a branch pipe and made to traverse the electrolytic cell, whereby continuous regeneration is effected.—G. F. M.

Ethylene oxide; Preparation of —. Badische Anilin- u. Soda-Fabrik. G.P. 299,682, 11.4.15.

IN the preparation of ethylene oxide much better results are obtained if, instead of adding ethylene chlorhydrin to warm caustic alkali solutions, bases are added to warm ethylene chlorhydrin or its solutions. By this means the tendency to glycol formation is minimised.—G. F. M.

Carboxyl groups; Elimination of —. [Production of phenols.] J. E. Marsh. E. P. 144,897, 14.7.19.

THE elimination of the carboxyl group from organic acids, particularly hydroxy-aromatic acids, such as gallic acid, is effected by heating the acid with a dry metallic salt, preferably an alkali halide. The reaction is advantageously carried out under reduced pressure, and if the product is volatile its recovery is effected in the same operation by distillation, the decomposition being conducted in a vacuum still. For example, pyrogallol in a technically pure condition is obtained by distilling a mixture of gallic acid with half its weight of sodium chloride at a temperature of 250° C. and a pressure of 10 mm. The yield amounts to 95% of the theoretical.

—G. F. M.

Ureas; Manufacture of asymmetric —. A. V. Blom. E.P. 125,584, 17.3.19. Conv., 17.4.18.

ASYMMETRIC ureas are produced by the interaction of phosgene with aminonaphtholsulphonic acids and primary aromatic diamines in presence of alkalis or alkali carbonates. The aromatic diamine must contain two primary amino groups having different reaction speeds, one either being protected by an ortho substituent as in the toluylenediamines, or else being formylated. The two components are brought together, in molecular proportions, continuously into reaction with the calculated quantity of phosgene in a large stirring vat at 40° C., whilst the alkali solution is allowed to run in in sufficient quantity immediately to neutralise the hydrogen chloride liberated during the reaction.—G. F. M.

Arsine derivatives; Preparation of —. A. Job and H. Guinot. E.P. 144,806, 18.3.19.

NITRILES of the general formula R_2AsCN , where R is a fatty or aromatic radicle, are converted into amides and acids of the type $R_2AsCONH_2$ and R_2AsCO_2H by the general methods for the hydration of nitriles. For example, cacodyl cyanide is converted into dimethylarsiniformic acid by boiling it

with about 3 times its weight of 50% sulphuric acid for 12 hrs. The acid is isolated through its calcium or barium salt and crystallised from acetone and alcohol. Diphenylarsinoformic acid may be similarly prepared from diphenylarsino cyanide, or the amide may first be prepared and isolated by treating the cyanide with the theoretical quantity of hydrogen peroxide at 40°–50° C. in alkaline solution and crystallising from alcohol the solid product which separates out. The amide is then converted into the acid by treatment with nitrous acid in the usual way. Diphenylarsinoformic acid is only slightly soluble in water, but most of its salts are very soluble.—G. F. M.

Selenium compounds; Preparation of aromatic —. A. Schoeller. G.P. 319,161, 10.5.19.

AROMATIC selenium compounds are obtained in quantitative yield by the action of the diazonium salts of the corresponding bases on the aqueous solution of polyselenides prepared by triturating or fusing together an excess of metallic selenium with a caustic alkali. It is not necessary to exclude air during the operation as in the case of alkali selenide or hydroselenide solutions. Examples are given of the preparation of diphenylselenide from phenyldiazonium chloride, and diphenyldiselenide and diphenylselenide carboxylic acids from diazotised anthranilic acid. The two acids are separated by oxidation with dilute nitric acid or with hydrogen peroxide, whereby the former is converted into the corresponding selenic acid, and the latter into the lactone of diphenylselenium oxide di-*o*-carboxylic acid. On treatment with cold dilute ammonia only the acid dissolves, and the compounds thus separated are reconverted into the original acids by reduction with zinc dust in alkaline solution.

—G. F. M.

Chlorosulphamides; Preparation of —. M. Claass. G.P. 318,899, 31.12.18.

THE stability of aromatic chlorosulphamides is increased by the introduction of a carboxyl group. The new compounds may be prepared by the usual methods and are useful germicides. The dichlorosulphamide of benzoic acid, prepared from *p*-sulphaminobenzoic acid and alkali hypochlorite solution by acidification with acetic acid, melts at 203° C. The monochloro compound, formed in small quantities in the same reaction, melts at 223° C.

—G. F. M.

Alcohols; Preparation of polyhydric —. O. Matter. G.P. 299,074, 19.4.16.

POLYHYDRIC alcohols are obtained by saponification of the corresponding halogen substituted hydrocarbons by means of aqueous solutions of carbonates, bicarbonates, or mixtures of the two, preferably in a closed vessel to prevent the escape of carbon dioxide. If the caustic alkali is added periodically or continually the saponifying agent is continually regenerated, and its concentration maintained; the velocity of the reaction is thereby considerably increased. Examples are given of the preparation of ethylene glycol from ethylene dichloride and sodium carbonate or bicarbonate at 130°–140° C.

—G. F. M.

Tropinonecarboxylic acid esters; Manufacture of —. R. Willstätter. G.P. 302,401, 27.1.17.

N-METHYLPYRROLEDIACETIC acid esters, prepared as described in G.P. 300,672 (J., 1920, 385 A), on reduction with hydrogen and platinum in acetic acid solution yield N-methyltetrahydropyrrolediadic acid esters. The diethyl ester, a thin, colourless oil with an alkaline reaction, has b.p. 162.5° C. (9 mm.), and is soluble in 60–70 parts of boiling water; the dimethyl ester has b.p. 155.5° C. (12 mm.), and is soluble in 6–7 parts of boiling water. By the action of sodium, sodium

alcoholate, or sodamide, N-methyltetrahydropyrrolediadic acid esters are converted into tropinonecarboxylic acid esters. The ethyl ester is difficult to separate from mixtures with other organic compounds, very readily decomposes and resinifies, is soluble in water and alcohol, and gives a strong reaction with ferric chloride. Tropinonecarboxylic acid ethyl ester on heating with dilute acids yields tropinone, which can be isolated as such, or as the picrate or dibenzal derivative. Reduction by electrolysis or by sodium-amalgam yields *r*-ecgonine ester.—L. A. C.

Acetic aldehyde; Preparation of — from acetylene. A. Guyot, Assr. to Comp. des Prod. Chim. d'Alais et de la Camargue. U.S.P. 1,313,715, 15.6.20. Appl., 5.4.18.

SEE E.P. 130,650 of 1918; J., 1919, 740 A.

Trichloroethylene; Preparation of — from tetrachloroethane. A. Guyot, Assr. to Comp. des Prod. Chim. d'Alais et de la Camargue. U.S.P. 1,313,716, 15.6.20. Appl., 18.6.19.

SEE E.P. 132,755 of 1919; J., 1919, 847 A.

Urea and intermediate products; Manufacture of —. C. Bosch, Assr. to The Chemical Foundation, Inc. U.S.P. 1,311,673, 29.6.20. Appl., 21.1.15.

SEE E.P. 21,117 of 1911; J., 1915, 924.

Metacholesteryl. G.P. 318,900—1. See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Colouring matters of flowers; Use of — in colour photography. P. R. Kögel, Phot. Korrr., 57, 86–91. Chem. Zentr., 1920, 91, IV., 60.

SEVERAL of the benzopyrillium compounds occurring in flowers are shown to have a relatively high light-sensitiveness. The anthocyanins which by themselves are light-fast, can be made very light-sensitive by the addition of sensitisers, *e.g.*, *o*-anethol to cyanidin hydrochloride. *o*-Anethol can occur as a degradation product in plants, so that it would appear that these dyestuffs can form their own sensitisers. An objection to the use of an anthocyanin in the bleach-out process is its low covering power.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Dicyanodiamide as explosive. J. Baumann. Chem.-Zeit., 1920, 44, 474.

MIXTURES of dicyanodiamide (1 pt.) with ammonium nitrate (3 pts.), and of dicyanodiamide (1 pt.) with potassium chlorate (3 pts.) had an explosive power (in the lead block test) exceeding that of TNT by 17% and 72% respectively. The ammonium nitrate mixture can be ground without danger, and is particularly suitable for mining purposes.

—C. A. M.

Nitrophenols. Vignon. See III.

PATENTS.

Explosive; Ammonium-nitrate —. W. O. Snelting, Assr. to Trojan Powder Co. U.S.P. 1,313,063, 8.6.20. Appl., 11.1.19.

THE explosive consists of over 40% of ammonium nitrate together with nitrostarch and a finely divided material of high covering power interspersed among the particles of ammonium nitrate.

—T. St.

Explosive; Ammonium-nitrate —. J. B. Bronstein and C. E. Waller, Assrs. to Trojan Powder Co. U.S.P. 1,343,077, 8.6.20. Appl., 5.2.20.

THE explosive consists of a substantially non-explosive mixture of ammonium nitrate and trinitrotoluene with a sufficient amount of a detonating agent to render the mixture explosive.

—T. St.

Nitrostarch; Process of stabilising —. B. J. Flurschein, Assr. to Aetna Explosives Co. U.S.P. 1,343,317, 15.6.20. Appl., 17.5.17. Renewed 5.3.20.

NITROSTARCH is boiled with an aqueous solution of cyanamide, and, after the final wash water has been eliminated, is dried.—T. St.

XXIII.—ANALYSIS.

Filter with wire-gauze layer. F. Mach and P. Lederle. Chem.-Zeit., 1919, 43, 831.

THE platinum gauze used in the filter described previously by the authors (J., 1919, 476 A) may be replaced by silver gauze.—W. P. S.

Iron basins; Use of — in analysis. H. Sertz. Z. angew. Chem., 1920, 33, 156.

IRON basins or crucibles are useful for sulphide fusions where platinum or silver vessels are unsuitable. The iron is attacked but slightly, and its resistance may be increased by treating the vessel with nitric acid.—W. P. S.

Weight burette for gas analysis. E. R. Weaver and P. G. Ledig. J. Amer. Chem. Soc., 1920, 42, 1477—1485.

AN apparatus is described whereby the volume of gas taken into a gas analysis burette and that absorbed by any reagent is measured by weighing the amount of mercury expelled or taken into the burette. Specimen analyses show that the mean error in a single gas absorption is 0.004%, with a maximum error of 0.014%.—J. F. S.

Analysis by fractional distillation. Method of maxima and minima. C. Moureu, C. Dufraisse, and P. Robin. Bull. Soc. Chim., 1920, 27, 523—527.

FOR the separation of mixtures of liquids with boiling-points close together, successive fractional distillations are recommended, the division into fractions being controlled not by the boiling-point, but by maximum or minimum values of physical properties such as the sp. gr., refractive index, viscosity, etc. The rectification is continued, similar fractions being united for further fractionation, until a fraction is obtained which when divided by slow distillation, gives sub-fractions all having identical boiling-points and physical properties. A simple vaseline manometer is described for detecting very slight variations in pressure during distillation, and by means of which a distillation may be interrupted and later readily resumed under the same reduced pressure.—W. G.

Tint photometer; The Hess-Ives — and its use with raw sugars. G. P. Meade and J. B. Harris. J. Ind. Eng. Chem., 1920, 12, 686—688.

THE scale readings of this photometer (J., 1917, 449) do not express directly the relative amounts of colour, and for solutions containing 1, 2, 3, 4, etc., units of material run in powers of the readings for 1 unit, considering the scale readings as decimal fractions. Expressed algebraically, this relationship between the scale readings and the amounts of colour becomes $y = K^x$, where y is any scale reading, K the reading for 1 unit of material, and x the

number of units of material to give the scale reading. To avoid repeated calculations, a table is given showing the units of colour corresponding with each scale reading from 100 to 1. The instrument may be used for determining the colour of raw sugars.

—W. P. S.

Melting-points; Method of avoiding temperature correction in the determination of —. M. François. Bull. Soc. Chim., 1920, 27, 528—536.

A MODIFIED form of the flask generally used as a bath in determining melting-points is described, by means of which the thermometer, including the stem, may be immersed in the liquid of the bath and be heated by the return flow of liquid in the convection current set up by heating the bulb of the flask. By a simple arrangement of mirrors it is possible to read the temperature on the thermometer and observe the material in the capillary melting-point tube simultaneously through one telescope.—W. G.

Boiling-point; Method of determining the — with small amounts of substance. V. Arreguine. Soc. Chim. Argent., 3 (Reprint).

A COMPACT and simple apparatus is described by means of which it is possible to determine the boiling-point of liquids with b.p. below 210° C., with only about 3 mg. of substance. (Cf. J.C.S., Sept.)—W. G.

Specific refraction; Significance of — in analytical chemistry. N. Schoorl. Rec. Trav. Chim., 1920, 39, 594—599.

THE author discusses the limitations of the use of the specific refraction, $(n-1)/d$, values for the identification of an unknown organic compound.

—W. G.

Reactions; Sensitiveness of qualitative —. I. Potassium ions. O. Lutz. Z. anal. Chem., 1920, 59, 145—165.

TESTE of the sensitiveness of various reactions for potassium were made under the same conditions as to temperature (18° C.), time of contact (5 mins.), and volume of potassium chloride solution (5 c.c.); the figures give the minimum concentration of potassium which could be detected:—Perchloric acid, 1:435; phosphomolybdic acid, 1:561; platinum chloride, 1:587; sodium borofluoride, 1:970; aniline silicohydrofluoride, 1:1022; sodium phosphotungstate, 1:2170; phosphotungstic acid, 1:2809; sodium cobaltinitrite, 1:25000; sodium-bismuth thiosulphate, 1:57000; sodium picrate, 1:840; sodium amino- β -naphtholsulphonate, 1:1022; sodium bitartrate, 1:1050. These results may be expressed in weight of potassium, e.g., 1:435 would be 11×10^{-3} g. K per 5 c.c.

Sulphuric acid; Gravimetric determination of —. L. W. Winkler. Z. angew. Chem., 1920, 33, 159—160, 162—163. (Cf. J., 1920, 331 A.)

THE presence of ammonium chloride to some extent decreases the interference of magnesium, zinc, manganese, and other salts on the precipitation of sulphuric acid as barium sulphate. Tables are given which show the corrections to be made for the effect of alkali chlorides on the precipitation. In solutions containing alkali sulphates or magnesium sulphate the sulphuric acid is most accurately determined by adding 1 g. of ammonium chloride and 1 c.c. of N/1 hydrochloric acid to 100 c.c. of the solution, boiling the mixture (a fragment of metallic cadmium is added to ensure smooth boiling), and adding a slight excess of 10% barium chloride solution. The barium sulphate is collected after 18 hrs., washed first with 25 c.c. of cold water, then with hot water, dried at 130° C., and weighed, or the precipitate may be ignited before being

weighed. The weight is corrected from tables to obtain the actual amount of barium sulphate.

—W. P. S.

Phosphoric acid; Estimation of small amounts of — as barium phosphomolybdate in the presence and in the absence of organic phosphorus compounds. S. Posternak. Bull. Soc. Chim., 1920, 27, 507—513, 561—568.

For the estimation of amounts of phosphorus as small as 0.05 mg. the following methods give accurate results. In the absence of organic phosphorus compounds 10 c.c. of the phosphate solution is mixed with 10 c.c. of a 20% solution of ammonium sulphate and 1 c.c. of sulphuric acid; the mixture is heated to boiling, 5—10 c.c. of a 10% solution of ammonium molybdate added, allowed to stand for 15 mins., and the ammonium phosphomolybdate collected, washed with a 5% solution of ammonium nitrate, dissolved in ammonia, and reprecipitated by the addition of a 10% solution of barium chloride. This precipitate is collected, washed with water, dried, ignited at a red heat, cooled, and weighed; it has the composition $4\text{Ba}_2[\text{PO}_4(\text{MoO}_4)_{12}]_3 \cdot \text{Ba}_2\text{SO}_4(\text{MoO}_4)_6$ (1 g. = 0.00739 g. P). In the presence of organic phosphorus compounds the mineral phosphate in 20 c.c. of solution is precipitated at 12°—15° C. in the presence of 10 c.c. of a 20% solution of ammonium nitrate. The precipitate is collected after two hours, and its subsequent conversion into barium phosphomolybdate is carried out as described above. In this case the precipitate has the composition $8\text{Ba}_2[\text{PO}_4(\text{MoO}_4)_{12}]_3 \cdot \text{Ba}_4(\text{MoO}_4)_6$, the factor being 0.00780.—W. G.

Iron; Estimation of — and separation of manganese from iron by nitrosophenylhydroxylamine ammonium (cupferron). E. H. Archibald and R. V. Fulton. Trans. Roy. Soc. Canada, 1919, 13, [iii.], 243—253.

THE cupferron precipitate of iron is slightly soluble in the acid solution from which it is precipitated. The solubility is apparently not affected by the substitution of sulphuric for hydrochloric acid, but is appreciably lowered by addition of ammonium chloride. The precipitate is slightly soluble in the wash-water. If corrections are applied for these solubilities, the determination of iron by this method becomes very exact. Iron can be separated from manganese in a solution containing four times as much manganese as iron, the amount of manganese carried along with the precipitate being inappreciable for most purposes. This amount increases with the acid dilution of the mother liquor. The presence of ammonium chloride to the extent of 5 g. in 100 c.c. of solution is an advantage in this separation.—J. R. P.

Iron; Volumetric determination of — in the presence of hydrochloric acid, phosphorus, oxy-acids, and organic matter. H. D. Richmond and E. M. Ison. Analyst, 1920, 45, 258—260.

To determine iron in pharmaceutical syrups etc. 5 or 10 c.c. of the sample is treated with a few drops of hydrochloric acid and *N*/10 permanganate solution is added until a transitory purple coloration is produced throughout the solution. An equal volume of concentrated hydrochloric acid is then added, a small quantity of sodium carbonate to give an atmosphere of carbon dioxide, and then *N*/10 stannous chloride solution, a drop at a time, until a drop of the mixture gives a blue coloration with ferricyanide solution. The mixture is then titrated with the stannous chloride solution until a drop no longer gives a coloration with thiocyanate solution. 1 c.c. of *N*/10 stannous chloride solution = 0.0056 g. Fe.—W. P. S.

Iron; Determination of small quantities of — as thiocyanate. R. Willstätter. Ber., 1920, 53, 1152—1154.

THE solution under investigation (0.5—1.0 c.c.) is treated with concentrated hydrochloric acid

(0.5 c.c.) and made up to 50 c.c. with freshly prepared ammonium thiocyanate solution (40% or, with larger amounts of iron, 10%). The coloration is matched by standard solutions of iron which are similarly treated. Difficulties are caused by the presence of iron in the purest commercial ammonium thiocyanate and by the instability of ferric thiocyanate in solution. The former is overcome by boiling the thiocyanate solution for a short time, after which it remains colourless on cooling. The latter is due to the gradual reduction of the ferric salt by the thiocyanate, the process not appearing to be catalysed by the ferrous salt formed. (Cf. J.C.S., Sept.)—H. W.

Calcium; Comparison of ten different methods of estimation. G. E. Ewe. Amer. J. Pharm., 1920, 92, 401—410.

CALCIUM was estimated by the following methods in a specimen of calcite containing 0.0407% of impurities (silica, iron, and alumina), the results being expressed as percentages of CaCO_3 :—(1) Precipitation as oxalate and weighing as carbonate, 99.57%. (2) Precipitation as carbonate and weighing as such, 100.07%. (3) Weighing carbon dioxide liberated by hydrochloric acid by absorption in potassium hydroxide solution, 100.03%. (4) Precipitation as oxalate and weighing as oxide, 99.86%. (5) Precipitation as sulphate and weighing as such, 99.68%. (6) Precipitation as oxalate and weighing as sulphate, 100.39%. (7) Precipitation as oxalate and titration with permanganate, 99.68%. (8) Precipitation as oxalate from a slightly acid solution, 99.87%. (9) Weighing by difference carbon dioxide liberated by hydrochloric acid in a Geissler apparatus, 99.80%. (10) Decomposition by excess of standard sulphuric acid and back titration of the excess, 99.75%.—G. F. M.

Mercury in organic compounds; Determination of —. M. François. Bull. Soc. Chim., 1920, 27, 568. (Cf. J., 1920, 280 A).

FOR the deposition of the mercury the following process is recommended: About 0.59% of the dry powdered organic matter is mixed with 30 c.c. of ether, 10 c.c. of alcohol, and 1 c.c. of strong hydrochloric acid, and 1 g. of zinc turnings is added immediately. The mixture is shaken and left for $\frac{1}{2}$ hr., after which another 1 g. of zinc is added, the mixture shaken, and again left for $\frac{1}{2}$ hr. A third 1 g. of zinc is added and the mixture left for 24 hrs. By this time all the mercury is deposited on the zinc, and the estimation is completed as described (*loc. cit.*).—W. G.

Acetic acid in acetates; Method for the determination of —. O. A. Pickett. J. Ind. Eng. Chem., 1920, 12, 570—571.

DISTILLATION with xylene as a carrier for the acetic acid is recommended; 2.5 g. of the acetate is mixed in a distillation flask with 40 c.c. of water, 20 c.c. of 85% phosphoric acid, and 350 c.c. of xylene, and the mixture is distilled until only a thin film of xylene remains on the surface of the phosphoric acid solution. The acidity of the distillate is titrated and allowance made for hydrochloric acid if the acetate contained chlorides.—W. P. S.

*Methyl alcohol; Detection of — by formation of methyl *p*-bromobenzoate.* W. Autenrieth. Arch. Pharm., 1920, 258, 1—14. Chem. Zentr., 1920, 91, IV., 29.

THE formation of crystalline methyl *p*-bromobenzoate (m.p. 77°—78° C.) by the action of *p*-bromobenzoyl chloride with subsequent conversion to *p*-bromobenzamide (m.p. 188° C.) may be employed for the detection of methyl alcohol. Formation of the ester is nearly quantitative, and 0.05 g. of the alcohol can be detected, even if highly diluted. Ethyl *p*-bromobenzoate is a liquid which does not

solidify even at -16° C.; the crystalline methyl ester is formed in an aqueous solution containing 0.2% of methyl and 0.8% of ethyl alcohol; in the case of mixtures of the alcohols, however, it is advisable to increase the proportion of methyl alcohol by fractional distillation, using the first fraction for the test. By this means 0.1 g. of methyl alcohol can be detected in 200 c.c. of urine.

—L. A. C.

Nitro compounds; Examination of — by means of *titanium chloride and sulphate*. C. F. van Duin. Rec. Trav. Chim., 1920, 39, 578—585. (Cf. Callan and others, J., 1920, 86 r.)

In the estimation of nitro-nitrogen by Knecht's method it is recommended to use titanium sulphate instead of the chloride for feebly substituted nitro-compounds. *s*-Trinitrobenzene may be successfully reduced to dinitroaniline by means of titanous chloride. From the results of the reduction of 2,3,4,6-tetranitroaniline and of trinitromethylnitroaminophenol by titanous chloride it is believed that the nitro group in position 3 has the structure $\cdot\text{NO}_2$ and not $\cdot\text{ONO}$.—W. G.

Nitro-groups; Rapid determination of — by a *modification of Young and Swain's method*. L. Desvergues. Ann. Chim. Analyt., 1920, 2, 141—143.

FROM 0.2 to 0.3 g. of the substance is dissolved in alcohol (solution of the substance in sodium hydroxide often gives a yellow coloration which interferes with the subsequent procedure). 100 c.c. of 3% stannous chloride solution and 25 c.c. of hydrochloric acid are added, and the mixture is heated under a reflux condenser for 2 hrs.; a current of carbon dioxide being passed through the mixture during the whole time. The mixture is then diluted to a definite volume and the excess of stannous chloride is titrated in an aliquot portion with iodine solution. The method may be used for a large number of nitro-compounds, but fails in the case of nitrophenols, nitronaphthalenes, and certain volatile compounds.—W. P. S.

See also pages (A) 564, *Thiocyanates* (Shaw), *Emulsions* (Glaser); 565, *Petroleum spirit* (Hess); 566, *Tar from coal* (Fischer and Schrader); 567, *Phenol etc.* (Chapin); 568, *Silk and wool* (Battagay and Voltz); 569, *Tin* (Bouman), *Ammonium hydrogen fluoride* (Chase); 574, *Oxygen in iron* (Oberhoffer and Von Keil), *Sulphur in iron etc.* (Vita), *Zirconium and titanium in steel* (Lundell and Knowles), *High speed steel* (Ward), *Oxygen in cyanide solutions* (Hamilton); 575, *Pickling solutions* (Boyle); 577, *Ink in writing* (Mitchell); 578, *Rubber* (Lewis and McAdams); 579, *Fertilisers* (Moore); 580, *Reducing sugars* (Van Nouhuys; Van der Linden), *Naphthol test for sugar* (Kauffman), *Diastatic power* (Flohil), *Alcohol* (Waldbott); 581, *Jelly* (Campbell), *Carbon dioxide in water* (Noll); 582, *Phytin* (Rippel).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Barbet et Fils et Cie. Recovery of volatile liquids from mixtures with air. 22,888. Aug. 3. (Fr., 1,8,19.)

Bloor. Apparatus for filtering liquids. 22,650. July 30.

Bloxam (Ges. f. Maschinelle Druckentwässerung). Presses for briquetting or drying. 22,748. July 30.

Brutzkus. Apparatus for chemical production and research. 23,039. Aug. 4. (Ger., 4,8,19.)

Buckham, Darnell, and Purvis. Mixing machines. 22,731. July 27.

Fleury and Robertson. Drying apparatus. 22,447. July 28.

Fuller-Lehigh Co. Pulveriser-mills. 22,738. July 30. (U.S., 8,11,19.)

Hinchley. Expressing liquids from materials. 22,880. Aug. 3.

Smallwood. Furnaces. 22,791. July 31. Thompson (Zip Manufacturing Co.) Grinding compounds. 22,321. July 27.

Welter. Producing finely granulated compounds. 22,273. July 26. (Ger., 24,7,19.)

COMPLETE SPECIFICATIONS ACCEPTED.

10,031 (1917). Ges. f. Linde's Eismaschinen. Separating constituents of air or other gaseous mixtures. (147,956.) Aug. 5.

8821 (1918). Barbet et Fils et Cie. Tubular evaporating, condensing, and cooling apparatus. (117,070.) Aug. 11.

2380 (1919). Imray (Jefferies). Cooling and liquefaction of gaseous fluids. (148,588.) Aug. 11.

7871 (1919). General Chemical Co. Apparatus for absorbing gases for a gaseous mixture. (124,761.) Aug. 5.

12,254 (1919). Morris and Haviland. See XI.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Ballantine and Harley. Drying peat etc. 22,248. July 26.

Blair. Treatment of peat. 22,885. Aug. 3.

Bradley. Composition for use as a fuel or building material. 23,053. Aug. 5.

Colmore and Field. Coal substitute. 22,993. Aug. 4.

Corthesy. Distillation of liquid hydrocarbons etc. 22,720. July 30.

Cummins. Gas-retort settings. 22,952. Aug. 4.

Davidson. Destructive distillation of coal etc. 22,487. July 28.

Engelke. Distillation of hydrocarbon oils, asphalts, etc. 22,373. July 27.

Evans, Stanier, and South Metropolitan Gas Co. Removing hydrogen sulphide from gases. 22,585. July 29.

Evans. Destructive distillation of carbonaceous substances. 23,110. Aug. 5.

Farbw. vorm. Meister, Lucius, u. Brünig. Process for desulphurising gases. 22,460. July 28. (Ger. 29,7,19.)

Johnson (Badische Anilin u. Soda Fabr.). Process for desulphurising gases. 23,031. Aug. 4.

MacLaurin, and Scottish By-Products, Ltd. Manufacture of lubricating greases. 22,407. July 28.

Pease. 22,534. See VII.

Tozer. Low-temperature carbonisation and gasification of coal etc. 22,637. July 30.

COMPLETE SPECIFICATIONS ACCEPTED.

3918 (1919). Smith. Furnace retorts. (123,739.) Aug. 11.

8559 (1919). Nielsen. Manufacture of peat fuel. (125,083.) Aug. 11.

13,607 (1919). Campbell. Apparatus for making gas. (148,027.) Aug. 5.

20,697 (1919). Wells. Gas-producers. (148,057.) Aug. 5.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

Atack and Robertson. Halogenation of anthraquinone derivatives. 23,113. Aug. 5.
 Cortesey. 22,720. *See* II.
 Engelke. 22,373. *See* II.

COMPLETE SPECIFICATION ACCEPTED.

4278 (1919). Sutton. Sulphonating processes. (147,967.) Aug. 5.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

Atack and Robertson. 23,113. *See* III.
 Atack and Soutar. Intermediates and dye-stuffs of anthraquinone series. 23,114. Aug. 5.
 Cowley (Cowley). Dye. 22,457. July 28.

COMPLETE SPECIFICATION ACCEPTED.

10,246 (1919). Maclaurin. Manufacture of dyes. (148,617.) Aug. 11.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Bland and Wack. Colouring surfaces of paper, cardboard, etc. 22,396. July 28.
 Bronnert. Manufacture of artificial silk. 22,898 and 23,165. Aug. 3 and 6.
 Bronnert. Production of high-percentage sulphite cellulose. 23,036. Aug. 4.
 Dreaper. Manufacture of artificial silk yarns etc. 22,827. July 31.
 Dreyfus. Manufacture of films, celluloid-like masses, etc. 22,974. Aug. 4.
 Lord, White, and Radcliffe Paper Mill Co. Manufacture of paper etc. 22,971. Aug. 4.
 Meldia Manuf. Co., and Melsom. Dope for balloon fabric etc. 22,574. July 29.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Fanshawe. Finishing fabrics. 22,441. July 28.
 Ferrand and Riley. Apparatus for treating textiles with fluids. 22,969. Aug. 4.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Candlot. Lime, cement, etc. kilns. 22,595. July 29. (Fr., 10,919.)
 Fabry. Obtaining sulphur from hydrogen sulphide. 22,185. July 26.
 Kaltenbach. Manufacture of sulphuric acid. 22,606. July 29. (Fr., 18,220.)
 Leek, Meeze, and Olympia Oil and Cake Co. Manufacture of hydrogen by the iron-steam process. 22,188. July 26.
 Mooney. Manufacture of a solution of chromic sulphate. 23,020. Aug. 4.
 Pease. Extraction of ammonia from gases. 22,534. July 29.
 Pfannenschmidt. Acid chambers, towers, etc. 22,655. July 30. (Ger., 31,719.)
 Reid and Spashett. Concentrating sea-water etc., and recovering salts therefrom. 22,380. July 27.
 Wilton and Wilton. Manufacture of sulphate of ammonia. 23,247. Aug. 7.

COMPLETE SPECIFICATIONS ACCEPTED.

1878 (1918). Marks (Hüttenwerk Niederschöne-weide A.G.). Manufacture and use of cuprous oxide. (147,958.) Aug. 5.
 10,071 (1919). Morel. Production of basic sulphate of chrome and its use in tanning etc. (148,615.) Aug. 11.
 31,908 (1919). Postalozza. Electrolytic apparatus for direct production of hypochlorites. (148,025.) July 5.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Marlow. Gas-fired ovens or kilns for making tiles, pottery, etc. 22,641. July 30.
 Meyer. Grinding or abrading substances. 22,816. July 31. (Ger., 21,119.)

IX.—BUILDING MATERIALS.

APPLICATIONS.

Ambrose. Method of accelerating setting of concrete etc. 22,920. Aug. 3.
 Bean. Manufacture of Portland, Russian, lime, etc. cements. 22,955. Aug. 4.
 Bradley. 23,053. *See* II.
 Candlot. 22,595. *See* VII.
 Planson, and Traun's Forschungslaboratorium Ges. Manufacture of artificial wood etc. 22,825. July 31.

COMPLETE SPECIFICATIONS ACCEPTED.

10,735 (1919). Henderson. Bituminous binders. (148,003.) Aug. 5.
 641 (1920). Smidth & Co. Method of making cement. (137,534.) Aug. 5.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Botten, Gibson, Hounslow, and McRorie. Soldering aluminium. 22,926. Aug. 3.
 Imray (Jackson and Co.). Preliminary treatment of ores or metallurgical products. 22,459. July 28.
 Lavandeyra. Aluminium alloys, and method of making and treating them. 22,562. July 29. (U.S., 7,819.)
 Lévy and Terrisse. Acid-proof castings. 22,907. Aug. 3. (Switz., 27,919.)
 Manchester Furnaces, Ltd., and Russell. Furnaces for heat-treating materials. 22,568. July 29.
 Meakin. Removing oxide from sheet metal. 22,312. July 27.
 Minerals Separation, Ltd., Sellers, and Wood. Ore concentration. 22,763. July 30.
 Tugwood (Dwight and Lloyd Metallurgical Co.). Ore-sintering mechanisms. 23,238. Aug. 7.

COMPLETE SPECIFICATIONS ACCEPTED.

10,333 (1919). Jackson. Production of iron. (147,999.) Aug. 5.
 10,439 (1919). Priest. Muffle and semi-muffle and like furnaces. (148,630.) Aug. 11.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Adam. Electrolysis. 22,214. July 26.
 Adam, Fieldhouse, and Stevenson. Electrolysis. 22,245. July 26.
 Alexander (Stuart Electrolytic Cells, Inc.). Electrode and electrolytic batteries. 22,980. Aug. 4.
 Mull. Utilising mica for electrical purposes. 22,973. Aug. 4.

Smith. 23,201. *See* XIX.
Vesme. Apparatus for producing by electrolysis gas under pressure. 22,260. July 26.

COMPLETE SPECIFICATIONS ACCEPTED.

10,130 (1919). Teixeira. Electric furnaces. (147,988.) Aug. 5.
12,254 (1919). Morris and Haviland. Electrical precipitation of particles from gases. (148,659.) Aug. 11.
22,913 (1919). Kremenezky and Dukes. Galvanic cells. (132,803.) Aug. 5.
31,908 (1919). Pestalozza. *See* VII.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Hough. Manufacture of scouring and emulsifying compounds. 22,677. July 30. (Fr., 26.3.20.)
Nicholson. Apparatus for extracting fats and oils. 22,386. July 27.

COMPLETE SPECIFICATIONS ACCEPTED.

2362 (1919). Dunham. *See* XX.
10,094 (1919). Force. Detergent compound. (125,986.) Aug. 5.
1977 (1920). Verein. Chem. Werke. *See* XVIII.
16,893 (1920). Dunham. Edible oil preparations in dry form. (148,734.) Aug. 11.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Wade (Redmanol Chemical Products Co.). Production of phenolic condensation products. 22,612-3. July 29.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Catalpo, Ltd., and Schidrowitz. Manufacture of caoutchouc etc. 22,491. July 28.
Joannides. Manufacture of dental rubber. 22,881. Aug. 3.
Stewart. Vulcanisation of rubber. 22,503. July 29.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Poppe. Removing grease from skins and hair with recovery of the solvent. 22,282. July 26.
Rautenstrauch. Unhairing skins and hides. 23,019. Aug. 4.

COMPLETE SPECIFICATIONS ACCEPTED.

4571 (1919). Rampichini & Co. Processes based on the action of enzymes for bating hides, and baths, mixtures, etc. for use therein. (124,718.) Aug. 11.
10,074 (1919). Morel. *See* VII.

XVI.—SOILS; FERTILISERS.

APPLICATION.

Broadbridge and Edser. Production of fertiliser. 23,162. Aug. 6.

XVII.—SUGARS; STARCHES; GUMS.

COMPLETE SPECIFICATION ACCEPTED.

1977 (1920). Verein. Chem. Werke. *See* XVIII.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Fleischmann Co., and Harrison. Production of yeast. 22,374-7. July 27.

COMPLETE SPECIFICATIONS ACCEPTED.

1977 (1920). Verein. Chem. Werke. Manufacture of glycerol from sugar. (138,331.) Aug. 5.
9321 (1920). Jarraud. Ageing spirits and alcoholic liquors. (141,687.) Aug. 5.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Fitzgerald. Preparation of commercial products from blood. 22,751. July 30.
Humphries and Penn. Preserving and sterilising canned fruit etc. 23,239. Aug. 7.
Jones, Watson, and Woodlands, Ltd. Manufacture of flour and bread. 22,267. July 26.
Luca and Petrecca. Preservatives. 22,983. Aug. 4.
Marshall and Sutcliffe. Preparation of edible products and food for animals. 22,962. Aug. 4.
Smith. Electrically treating foods etc. 23,201. Aug. 6.

COMPLETE SPECIFICATIONS ACCEPTED.

10,176 (1919). Namal. Vennoots. Jurgens Vercen. Fabr. Artificial milk products. (128,544.) Aug. 5.
16,893 (1920). Dunham. *See* XII.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Hewitt. Production of tasteless derivative of quinidine. 23,046. Aug. 5.
Kuh. Production of neutral alkyl esters of sulphuric acid. 22,873. Aug. 3. (Austria, 8.8.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

1600 (1919). Atack and Haworth. Manufacture of aromatic alkylamino compounds. (147,964.) Aug. 5.
2362 (1919). Dunham. Medicinal oil preparations in dry form. (148,587.) Aug. 11.
23,372 (1919). Napp (Hoffmann-La Roche & Co.). Manufacture of complex silver salts of aliphatic amino acids. (148,074.) Aug. 5.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Christensen. Photographic reproduction. 22,760. July 30. (Denmark, 25.3.20.)
Price. Photographic printing. 22,589. July 29.

XXII.—EXPLOSIVES; MATCHES.

APPLICATION.

Marks (Du Pont de Nemours and Co.). Propellant, and process of producing same. 22,711. July 30.

XXIII.—ANALYSIS.

APPLICATION.

Hartridge and Peters. Method of determining acidity or alkalinity of fluids. 22,197. July 26.

I.—GENERAL; PLANT; MACHINERY.

Heat transmission; Effect of air in steam on the coefficient of —. C. S. Robinson. J. Ind. Eng. Chem., 1920, 12, 611–616.

For calculating the coefficient of the transference of heat from vapour to cooling fluid, data are required for the film resistance on the vapour side, the resistance of the metal, and the film resistance on the liquid side. An equation is given for calculating the last-named factor. Curves are also plotted showing the relationship between the film coefficient for condensing vapour and the percentages of steam in mixtures of steam and air, and the effect of air upon the volume percentage of steam and the film coefficient. In the empirical equation a mean value is assumed for the rate of removal of air.—C. A. M.

Colloidal dispersions ["liquid coal" etc.]; Preparation of — on a manufacturing scale. H. Plauson. Chem.-Zeit., 1920, 44, 553–555, 565–567.

About 30 parts of the finely-divided substance is treated with 70 parts of the dispersive agent for a certain time, then diluted with suitable quantities of the agent, thoroughly mixed, and the mixture allowed to stand for 15 mins. to 24 hrs. The supernatant liquid is withdrawn, and the lower layer treated with fresh portions of the agent and again mixed. The liquid then drawn off will behave as a colloidal solution. In Block's "colloid mill" the mixing is effected in globular vessels which revolve eccentrically at an excessively high speed and fling the mixture against baffle bars while, if desired, the vessels may be heated or cooled. By means of this mill the oil may be recovered from crude petroleum sludge; this is emulsified with 80–90% of water, and the oil subsequently separated from the emulsion by heating it at 100° C. In this way 19.8% of petroleum was recovered from a sludge containing 24.6%. Oil may be separated from oil shales, and from oil-cake, filter residues, etc., by emulsification with hot water more effectively than by extraction with solvents etc. For the preparation of "liquid coal" the coal is finely ground and emulsified for $\frac{1}{2}$ to 3 hours in the mill with 60 to 70% of neutral tar oil, peat oil, mineral oil, animal or vegetable oils, etc., at a very high velocity, and for a shorter time if protective colloids are added. Up to 15% of water may be added to the emulsion without causing the coal to separate. "Liquid coal" for Diesel engines must be prepared from wood charcoal or a coal containing not more than 0.8–1.2% ash. The "colloidal mill" may also be used for many other purposes, including the extraction of pure resins, the preparation of fine pigments from coarser kinds of ultramarine etc., and of graphite, and lampblack emulsions, the hydrogenation of oils, and the saponification of fats. Phosphates may be decomposed into a highly-dispersive form in which they are readily absorbed by humic acid. The emulsification may be effected without or with only 1–2% of sulphuric acid.

—C. A. M.

Glass to metal joints. McKelvy and Taylor. See VIII.

PATENTS.

Gas condenser. F. Ryffel. E.P. 124,423, 4.3.19. Conv., 4.3.18.

In a multiple, horizontal, tubular condenser consisting of a series of co-axial tubes, the gas passes downwards through the annular space between the tubes, and the cooling water upwards through the inner tubes. The water collects in a tank which

extends over the top of the tubes, and is distributed over the outside of the outer tubes as spray.

—W. H. C.

Absorbing gases from a gaseous mixture; Apparatus for —. General Chemical Co., Assees. of F. W. de Jahn. E.P. 124,761, 28.2.18. Conv., 3.11.17.

The apparatus is intended more particularly for purifying a nitrogen-hydrogen mixture under high pressure prior to the synthesis of ammonia. The gas at a pressure of about 1400 lb. per sq. in. is washed with water in a coke tower, and then passed through a heater to a gas lift, where it draws in hot caustic soda solution of about 25% strength from a heater. The gas and solution pass to the top of a tower containing pieces of iron and then downward to a tank where the purified gas is separated and passes through a cooling coil to condense water vapour. The separating tank is connected with an overflow tank, and this in turn with a float vessel which controls a discharge valve. The heaters for the gas and the caustic soda are preferably of seamless chromium vanadium steel or tungsten steel. The system provides means for regulating and maintaining automatically a constant level of the absorbing liquid without the use of valves etc. where they would be subject to attack by the liquid.—W. F. F.

Separating liquid mixtures or emulsions; Method of and apparatus for —. J. P. Pedersen. E.P. 125,981, 22.4.19. Conv., 20.4.18.

The liquid to be separated is guided through a circuitous path as an upper layer to a heavier liquid, preferably the pure heavier constituent of the mixture, or as an under layer to a lighter liquid, which may be the lighter constituent of the mixture, by means of a spiral or zig-zag wall that does not extend far into the layers of separated lighter and heavier liquids, so that the main bulk of the liquids after separation remain stationary except for the slow downward or upward flow caused by drawing off the separated liquids continuously through outlets, the level of which may be adjusted. The level of the liquid may also be adjusted (e.g., by means of a float valve) independently of the outlets.—B. M. V.

Catalysis; Processes and apparatus for carrying out chemical reactions by —. Soc. Chim. des Usines du Rhône, anc. Gilliard, P. Monnet et Cartier. E.P. 126,279, 4.4.19. Conv., 3.5.18.

The reacting gases are passed through an insulated tube packed with finely-divided catalytic material, maintained at the requisite temperature by the resistance it offers to the passage of an electric current which is passed through it. No other source of heat is employed. Examples given are the production of acetic acid from acetaldehyde, of ethylene from alcohol, and of acetone from acetic acid.

—W. H. C.

Evaporating liquids [by spraying]; Process of —. G. A. Krause. E.P. 132,256, 2.9.19. Conv., 11.7.17.

In evaporating a liquid by spraying it through air or other drying agent, streams of either external air or of part of the main drying air are directed to the zones of low pressure that would otherwise be caused by the suction of the spraying device and might cause incrustation.—B. M. V.

Evaporators. W. J. Mellersh-Jackson. From The Griscom-Russell Co. E.P. 146,730, 28.7.19.

To keep the levels in a series of evaporators at predetermined heights, the overflow from one evaporator to the next takes place through a narrow slot in a vertical tube contained in an external vessel interposed between the evaporators. The opening of the slot is adjusted by a sleeve which slides up and down the vertical tube and is manipulated from without.—W. H. C.

Stills for the dissociation of chemical solutions by heat; Continuous—R. Fabry. E.P. 146,058, 10.2.20.

PARTLY dissociated liquor (ammoniacal liquor, alkali bicarbonate solution) flows from each tray of a column still through a tubular heat exchanger (where it is heated by the dissociated liquid which flows in series through the heaters) back to the tray next below.—W. H. C.

Drying machines; Rotary cylinder—W. G. and F. R. Simon. E.P. 146,635, 12.4.19.

THE wet material is applied to the heated cylinder or cylinders by one or more unheated cylinders which lift a film of liquid from the feed trough in which it is kept agitated. Any insufficiently dried material is returned to the feed trough.

—W. H. C.

Drying machines. T. Allsop and W. W. Sibson. E.P. 146,744, 3.9.19.

THE goods are placed on a conveyor which takes a circuitous course through a drying chamber, in which air is heated and continuously circulated, the stream of air being divided and guided in various ways so as to cause it to impinge on the goods at various angles.—B. M. V.

Drying apparatus. F. T. Dow. U.S.P. 1,347,473, 20.7.20. Appl., 29.4.19.

MATERIAL is delivered from a hopper on to the lower part of an endless conveyor working in an inclined chamber which is divided longitudinally into two passages. The conveyor is provided with a number of carrier plates which stand out horizontally to receive the material and carry it upwards through the inclined passage. A heating medium is passed through the passage containing the charged carrier plates.—W. F. F.

Muffle furnaces; Rotary—J. R. C. August. E.P. 146,673, 8.5 and 17.6.19.

A HORIZONTAL, rotary muffle furnace is mounted concentrically within a rotary heating chamber, which is divided into flues by longitudinal, radial partitions, which also form a support for the muffle. The muffle is closed at one end, which abuts against the closed inner end of the combustion chamber. This end of the combustion chamber rotates in contact with a flat plate which forms one side of the flue leading from the furnace. The flat plate is provided with two openings which register with the ends of the longitudinal flues in the combustion chamber during its rotation. The flues are connected in such a way that the hot gases are forced to take a zig-zag course. A horizontal partition in the vertical flue leading from the furnace diverts the hot gas through one of the openings in the flat plate into the combustion chamber flues, whence it returns to the vertical flue through the other opening in the plate. A modification is described in which the muffle is heated by a gas or liquid-fuel burner arranged concentrically with the muffle at its rear end.—W. F. F.

Tunnel furnaces. A. Bigot. E.P. 147,391, 12.11.19. Conv., 6.9.19.

THE tunnel traversed by the goods to be heated is surrounded by hollow walls, the spaces in the walls being filled with chequerwork. The combustion of the (gaseous) fuel and air may take place either in the chequerwork, the goods being heated by radiation, or in the central goods tunnel, thus heating the goods directly, or partly in both. The gas for combustion is supplied underneath the hollow walls at the end where the goods emerge, and always passes through at least a portion of the chequerwork, passing (if desired) to the central tunnel through inclined ports. The air for com-

bustion after passing over the emerging goods in an extension of the furnace, thus cooling them, is free to pass either to the chequerwork in the hollow walls or to the central tunnel as determined by dampers in the outlet flues at the chimney end. As an alternative to, or in addition to passing the incoming air over the outgoing goods in the cooling extension, the walls of the extension may be hollow and the spaces filled with chequerwork, and air may be drawn through by suction and used for drying or other purposes.—B. M. V.

Tunnel-kiln. F. J. Leisen and E. S. Dunn. U.S.P. 1,345,605, 6.7.20. Appl., 31.1.19.

THE gases from a series of fire "zones," spaced apart along the length of the tunnel, are caused to pass through the kiln to the stack in the same direction as the materials under treatment.

—W. H. C.

Treating goods with fluids; Method of and apparatus for—H. R. Anders, Assr. to The Roessler and Hasslacher Chemical Co. U.S.P. 1,345,341, 6.7.20. Appl., 1.12.16.

THE materials are placed in separate compartments contained in a rotary vessel, and the treating liquid is circulated through the compartments in succession.—W. H. C.

Distillation-retort and separating tank. G. F. Rendall, Assr. to Carbon Products Co. U.S.P. 1,345,404, 6.7.20. Appl., 13.6.17. Renewed 26.5.20.

A RETORT is heated by gas burners which are supported on a perforated partition dividing the combustion chamber from a chamber for preheating the air supply for the burners.—B. M. V.

Grinding-mill. Crushing machine. W. G. Stevenson. U.S.P. (A) 1,345,714 and (B) 1,345,715, 6.7.20. Appl., 3.6. and 5.10.18.

(A) A pit is bridged by a framework which carries a pan rotating about a vertical axis. The framework also supports grinding rollers which are rotated about their own horizontal axes by the action of grinding, and have a slight vertical movement, but do not rotate about the axis of the mill.
(B) A table with several concentric troughs, each with its set of narrower rollers, is substituted for the pan and single set of grinding rollers. Scoops are provided to transfer the material under treatment from the innermost trough to the next outwards, and so on.—B. M. V.

Lifting and forcing apparatus for solids and semi-solids; Fluid or air direct-pressure—S. H. and C. H. Adams. U.S.P. 1,347,358, 20.7.20. Appl., 28.2.19.

SEE E.P. 136,583 of 1918; J., 1920, 144 A.

Furnaces C. S. E. A. Cossevin. E. P. 125,987, 22.4.19. Conv., 20.4.18.

Furnace control. E. McLean. E. P. 147,295, 17.4.19.

Mixing apparatus. E. Fiorini. E. P. 135,210, 14.11.19. Conv., 25.11.13.

Conveying and screening materials; Apparatus for—R. A. Hadfield and W. T. W. Miller. E. P. 146,600, 7.4.19.

Heat exchangers or regenerators. W. P. Thompson. From Four et Procédés Mathy Soc. Anon. E.P. 146,633, 12.4.19.

Heat exchange apparatus. J. G. McKean and R. F. Jones. E. P. 147,397, 6.12.19.

Exchanging the heat of liquids; Process and apparatus for—F. Duvieusart. E. P. 146,579, 3.4.19.

Heating liquids by waste flue gases. J. O. Frazier. E. P. 148,041, 8.7.19.

Crushing or mixing mills and the like; Means for removing or discharging the contents from —. L. R. Orr. E. P. 147,308, 28.4.19.

Feeding granular or powdered material in measured quantities. The Associated Portland Cement Manufcs. (1900), Ltd., and J. G. Baxter. E. P. 147,341, 2.7.19.

Preventing corrosion. E. P. 146,676. See X.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Banded bituminous coal; Oxidation of the ingredients of —. Studies in the composition of coal. F. V. Tidswell and R. V. Wheeler. Chem. Soc. Trans., 1920, 117, 794—801.

SAMPLES of the four ingredients of the Hamstead Thick Coal (see J., 1919, 452 A, 614 A) have been subjected to the action of air or oxygen to determine their rates of oxidation and relative ignition temperatures. Determinations by a method previously described (J., 1919, 65 A) gave ignition temperatures ranging between 170° and 175° C. for vitrain, clarain, and durain. The rate of absorption of oxygen by each separate constituent was measured at different temperatures, the coal being subjected in each experiment to a preliminary exhaustion at 200° C. Of the three main ingredients of banded bituminous coal, the bright, vitrain and clarain, show greater liability to oxidise and to ignite than the dull durain. The difference between these ingredients is not, however, so great as to warrant any suggestion that the bright portions of Hamstead coal are primarily responsible for its liability to spontaneous combustion. The influence of fusain is uncertain. The authors consider it improbable that fusain has a preponderating influence in determining the actual ignition of the coal after self-heating has begun; but it is conceivable that the rapid absorption of oxygen by fusain at low temperatures may be attended by a sufficient evolution of heat to raise appreciably the temperature of the main mass of the coal, thereby causing the most inflammable ingredient (vitrain) to react more readily with oxygen—W. P.

Coal and the gases resulting from its distillation. L. Vignon. Ann. Chim., 1920, 13, 284—301.

ANILINE as a solvent furnishes a means of differentiating between different grades of coal. The "fat" coals contain a relatively high proportion of material soluble in aniline, whilst "lean" coals contain little soluble matter. The soluble portion of the coal is richer in hydrogen, poorer in ash, and gives a better coke than the insoluble portion. Boiling quinoline also exerts a considerable solvent action on "fat" coals. In the distillation of coal at successively increasing temperatures, the unsaturated hydrocarbons mostly pass over below 600° C., whilst methane and the saturated hydrocarbons are very abundant up to 800° C., their proportion then decreasing rapidly with rise in temperature. Hydrogen is only present in small amounts at 600° C., but is the predominating gas from 800° to 1000° C., diminishing somewhat from 1000° to 1200° C. Carbon monoxide is present in considerable amount above 1000° C. The more oxygen a coal contains, the more carbon monoxide and dioxide does it give on distillation, the relative proportions of these two gases depending on the conditions of distillation.—W. G.

Coal; Briquetting Oklahoma —. J. C. Davis. Chem. and Met. Eng., 1920, 23, 101—102.

SATISFACTORY briquettes were made from soft Oklahoma coal with the following average composition: Moisture, 2.04; ash, 10.93; vol. matter, 35.83; and carbon, 53.23%. The coal was ground to pass a 10-mesh sieve, mixed with an asphalt binder (penetration test 20, m.p. 71° C.) derived from crude petroleum oil, and compressed into briquettes under a pressure of 2000 lb. per sq. in. The finished briquettes had a heat value of 10,513 B.Th.U., this value being increased by about 20% by washing the coal. The best results were obtained when the temperature was maintained between 180° and 200° F. (82°—93° C.) and the moisture 3—5%. —C. A. M.

Coal; Transition from — to coke. E. Sinkinson. Chem. Soc. Trans., 1920, 117, 839—843.

COKE being a conductor of electricity, whilst coal is not, the transition from coal to coke can be followed by recording the change in conductivity which takes place when coal is heated. Preliminary tests with samples of coke briquetted with different proportions of pitch showed that more than 10% of free carbon had to be present in the non-conducting medium, pitch, to form an electrically conducting bridge. A series of cokes was prepared by carbonising samples of the same coal at temperature intervals of 100° up to 900° C. These were briquetted, those obtained from 700° to 900° with the addition of 10% of pitch. The resistance of the briquettes became too great to measure below a carbonising temperature of 500° C. The determinations showed that the critical temperature of the formation of coke lies between 500° and 550° C. This result was confirmed by experiments in which a continuous record of the conductivity was taken whilst the coal was being carbonised. Values obtained for cellulose were close to those obtained for coal, suggesting that the formation of free carbon at this temperature is due to the decomposition of the cellulosic constituents of the coal.—W. P.

Coal substitutes in gas manufacture. P. Schumann. J. Gasbeleucht., 1920, 63, 447—449.

GAS-COAL substitutes available in Bavaria gave the following results when carbonised in gas retorts:—

	% Coke.	Cub.m. gas.	Cal. val. of gas. K.-cal.	Sp. gr. of gas.	% CO, in gas.
Upper Bavarian coal	50	31.32	5,100	0.58	12.13
Lignite	24	30	3,400	0.60	14
Peat	15	30	3,400	0.75	22
Wood	15	30	4,000	0.70	25
Oil shale	60	30	3,800	0.90	32

All were defective in some respects, such as high content of ash or moisture, and high content of oxygen which led to the production of low-grade gas. The large proportion of carbon dioxide and moisture in the gas caused the removal of carbon deposits from the retort material and consequent loss by leakage. Purification was difficult and costly, especially when the sulphur content was excessive. No material yielded a satisfactory coke. The relative costs of production of 1 cub. metre of gas were as follows (the figures in parentheses are costs per unit of heat): From gas coal 1 (1), Upper Bavarian coal 1.201 (1.1), oil shale 1.709 (2.44), wood 3.018 (4.312), peat 2.111 (3.267), lignite 2.432 (2.34).—H. J. H.

Tar; Determination of — in gas. C. H. Stone and N. F. Prince. Gas Age. Gas. J., 1920, 151, 238.

THE gas is led into a wide glass tube which fits into a Gooch crucible containing a thick layer of

asbestos, the connexion being made by means of a rubber band. The bottom of the crucible is fitted into a rubber connexion on a glass tube, the base of which is constricted to pass through a hole in the stopper of an Erlenmeyer flask, whilst a second tube from the flask is connected with the gas meter. Purified dried gas is passed through the crucible for 1½ hrs., and the crucible then left for a few mins. in the desiccator before weighing. It is then again connected with the apparatus, and the gas under examination passed through it at about 2 to 6 cub. ft. per hr. for a period depending on the proportion of tar. Finally the dried purified gas is again conducted through the apparatus for 2 hrs. to remove the moisture, and the crucible containing the tar weighed as before.—C. A. M.

Ignition of gases. Part I. Ignition by the impulsive electrical discharge. Mixtures of methane and air. R. V. Wheeler. Chem. Soc. Trans., 1920, 117, 903—917.

THE relative "ignitibilities" of a series of mixtures of methane and air have been compared by the determination of the "minimum igniting currents" necessary to inflame the mixtures. These are the currents in the primary circuit of an induction coil, which, when broken at a constant rate, induced secondary discharges, at a fixed spark gap, just capable of igniting the mixtures. The gap in these experiments being formed by sharply pointed electrodes, the sparking voltage for a gap of constant width follows, over a wide range, a straight line relationship with the primary current. The steel contacts of the primary circuit breaker must be kept polished, and the sparking points clean, since any oxidation of the former or a speck of dust on the latter result in irregular values being obtained. The neglect of such precautions is the most probable cause of the "stepped" curves obtained by Thornton (Proc. Roy. Soc., 1914, A, 91, 17) and by Paterson and Campbell (Proc. Phys. Soc., 1919, 31, 193). A similar curve to the one obtained by plotting minimum igniting currents with a fixed spark gap against percentage of methane was obtained by plotting minimum spark gaps with constant current through the primary against percentage composition. The characteristics of these curves are the greatly increased difficulty of ignition as the limits of inflammability are approached, particularly on the high limit side; and the position of a range for optimum ease of ignition between 8.2 and 8.5% CH₄. It is suggested that the curve for relative igniting currents is compounded of the relative ignition-temperature curve and the inverse of the curve relating composition of the mixtures with initial speed of propagation of flame therein.—W. P.

Colloidal dispersions ["liquid coal"]. Planson. See I.

Adsorption by charcoal. Lowry and Hulett. See VII.

Fatty acids from paraffin. Löffl. See XII.

Gas calorimeters. Madsen and Herber. See XXIII.

Carbon dioxide in flue gases. Von Haken. See XXIII.

PATENTS.

Coke; Apparatus for producing—. A. M. Smith. U.S.P. 1,346,515, 13.7.20.

THE coking chamber is provided with an extension which forms a quenching chamber, and is provided with an air-tight door at its lower end. A portion of a previously coked mass is severed and held as a plug at the end of the coking chamber, against which plug the new charge of coal to be coked may

rest. The previously coked mass is passed into the quenching chamber, and is separated from the plug by an air space until the mass in the quenching chamber is sufficiently cool.—A. G.

Furnace retorts [for partial carbonisation of coal]. C. H. Smith. E.P. 132,487, 16.4.19. Conv., 9.9.18.

THE retort is provided with a paddle-shaft, journalled in bearings which permit of limited universal movement, e.g., ball and socket members. The bearing has a spherical portion which is forced against a seat by springs or the like. The tubular shell of the retort is made of calorised steel (steel) with a coating of aluminium and is fitted with intermediate metal reinforcing bands contacting with partitions or walls which, with the above bands, define the circumferential heating flues for the retort. The end plates of the retort are formed of steel lined with masonry having a facing of cast iron and are connected with the tubular shell and adapted to support the universal bearings for the paddle-shaft. The end plates are relieved from excessive stress by means of counterweight devices.—A. G.

[Gas] retort mechanism. C. H. Smith. E.P. 147,965, 17.2.19.

A HORIZONTAL retort for coal distillation, of the kind in which a part of rotary conveyors operate in the two parts of a retort of heart-shaped cross-section, is provided with change-speed gearing between the conveyor shafts and the constant-speed motor which drives them. The conveyor shaft is journalled in bearings which are supported on a separate platform so that the shaft is not affected by movements of the end walls of the retort due to expansion or contraction. The end walls are formed so as to provide a recess for the bearing members.—W. F. F.

Gds producers. A. Ritte, and Soc. Franc. de Matériel Agricole et Ind. E.P. 145,807, 21.3.17.

THE producer is provided with several series of supplementary air inlets at different levels above the grate, which is divided into a number of sections, each of which can be agitated from the outside. Several gas outlets from the ash-pit are also provided to ensure an equal draught all over the grate, and a number of feed hoppers at the top of the shaft.—A. G.

Gas producers. H. H. Spicer. From J. F. Wells. E.P. 145,873, 1.4.19.

THE producer is furnished with an inclined grate disposed to support a body of fuel contained in a hopper, the fuel acting as a seal to the chamber. The hopper is flush with one side of the gas chamber and is provided with a movable gate for controlling the supply of fuel to the grate. The producer is intended specially for poor fuels, e.g., "cotton sticks," leaves, chopped straw, etc.—A. G.

Water-gas; Process and apparatus for producing enriched — and products and by-products of such process. J. W. Gibson and R. L. Wyman. E.P. 145,947, 30.5.19.

TAR is atomised and vaporised by admitting it to a current of steam and the current of the resulting mixture then meets the current of water-gas to be enriched. The tar residues are deposited as a by-product. The tar may be vaporised inside or outside the carburation apparatus, and it is exposed to a relatively large surface such as is provided by wooden grids.—A. G.

Coal gas and the like; Apparatus for washing and scrubbing —. F. G. Brockway. E.P. 145,856, 31.3.19.

SUPERPOSED tiers of troughs each have a downwardly directed inlet passage down the middle of

which liquid is caused to descend in the form of a curtain dividing the flow of gas. The two streams of gas pass down on either side of the curtain, and a certain volume is carried down into the main body of the liquid in the trough and is compelled to bubble up through the liquid, whilst the remainder is exposed to the action of the washing liquor running down over sloping plates composed of ribbed or corrugated metal. The gas leaves the troughs through outlet chambers filled with metal or other suitable scrubbing material on to which liquor is sprayed by jets.—A. G.

Ammonium chloride; Method of and apparatus for recovering — [from coal gas]. M. E. Mueller. U.S.P. 1,346,967, 20.7.20. Appl., 21.7.19.

THE gas, before it is cooled and separated from tar, passes along a main through which a quantity of aqueous and tarry liquor from the coolers and tar-extractors is circulated. The temperature in the main is kept above that at which much water vapour condenses from the gas, and quantities of the liquor are withdrawn from time to time, and are replaced by fresh liquor.—L. A. C.

Bituminous binders and processes for making the same. G. A. Henderson. E.P. 148,003, 30.4.19.

FROM 10 to 15% of water is added to natural or artificial bitumen or pitch preheated to above 212° F. (100° C.), and a mixture of petroleum (10% by vol.) and sulphur (10% by weight, of the pitch), previously heated together to about 400° F. (205° C.), is added. Chlorine is introduced into the mass until 3% by weight has been absorbed, and the mass is simultaneously heated and agitated under suction until the water and petroleum have been removed. Alternatively, vegetable matter, such as "denatured" hard wood chips, or mineral matter, such as particles of stone, slag, or the like, containing 10–15% of water, may be added instead of the water alone, or sulphur chloride may be used instead of sulphur and chlorine.—L. A. C.

Gases containing hydrogen sulphide; Methods of treating —. W. G. Leamon. E.P. 120,554, 25.9.18. Conv., 8.11.17.

SEE U.S.P. 1,317,583 of 1919; J., 1919, 891 A.

Coke-ovens and the like; Valve for —. W. E. Roberts, Assr. to Foundation Oven Corp. U.S.P. 1,347,056, 20.7.20. Appl., 17.3.19.

Ammonium sulphate. E.P. 146,456. See VII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Tar-still operation in hardwood distillation plants. L. F. Hawley and N. H. Calderwood, jun. J. Ind. Eng. Chem., 1920, 12, 684–685.

IN recovering the acetic acid remaining in the tar after distillation of the pyroligneous acid, the distillation with steam should be effected at as low a speed as possible, so as to increase the concentration of the distillate. More acetic acid can be obtained with less steam when the tar is kept at a higher temperature, i.e., when a higher pressure is maintained in the closed coils. Live steam is subsequently introduced, while the steam in the closed coils still remains at 50 lb. or higher, until 91 c.c. of distillate is required to neutralise 10 c.c. of $N/2$ alkali. The flow of live steam is then reduced to at least one-half its original speed (e.g., 30–35 galls. per hr.), and the distillation continued until not more than 9.4 c.c. of distillate is required to neutralise the alkali.—C. A. M.

Potash from kelp. Spencer. See VII.

PATENTS.

Hydrocarbons from coal or other carbonaceous matter; Process of and apparatus for extracting —. W. D. Roek. E.P. 145,906, 16.4.19.

PULVERISED coal is blown into the distillation chamber against the under side of a dome arranged near the top and having a diameter slightly less than that of the chamber. Superheated steam, with or without heated air, is blown in near the bottom of the chamber, and passes upwards so that the falling coal dust is subjected to the action of successive zones of increasing temperature. The residue falls into water at the bottom of the tower, from which it is periodically discharged, and the distilled hydrocarbons are drawn off through pipes at the top.—W. F. F.

Solid fuels; Destructive distillation of —. W. Beswick and N. E. Rambush. E.P. 147,311, 1.5.19.

CRUDE producer gas made in an ordinary by-product producer of the Mond type is introduced, without substantial cooling, into an internally heated gas retort. The gas as it leaves the producer contains the proportion of steam to producer gas which gives the best yields of valuable products, and especially ammonia, in the distillation of ordinary fuel in an internally heated retort.

—C. A. M.

Coal, shale, and so forth; Recovery of valuable products from the gases evolved in the destructive distillation of —. The Skinningrove Iron Co., Ltd., and E. Bury. E.P. 147,360, 16.8.19.

SULPHURIC acid is used for absorbing olefines from the gases produced in the destructive distillation of coal, etc., the heat for the absorbing plant being derived from the waste heat of the gases. These may first be freed from ammonia, benzene, etc., and then re-heated by means of the hot gases issuing from the carbonising plant. The sulphur is recovered by the use of hydrogen sulphide purifiers in combination with an apparatus for supplying sulphur dioxide derived from the plant in which alcohol is distilled from ethylsulphuric acid, and from the sulphuric acid concentration and heating plant, and this sulphur is used in the manufacture of sulphuric acid for the olefine scrubbers etc. The several processes are carried out in a closed cycle. (Cf. J., 1920, 94 A.)—C. A. M.

Filaments for electric lamps and the like; Methods of and apparatus for forming —. British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 147,955, 9.5.17.

III.—TAR AND TAR PRODUCTS.

Phenolsulphonic acids; Rapid method for the analysis of —. L. Desvergues. Ann. Chim. Analyt., 1920, 2, 211–214.

FROM 5 to 6 g. of the sample is dissolved in water to 200 c.c., care being taken that the mixture does not become heated. *Total sulphuric acid (T)*. Ten c.c. of the solution is mixed with 25 c.c. of water and 25 c.c. of hydrochloric acid saturated with bromine, boiled for 15 mins., diluted with 200 c.c. of water, cooled, filtered, and the sulphuric acid in the filtrate determined as barium sulphate. *Combined sulphuric acid*. Ten c.c. of the solution is titrated with $N/2$ sodium hydroxide solution, using methyl-orange as indicator; this gives the free sulphuric acid plus one-half the combined sulphuric acid, or S , hence the combined sulphuric acid = $2(T-S)$. *Phenol*. Ten c.c. of the solution is

heated at 50° C. in a closed vessel for 30 mins. with 100 c.c. of water, 50 c.c. of hydrochloric acid (sp. gr. 1.18), and 25 c.c. of standardised potassium bromate-bromide solution; the mixture is then cooled and the excess of bromine determined iodometrically. The amount of free sulphuric acid present is calculated from the results obtained, and the proportion of phenol to combined sulphuric acid gives the quantities of mono- and disulphonic acids.

—W. P. S.

Aniline and ethyl alcohol; Catalysts which promote reaction between —. T. B. Johnson, A. J. Hill, and J. J. Donleavy. *J. Ind. Eng. Chem.*, 1920, 12, 636—644.

THE highest yields of diethylaniline were obtained by heating 100 g. of aniline hydrochloride with 360 g. of alcohol, 10 g. of sodium bromide, 5 g. of cupric chloride, and 10 g. of calcium chloride for 8 hrs. in an autoclave at 175°—180° C. The use of 95% alcohol instead of anhydrous alcohol reduced the yield of diethylaniline by about 7%. In the best results thus obtained the product contained only 5% of monoethylaniline. Zinc chloride is less efficient as a catalyst than calcium chloride, whilst potassium iodide is slightly more effective than sodium bromide. (*Cf. J.C.S.*, Sept.)—C. A. M.

PATENTS.

Sulphonating processes and apparatus for carrying out the same. F. C. Sutton. *E.P.* 147,967, 20.2.19.

AN aromatic hydrocarbon and sulphuric acid are charged through atomisers arranged opposite one another at the upper end of a tower. The tower is fitted with a condenser on top which returns any condensed hydrocarbon to the charging apparatus for the same, and the reaction product flows down baffle plates to the bottom of the tower, whence it is removed.—L. A. C.

Halogenated homologues of benzene; Production of —. L. P. Kyrides, Assr. to General Chemical Co. *U.S.P.* 1,345,373, 6.7.20. *Appl.*, 26.7.17.

TOLUENE is treated with chlorine in the presence of sulphur chloride.—A. J. H.

IV.—COLOURING MATTERS AND DYES.

Indigoid dyestuff; New —, 5-(dioxy-2,4-pyrimidine)-2-indoleindigo. J. Martinet and O. Dornier. *Comptes rend.*, 1920, 171, 184—187.

WHEN the technical melt of indoxyl is added to an acetic acid solution of alloxan heated on a water bath, 5-(dioxy-2,4-pyrimidine)-2-indoleindigo is obtained. It crystallises in opaque violet needles and dyes wool and silk heliotrope. Its sulphonic derivative dyes wool and silk violet. It sublimes above 300° C., giving violet-red needles. (*Cf. J.C.S.*, Sept.)—W. G.

Diazo compounds; Volumetric determination of — by reduction. E. Knecht and L. Thompson. *J. Soc. Dyers and Col.*, 1920, 36, 215—219.

BENZENEDIAZONIUM chloride may be titrated with titanium trichloride solution, using H acid as external indicator:



The titration must be made in dilute hydrochloric acid solution, since the reaction does not take place in the presence of moderately concentrated hydrochloric acid. The determination may also be made by adding an excess of titanium trichloride solution and titrating the excess with standardised Acid Green solution in the presence of sodium tartrate. Sodium hydrosulphite solution reduces benzenediazonium chloride, in hydrochloric acid solution,

quantitatively to phenylhydrazine hydrochloride; in this case the reduction is effected at 0° C. with an excess of the reagent and the excess then titrated with Acid Green solution. In cold alkaline solution *p*-nitro-sodiazobenzene is reduced directly to *p*-nitrophenylhydrazine by sodium hydrosulphite solution. Rosinduline or Safranin is used as internal indicator; the latter is not reduced until the reduction of the diazo group is complete but is reduced before the nitro group is attacked. In slightly alkaline solution containing sodium tartrate, *p*-nitro-sodiazobenzene is reduced by an excess of titanium trichloride to *p*-phenylenediamine and ammonia; the excess of the reducing agent may be titrated with Crystal Scarlet solution which acts as its own indicator.—W. P. S.

Henna (Lawsonia inermis). Chemical constitution of lawson. II. G. Tommasi. *Gaz. Chim. Ital.*, 1920, 50, I., 263—272.

LAWSONE, $\text{C}_{10}\text{H}_8\text{O}_3$, the colouring matter contained in henna leaves, is fixed well by wool and silk and rapidly and tenaciously by the skin. This compound is shown to be a hydroxynaphthoquinone, and is probably identical with 2-hydroxy-1,4-naphthoquinone. Its reactions with various reagents and its chemical transformations are described. (*Cf. J.C.S.*, Sept.)—T. H. P.

Diazo compounds. Korczynski and others. *See XX.*

Fluoremetry. Desha. *See XXIII.*

Methyl red. Desvergnès. *See XXIII.*

PATENTS.

Sulphur colour and process of manufacturing the same from (A) aloe resin and (B) aloes. A. T. Appelbaum, Assr. to The Import and By-Products Co. *U.S.P.* (A) 1,346,153 and (B) 1,346,154, 13.7.20. *Appl.*, 10.6.18 and 5.2.19.

(A) ALOE resin is heated with sulphur and a solution of sodium sulphide. (B) Aloes, sulphur, and caustic soda liquor are heated under pressure, the product dissolved in water, and the dye subsequently precipitated.—A. J. H.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cellulose; Adsorption capacity of —. *Electrometric micro-analysis of chlorine*. P. Rona and L. Michaelis. *Biochem. Zeits.*, 1920, 103, 19—30.

CELLULOSE does not appreciably adsorb surface-active non-electrolytes (heptyl alcohol, secondary octyl alcohol). Only in the case of extremely surface-active substances (*n*-octyl alcohol) can very slight adsorption be established. The more marked adsorption of electrolytes (dyes) by cellulose is due to the mineral content of the cellulose, principally calcium silicate, which is present in small quantities, sufficient to account for the adsorption observed, even in so-called ash-free filter paper. The estimation of chlorine by the electrometric method with a calomel electrode is described and discussed.—S. S. Z.

Sulphite cellulose waste liquor; Utilisation of —. R. H. McKee. *Pulp and Paper Mag.*, 1920, 18, 715—718.

A NEW method is described in which the waste sulphite liquor is freed from most of its sulphur dioxide by the action of a counter-current of steam in a desulphurising column, 16 ft. high, and is then evaporated to about half its vol. in an evaporator of the thermo-compressor type. It is then cooled to about 29° C. by means of a counter current of water, and pitched with yeast, together with hydrolysed

yeast, ammonium sulphate, and calcium acid phosphate, while a slow current of air is introduced into the bottom of the fermentation tank. After 60–72 hrs. the alcohol is distilled, preferably in a continuous still, and redistilled in an intermittent still on to the upper plates of which is run a dilute solution of soda ash to neutralise the free sulphur dioxide. The yield of 95% alcohol varies from 0.55 to 1.35% of the vol. of waste liquor; av. 1.0%, at a total cost of 28.4 cents per U.S. gall. The residual sulphite liquor from the stills yields a dry residue suitable for fuel. A typical liquor yielded a residue with heat value of 7950 B.Th.U. per lb., ash 13.9; sulphur in residue 5.4; and sulphur in ash, 1.9%. Each ton of pulp yields about 2772 lb. of a mobile pitch containing 50% of solids.—C. A. M.

PATENTS.

Flax and other fibres; Retting and drying of —. The Portadown Weaving Co., and T. J. Greeves. E.P. 146,702, 12.6.19.

FLAX, jute, and hemp are retted by steeping in a tank having a perforated bottom. After running off the liquid the fibres are dried by drawing air through the tank.—A. J. H.

Artificial silk; Method of producing — for textile industrial purposes. Deutsche Gasglühlicht A.-G. (Auerger). E.P. 116,103, 22.5.18. Conv., 22.5.17.

ARTIFICIAL silk is permanently loaded by treatment with the by-products of thorium manufacture, which contain cerium, lanthanum, and didymium, in the form of a solution of 40°–50° B. (sp. gr. 1.38–1.530), drying and treating with alkaline gases as ammonia and methylamine. The fibre becomes swollen. The loaded material may be bleached with a 2–3% solution of sodium hydrosulphite. Dyeing should precede the loading treatment.—A. J. H.

Paper; Waterproof — and process of producing the same. A. L. Clapp. U.S.P. 1,345,476, 6.7.20. Appl., 11.7.18.

FIBROUS paper pulp containing a soluble soap is treated with calcium hydroxide to precipitate the calcium soap, and aluminium resinate is then precipitated in the product.—A. J. H.

Textile fibrous material; Production of —. M. Hofsté. U.S.P. 1,345,776, 6.7.20. Appl., 2.1.20. SEE E.P. 136,804 of 1919; J., 1920, 541 A.

Drying textile and other fabrics; Apparatus for —. J. W. Chadwick. E.P. 148,037, 23.6.19.

Paper-making machines. J. Thompson. E.P. 145,845, 28.3.19.

Straining apparatus for use in the manufacture of paper and wood pulp. C. Martin, and E. Lloyd, Ltd. E.P. 147,356, 30.7.19.

Paper pulp; Tub-beaters for —. W. J. Mellersh-Jackson. From Griley-Unkle Engineering Co. E.P. 148,107, 1.4.20.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Mergerisation [of cotton-viscose silk unions]. S. M. Jones, Assr. to Arnold Print Works. U.S.P. (A) 1,346,802 and (B) 1,346,803, 13.7.20. Appl., 12.5.20.

COTTON fabrics containing viscose silk are mergerised without damage when (A) glycerin or (B) monoacetin is added to the caustic soda solution. (Cf. U.S.P. 1,343,138–9; J., 1920, 542 A.)—A. J. H.

Yarn in hank form; Machine for treating —. J. S. and S. S. Lord. U.S.P. 1,346,833, 20.7.20. Appl., 15.12.19.

SEE E.P. 136,666 of 1918; J., 1920, 186 A.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Phosphoric acid; Production of — by smelting phosphate rock in a fuel-fed furnace. W. H. Waggaman and T. B. Turley. J. Ind. Eng. Chem., 1920, 12, 646–650.

MIXTURES of calcium phosphate, quartz flour, and carbon yield nearly the whole of the phosphorus as P_2O_5 , when heated in an open or closed crucible in an oil-furnace, provided that the temperature is kept at about 1500° C., and that reducing conditions are maintained until a fusible slag has been formed. The addition of a small amount of alumina is advantageous, since it lowers the m.p. of both acid and basic slags. The proportion of silica required varies with the composition of the phosphate rock. In experiments on a large scale the material was used in the form of small briquettes prepared from a mixture of 20 pts. of finely-ground natural phosphate (containing 20–25% clay-like constituents), 16.6–22 pts. of silica, and 4.8–5.6 pts. of coke, with 10% water. The flames and gases of the furnace were made to pass through a central shaft, and so brought into direct contact with the material. Copious fumes containing up to 93.55% of the P_2O_5 were liberated, whilst the residual briquettes contained from 1.63 to 11.52% P_2O_5 , according to their degree of fusion.—C. A. M.

Potash from kelp. II. Distillation of kelp at low temperatures. G. C. Spencer. J. Ind. Eng. Chem., 1920, 12, 682–684.

THE distillation of dried kelp in an oil-jacketed wood-distillation retort at temperatures not exceeding 320° C., yielded a residual charcoal which had not been sufficiently heated either to form a suitable filtering medium or for the extraction of potash. Hence kelp should be distilled at a much higher temperature.—C. A. M.

Ammonia; Iodic acid as a characteristic micro-chemical reagent for gaseous —. G. Denigès. Comptes rend., 1920, 171, 177–179.

WHEN a drop of a 10% solution of iodic acid is exposed to an atmosphere containing ammonia it becomes covered with a crystalline film of ammonium iodate. These crystals may readily be identified under a microscope, and the reaction is not given by volatile amines under these conditions. For the detection of traces of ammonium salts in a solution, the latter is first evaporated to a small bulk and then the ammonia is liberated by the action of magnesia and detected as above.—W. G.

Evaporation [of solutions of salts]; Spontaneous —. H. B. Weiser and E. E. Porter. J. Phys. Chem., 1920, 24, 333–341.

SOLUTIONS of copper sulphate, potassium ferrocyanide, and potassium tartrate all evaporate more slowly than pure water. The rate of evaporation furnishes an approximate method of estimating the lowering of the vapour pressure of solutions. Chromic anhydride in aqueous solution exists mainly as dichromic acid.—J. F. S.

Zinc oxide, phosphorus pentoxide, and water; The system — at 25° C. and 37° C. N. E. Eberly, C. V. Gross, and W. S. Crowell. J. Amer. Chem. Soc., 1920, 42, 1433–1439.

THE tetrahydrate of normal zinc phosphate, $Zn_3(PO_4)_2 \cdot 4H_2O$, is prepared by saturating a solu-

tion of phosphoric acid (50% P_2O_5) with zinc oxide at the boiling point, cooling in ice-cold water and stirring violently while 10 vols. of ice-cold water are added, filtering and warming in a water bath, when shining orthorhombic plates of the tetrahydrate separate. At $25^\circ C$. the compounds $Zn_3(PO_4)_2 \cdot 4H_2O$, $ZnHPO_4 \cdot 3H_2O$, and $Zn(H_2PO_4)_2 \cdot 2H_2O$ separate from the system zinc oxide, phosphorus pentoxide, and water, whilst at $37^\circ C$. the additional compound $ZnHPO_4 \cdot H_2O$ is obtained.—J. F. S.

Manganese salts; Simplification of Caron and Raquet's reaction for —. G. Denigès. *Ann. Chim. Analyt.*, 1920, 2, 215–216. (*Cf. J.*, 1919, 574 A, 662 A.)

FIVE c.c. of the manganese salt solution is treated with a few drops of sodium hydroxide solution and shaken until a brown coloration is obtained; saturated oxalic acid solution is then added drop by drop. The brown coloration disappears and is replaced by the red coloration characteristic of manganese salts.—W. P. S.

Zirconium; Investigations on —. J. W. Marden and M. N. Rich. *J. Ind. Eng. Chem.*, 1920, 12, 651–656.

Preparation of zirconium oxide:—Good yields were obtained from zircite by fusion with sodium carbonate and hydroxide, lixiviation with hot water, digestion of the residue with hydrochloric acid (1:1), neutralisation of the filtrate with sodium carbonate, and precipitation of the zirconia by means of sulphur dioxide: by fusion with nitre cake, extraction with hot water, and precipitation as zirconium phosphate; and by decomposition with sulphuric acid, extraction with cold water, neutralisation of the filtrate with sodium carbonate, and ignition of the precipitated basic zirconium sulphate: this yields 99.8% ZrO_2 . *Pure potassium zirconium fluoride* is prepared by dissolving zirconium oxide in hydrofluoric acid, treating the solution with potassium carbonate in sufficient quantity to form K_2ZrF_6 , and recrystallising the salt three times. When fused in a graphite vessel at $900^\circ C$. and treated with $1\frac{1}{2}$ times its weight of aluminium it yields an alloy, which when freed from excess of aluminium by treatment with sodium hydroxide solution, and then with hydrochloric acid (1:1), consists of silvery-white crystals containing 30% Al and 68% Zr, with about 1% Fe, Si, Ti, etc. This aluminide (Zr_3Al_4) may be prepared in impure condition by fusing zircite with sodium fluoride and then adding aluminium. *Metallic zirconium* was prepared in its various forms, the physical and chemical properties of which are described. Coherent metal obtained by aluminothermic reduction contained: Zr, 99.40–99.50; Ti, 0.45–0.40; Fe, 0.01–0.02; and Al and Si (diff.) 0.14–0.08%. (*See J.*, 1910, 218, 358; 1913, 197.) Methods for determining zirconium and separating it from other metals are also discussed. (*Cf. J.C.S.*, Sept.)—C. A. M.

Mercuric oxycyanide. A. J. Jones. *Pharm. J.*, 1920, 105, 87–89.

THE composition of commercial mercuric oxycyanide is very variable and the pure salt is never supplied. The pure salt is stated to be dangerous to handle, and explosions have occurred during its manufacture. It was prepared by grinding 40 g. of mercuric cyanide and 30 g. of yellow mercuric oxide with 15 c.c. of water and, after 15 mins., adding 0.5 c.c. of 20% sodium hydroxide solution. Next day the mass was diluted with 200 c.c. of water, acidified slightly with acetic acid and added to 700 c.c. of boiling water containing 20 g. of mercuric cyanide. The solution was filtered, the filtrate

cooled, the separated crystals collected, washed with a small quantity of cold water and dried over sulphuric acid.—W. P. S.

Hydrogen peroxide; Preservation of —. H. R. Jensen. *Pharm. J.*, 1920, 105, 87.

HYDROGEN peroxide may be preserved by the addition of acetanilide (0.002%) and hydrochloric acid (2%) or phosphoric acid (0.1%); boric acid and amino compounds in general also act as preservatives.—W. P. S.

Finely divided gases; Action of extremely —. [*Reduction of carbon dioxide by hydrogen.*] C. Zenghelis. *Comptes rend.*, 1920, 171, 167–170.

THE reduction of carbon dioxide in aqueous solution by very finely divided hydrogen (*J.*, 1920, 363 A) steadily diminishes in velocity with the time. It takes place very slowly in the dark, but is markedly accelerated by sunlight and still more so by ultraviolet rays. In all cases the formaldehyde first produced undergoes polymerisation.—W. G.

Acetic acid; Distribution of — between water and charcoal. A. Pickles. *Chem. News*, 1920, 121, 49.

WOOD charcoal removes acetic acid from its solutions, rapidly at first, but several hours are required for equilibrium to be reached. The concentration of the acid in the charcoal varies with the nature and condition of the charcoal. Animal charcoal, which removes the acid almost entirely by adsorption, was the most efficient agent, and fruit-stone dust charcoal the best of the vegetable charcoals tried. (*Cf. J.C.S.*, Sept.)—C. A. M.

Charcoal; Adsorption by —. I. Relation of service time to adsorption and absorption. II. Relation of oxygen to charcoal. H. H. Lowry and G. A. Hulett. *J. Amer. Chem. Soc.*, 1920, 42, 1393–1419.

THERE is no relationship between the length of time charcoal has been in use and the adsorption of gases such as nitrogen and carbon dioxide. The adsorptive power of charcoal is changed as much as 100% by changes in the mode of preparation. Water is not adsorbed by charcoal, but absorbed, that is it is held by capillary action. The diameter of the capillaries of charcoal varies from 2.8×10^{-7} cm. to 9.2×10^{-7} cm., and the surface varies from 160 sq. m. to 436 sq. m. per g. The variation in surface is not always accompanied by a corresponding variation in adsorptive capacity. The adsorption of oxygen by charcoal is anomalous owing to a surface combination of the oxygen with the carbon at ordinary temperatures. The product is a solid stable oxide of carbon, which on heating breaks up into carbon monoxide and carbon dioxide and is to be regarded as an intermediate compound in the combustion of charcoal. (*See Armstrong, J.*, 1905, 473.)—J. F. S. *Calcium cyanamide waste product.* Baumann. *See IX.*

Anode corrosion of lead. Brown and others. *See XI.*

Diphenylamine-sulphuric acid reagent. Haun. *See XXIII.*

Hydrocyanic acid. Morris. *See XXIII.*

PATENTS.

(A, B) *Ammonia; Process of oxidising — to nitric acid.* (C) *Process of catalytically combining gases.* A. Henwood. U.S.P. (A) 1,347,158, (B) 1,347,159, and (C) 1,347,160, 20.7.20. Appl., (A) 8.7.18, (B and C) 9.1.19.

(A) A mixture of gases is caused to react by passage through a catalyst in the form of a diaphragm so thin that practically the whole of the material is active. (B) In the oxidation of ammonia to nitric acid the temperature is reduced by non-automatic

means, and if the minimum permissible temperature is approached heat is supplied automatically. (c) A mixture of gases is passed over a catalyst supported on a carrier provided with flexible supports, to allow for expansion and contraction under temperature variation.—L. A. C.

Ammonium sulphate; Production of —. C. W. Bailey, H. S. Denny, W. H. H. Norris, and H. E. F. Gould-Adams. E.P. 146,516, 5.1.18.

NITRE cake, sodium sulphate, ammonium sulphate, and water are brought together under such conditions that a quantity of anhydrous sodium sulphate is precipitated equivalent to the sodium present in the nitre cake, and, after removal of the precipitate, the liquor is used instead of sulphuric acid to absorb ammonia, e.g., from Mond gas, coke-oven gas, or the like. The proportions of nitre cake, sodium sulphate, and ammonium sulphate in the original mixture may either be so adjusted that after absorption of the ammonia and subsequent evaporation of a portion of the water, ammonium-sodium sulphate crystallises out, and after removal of the crystals and further evaporation of a portion of the remaining water, pure ammonium sulphate separates on cooling, or that after absorption of the ammonia and evaporation of a portion of the water pure ammonium sulphate crystallises out, in which case the liquor separated from the crystals is suitable for the treatment of a fresh supply of nitre cake.—L. A. C.

Ammonium salts; Purification of —. V. Bredlik. U.S.P. 1,346,106, 13.7.20. Appl., 23.8.19.

AMMONIUM salts containing free acid are neutralised with crude ammonia which has been freed from organic impurities.—L. A. C.

Alumina; Method of producing —. Norsk Hydro-Elektrisk Kvaestofaktieselskab. E.P. 134,531, 25.10.19. Conv., 26.10.18.

ALUMINA is precipitated in a granular form, which can be readily filtered and washed, by treating a solution such as that obtained by dissolving labradorite or the like in acids with ammonia above 100° C. and under a pressure of 10–15 atm. Ammonia is regenerated from the liquor after separation of the precipitated alumina by treatment with finely ground labradorite, under a pressure slightly higher than that in the precipitation vessel, and the ammonia gas generated can thus be passed directly into the precipitation vessel for treating a fresh supply of solution.—L. A. C.

Cuprous oxide; Process for the manufacture and use of —. E. C. R. Marks. From Hüttenwerk Niederschöneweide A.-G. vorm. J. F. Ginsberg. E.P. 147,958, 1.2.18.

CUPROUS oxide in a form especially suitable for the manufacture of copper compounds, such as copper sulphate, cuprammonium salts, etc., is prepared from copper which has been refined by blowing, to about 99% purity, by continuing the oxidation until the whole of the liquid copper has been converted into a thin liquid mass of cuprous oxide. When cold the product is ground to a fine powder.—L. A. C.

Hypochlorites; Electrolytic apparatus for the direct production of —. P. Pestalozza. E.P. 148,095, 19.12.19.

A SOLUTION of a metallic chloride is fed into one end of a vat divided into a number of cells by transverse partitions provided with openings so arranged that the liquor passes through the series in a zig-zag course. Each cell is provided with a vertical anode plate of platinum, two vertical cathode plates of carbon, graphite, or the like, and glass cooling

coils. The vat and electrode supports are formed by moulding under pressure a mixture of 80–85% of Portland cement and 20–15% of asbestos fibres, to which the requisite amount of water has been added; after drying, the vat is rendered non-porous and inert to chlorine by immersion for 1 hr. in a bath of paraffin and a neutral oil at 200° C.

—L. A. C.

Alkali-metal hydrates [hydroxides]; Process of producing —. H. W. Charlton, Assr. to American Potash Corp. U.S.P. 1,346,002, 6.7.20. Appl., 12.10.16.

MINERAL material containing an alkali metal is heated and digested with a mixture containing an alkali hydroxide and an alkaline-earth hydroxide under a pressure and for a time sufficient to produce the desired alkali hydroxide, which is then extracted from the mixture.—L. A. C.

Pyrophosphates; Process of producing —. H. A. Webster. U.S.P. 1,346,148, 13.7.20. Appl., 24.9.19.

MONOCALCIUM phosphate is allowed to react with a salt containing a stronger basic ion than the calcium ion and a weaker acid ion than that of pyrophosphoric acid.—A. J. H.

Asbestos; Process of bleaching —. C. L. Hill. U.S.P. 1,346,316, 13.7.20. Appl., 1.5.20.

Moist asbestos fibres are treated with an acid gas, and the material is then washed with water to remove soluble salts.—L. A. C.

Silicates; Apparatus for treating —. W. Glaeser, Assr. to Potash Extraction Corp. U.S.P. 1,347,024, 20.7.20. Appl., 19.9.17.

A STATIONARY furnace chamber is connected by means of a rotary tubular shell with a stationary stack chamber. A kiln mounted in the shell extends through the chambers and projects through the front wall of the furnace chamber into a receiver.—J. W. D.

Hydrogen; Process of and apparatus for producing —. L. S. Abbott, Assr. to The Improved Equipment Co. U.S.P. 1,345,905, 6.7.20. Appl., 6.11.15.

AN apparatus for the manufacture of hydrogen by the process in which there are separate heating, steaming, and reducing stages contains a number of units each of which is so constructed that the three stages can be performed therein; the number of units is adjusted according to the times of operation of the different stages, so that by simultaneous operation of the stages in different units a continuous supply of hydrogen is obtained.—L. A. C.

Cyanogen and ammonia; Method of producing —. R. L. Gould. From Balfour-Guthrie Investment Co. E.P. 145,824, 25.3.19.

SEE U.S.P. 1,306,862 of 1919; J., 1919, 576 A.

Alkali-metal minerals; Decomposition of —. E. Bergve, Assr. to Norsk Hydro-Elektrisk Kvaestofaktieselskab. U.S.P. 1,346,365, 13.7.20. Appl., 30.4.19.

SEE E.P. 127,566 of 1919; J., 1920, 541 A.

Separating the constituents of air or other gaseous mixtures. Ges. f. Linde's Eismaschinen A.-G. E.P. 147,956, 11.7.17. Addn. to 24,735 of 1914.

SEE G.P. 319,992 of 1916; J., 1920, 545 A.

Absorbing gases. E.P. 124,761. See I.

Ammonium chloride. U.S.P. 1,346,967. See IIA.

VIII.—GLASS; CERAMICS.

Glass-pot mixtures; Porcelain —. D. H. Fuller. J. Amer. Ceram. Soc., 1920, 3, 569—570.

SEVEN glass-pot bodies of a porcellaneous character were made from felspar, ball clay, kaolin, and grog (white ware bisque) of various grades, and the water content in plastic state, shrinkage on drying, modulus of rupture in dry and fired states, contraction under a load of 50 lb. per sq. in. at 1350° C., and refractoriness determined. The water-content and drying shrinkage were similar in all the mixtures; the transverse strength in both dry and fired states was greatest with little 10–20-mesh grog and a larger proportion of 20–80-mesh grog. These bodies withstood load conditions at working temperatures satisfactorily. The most rapid rate of vitrification occurred when the bodies were fired between cones 11 and 13; coarser grog retarded vitrification and finer grog promoted it. The effect of the addition of felspar in promoting vitrification was greater the finer the grog, but more than 8% of felspar could not be used without impairing the rigidity of the pots at 1450° C. The most satisfactory body appears to be felspar 6·5, ball clay 22·0, kaolin 23·5, 10–20-mesh grog 16·8, 20–40-mesh grog 19·2, 40–80-mesh grog 9·6, and 80-mesh to dust grog 2·4%.—A. B. S.

Glass pots; Lining for —. S. R. Scholes. J. Amer. Ceram. Soc., 1920, 3, 498—500.

A NON-POROUS lining for glass pots is made by adding 5–10% of felspar to the mixture ordinarily used for the pots, lining the glass-pot to a thickness of about $\frac{1}{2}$ in. up to the metal line and to a gradually diminishing thickness for a further 8 in. with the felspathic mixture, and then firing in the usual manner. Owing to the density and porcellaneous structure of the lining, the attack of the glass is reduced to a minimum, the useful life of such lined pots when used for making potash-lead glass being two or three times that of ordinary pots.—A. B. S.

Glass to metal joints. E. C. McKelvy and C. S. Taylor. J. Amer. Chem. Soc., 1920, 42, 1364—1377.

SOLDERED joints between metal and glass tubes are best made by roughening 15–20 mm. of the glass tube, which must fit easily into the metal tube, and then coating it with platinum by covering it with a suspension of platinum chloride in lavender oil and burning off the volatile matter in a Bunsen flame. The inside of the metal tube is coated with tin, solder, or lead, heated to melt the coating, the platinised end of the glass tube then inserted, and the whole allowed to cool slowly. Fused joints between metal and glass are made by coating the inside of the metal tube with Kraus flux (equal weights of zinc oxide, borax, and powdered soda-glass fused together), the glass tube is flared out to a flat ring of a width equal to three times the thickness of the glass wall and whilst hot pressed into the flux-lined metal tube. Fusion is completed by heating the outside of the metal tube in such a way that the glass tube is kept cool enough to prevent deformation.—J. F. S.

Refractories; Possible explanation of failure under load at high temperatures as displayed by fire-clay —. A. S. Watts. J. Amer. Ceram. Soc., 1920, 3, 448—459.

FIRECLAY refractories may fail at 1300° C. when under a sufficient pressure even when the proportion of fluxes is extremely low and when the $\text{Al}_2\text{O}_3:\text{SiO}_2$ ratio resembles that of pure kaolinite. Refractory articles with a high content of ball clay do not show this weakening and as they do not develop sillimanite, it is suggested that the production of

sillimanite when fireclays are heated to 1300° C. is an important cause of weakening at that temperature.—A. B. S.

Firebricks; Behaviour of — in malleable furnace bungs. H. G. Schurecht. J. Amer. Ceram. Soc., 1920, 3, 556—559.

COMPLETE 7-ft. arches each containing 40 bung-bricks arranged as headers, were built in over the firebox of a 40-ton malleable iron furnace and heated repeatedly. Of six different kinds of bricks examined, three, made by the soft mud process, withstood 18 heatings; these contained over 40% Al_2O_3 and had porosity about 15%, bulk sp. gr. about 2·0, refractoriness corresponding to cone 32, deformation when heated at 1350° C. under a load of 25 lb. per sq. in. 6–15%. Two other bricks of similar composition, refractoriness, and porosity but only 1·9 bulk sp. gr. withstood 12 and 14 heatings respectively. A dry-press brick containing 29·8% Al_2O_3 , sp. gr. 2·03, porosity 13·4%, and deformation under the load-test 11·8%, only withstood three heatings.—A. B. S.

[Pottery] *sagger mixtures; A high temperature transverse-strength test as a method of testing* —. S. C. Linbarger and C. F. Geiger. J. Amer. Ceram. Soc., 1920, 3, 543—555.

TEST-PIECES, measuring $8 \times 2\frac{1}{2} \times 2$ in., of various mixtures to be used in the manufacture of saggars, fired to cone 12 in commercial sagger kilns, and ground so as to have two flat and parallel faces, were supported on two carbofrax prisms with a 6 in. span, heated to 1300° C. for 45 mins., and the transverse strength determined at that temperature. The load was transmitted from a weighted lever through a carbofrax cylinder resting on a carbofrax prism, the effective load on the test-piece being increased at the rate of 6 lb. per min. The modulus of rupture,

$M = \frac{3Pl}{2bd^2}$, in which P is the breaking load in lb., l the length of span in inches, and b and d the width and depth of the test-piece in inches. During the test the piece must be protected from direct contact with the flame. When cold some sagger mixtures had moduli as low as 450, others as high as 3000 lb. per sq. in. At 1300° C. the weakest mixture tested had a modulus of 70 lb. per sq. in. and the strongest 600 lb. per sq. in. The tests already made suggest that mixtures with a modulus of over 350 lb. per sq. in. at 1300° C. are suitable for saggars.—A. B. S.

Terra-cotta glazes and colours; Composition of kiln gases and their effect on —. F. B. Ortman. J. Amer. Ceram. Soc., 1920, 3, 476—488.

THERE is an appreciable amount of sulphur trioxide in the gases passing through kilns in which terra-cotta is burned, but in the early stages of heating it is rapidly removed by the excessive ventilation. The fused glaze is not readily susceptible to the action of sulphur trioxide on its surface. The danger of sulphuring is greatest between "red heat" and the fusion of the glaze, because the sulphur dioxide produced on heating from the pyrites in the clay encounters a moist atmosphere, and forms sulphuric acid, which may cause any or all of the following defects:—Sulphur dioxide may be displaced by silica from combined sulphuric acid during the fusion of the glaze and the escaping gas may form blisters. The sulphuric acid may displace boric acid in the glaze. The sulphuric acid may react with colouring oxides and other fluxes and prevent the development of the desired colour and texture. The occurrence of these defects may be lessened by providing the maximum ventilation consistent with satisfactory heating of the kiln; by firing so as to avoid choking the flues with large quantities of gas; by maintaining an adequate supply of secondary

air in the fire-boxes, and by maintaining an adequate draught and oxidising conditions throughout, particularly after the trial holes have been closed.

—A. B. S.

Fish-scaling [in enamelled ware]. J. B. Shaw. J. Amer. Ceram. Soc., 1920, 3, 489—497.

The defect known as "fish-scaling" on enamelled sheet steel consists of small pits formed by a speck of the ground-coat enamel "jumping off." The pits may be very small and numerous, or they may be $\frac{1}{8}$ — $\frac{1}{4}$ in. diam. and less numerous. The defect is commonest on heavy gauge steel but must be distinguished from blistering (which is due to impurities rolled into the steel) and from pitting due to rusting of the metal. In the author's opinion, fish-scaling is due to several causes, the chief of which are to be found in the preparation, application, and burning of the enamel and particularly in the use of enamels with too large a proportion of finely ground material.—A. B. S.

Enamels; Relative action of acids on —. W. J. Amer. Ceram. Soc., 1920, 3, 560—567.

In further work by the Sub-Committee on Enamels of the Standards Committee of the Amer. Ceram. Soc. (cf. Poste, J., 1919, 287 A), various enamelled vessels were filled with a solution containing 6 oz. of citric acid in 1 quart of water (=15.73%) and kept for 23 hrs. at 12°—20° C. The liquid was then transferred to a measuring flask, the vessel rinsed with water which was added to the liquid, and the whole diluted to 500 c.c. Two portions of 100 c.c. each were withdrawn, evaporated to dryness in weighed platinum dishes, and ignited. The gain in weight in each case, less the ash in the citric acid, indicates the amount of enamel removed by the acid. If a 0.3% solution of malachite-green is poured into the vessels which have been treated with acid, and after five minutes the solution is poured out and the dishes rinsed with water, the resulting discoloration of the etched portion appears to be approximately proportional to the activity of the acid used.—A. B. S.

PATENTS.

Glass-furnace. J. R. Seohy. U.S.P. 1,347,181, 20.7.20. Appl., 28.11.19.

A GLASS furnace is provided with floaters and adjacent pockets for collecting the scum held back by the floaters.—H. S. H.

Brick; Light-weight refractory —. R. C. Purdy and M. F. Beecher, Assrs. to Norton Co. U.S.P. 1,345,632, 6.7.20. Appl., 1.11.18.

Porous bricks are made from burned refractory lignite clay.—L. A. C.

Resistance material. H. T. Bellamy and B. T. Sweely, Assrs. to Western Electric Co. U.S.P. 1,346,874, 20.7.20. Appl., 16.11.18.

A compound having a high electrical resistance at high temperatures, comprises talc, silica sand, ball clay, and a glass binder.—H. S. H.

Refractory composition and process of making same. A. T. Hineley, Assr. to National Carbon Co. U.S.P. 1,346,959, 20.7.20. Appl., 9.10.19.

A REFRACTORY composition comprises fully shrunk, graded carbon particles in mutual contact, and a ceramic bonding material proportioned to fill the interstitial spaces.—H. S. H.

Glass; Process and apparatus for separating — into mould charges. W. J. Mellersh-Jackson. From Hartford-Fairmont Co. E.P. 146,617, 10.4.19.

IX.—BUILDING MATERIALS.

Calcium cyanamide; Preparation of Portland cement from a waste product from —. J. Baumann. Chem.-Zeit., 1920, 44, 562.

In the decomposition of calcium cyanamide into calcium carbonate and ammonia a waste sludge is left in the autoclave. When dried at 100° C. this is a black powder of variable composition. One sample contained: SiO₂, 3.55; SO₃, 0.98; Fe₂O₃, and Al₂O₃, 2.05; CaO, 43.85; MgO, trace; free carbon, 15.00; and loss on ignition, 34.67%. When mixed with the calculated proportion of dried clay and heated in a furnace to a white heat it yielded a cement with the properties and approximate composition of normal Portland cement.—C. A. M.

PATENTS.

Cement; Method of making —. F. L. Smidth & Co. E.P. 137,534, 8.1.20. Conv., 8.1.19.

MOLER, silicious earth, diatomite, or infusorial earth, previously burnt to about 900° C., is mixed and ground with Portland cement. The amount of moler which can be added advantageously is doubled by burning, while a mixture containing 25% by weight of burnt moler (referred to the cement) has a strength more than 50% greater than a mixture composed of the same proportions of raw, dried moler and cement.—H. S. H.

Wood; Fireproofing of —. H. F. Weiss, Assr. to C. F. Burgess Laboratories. U.S.P. 1,346,287, 13.7.20. Appl., 17.8.18.

WOOD impregnated with a solution of barium sulphide is treated with gaseous carbon dioxide, whereby hydrogen sulphide is expelled and barium carbonate is formed within the body of the wood.—L. A. C.

Cement, cement-concrete, reinforced concrete, lime trass mortar, natural stone or bricks, and the like; Process for forming a covering and hardening coating on, or impregnating articles or plastering of —. L. A. and A. J. Sanders. E.P. 145,806, 26.4.16. Addn. to 102,042.

ARTICLES or plastering made according to the original patent (J., 1919, 863 A) are heated, wholly or locally.

Woods; Composition for hardening and preserving impregnable soft —. A. Lambert. U.S.P. 1,346,830, 20.7.20. Appl., 4.1.19.

SEE E.P. 139,974 of 1919; J., 1920, 336 A.

Bricks, tiles, clinker, and the like; [Plant for] manufacture of —. Burners for firing brick kilns, furnaces, or the like. H. Atkins and J. A. Colquhoun. E.P. 147,239 and 147,240, 16.11.18.

Bituminous binders. E.P. 148,003. See IIA.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Electrolytic iron; Annealing of —. J. Cournot. Comptes rend., 1920, 171, 170—171.

HEATING at 950° C. for 2 hrs. or at 1050° C. for 1 hr. is necessary to obtain complete annealing of electrolytic iron. At 850° C. annealing is not complete even after 4 hrs. The characteristic structure due to the presence of the hydride is present after annealing at 650° or 750° C., but it has disappeared after annealing at 850° C. for 1 hr., although the hardness has not reached its minimum.—W. G.

[Steel;] *Flaky fracture and segregation phenomena* [in —]. P. Oberhoffer. *Stahl u. Eisen*, 1920, 40, 705—713, 872—878.

MICROGRAPHIC examination after etching with an ammoniacal copper reagent showed that the so-called flaky fracture of steel (*cf.* J., 1919, 289 A, 368 A, 683 A, 733 A) is closely connected with phosphorus-segregation resulting from the formation of blow-holes in the interior of the metal.

Steels; Dynamic tests on special —. A. Gessner. *Stahl u. Eisen*, 1920, 40, 781—783.

DYNAMIC tensile tests on an Amsler machine and static tests were carried out with two qualities of nickel-chrome steel and on ordinary carbon steel after they had undergone various heat treatments. With the special steels, which were of marked tenacity and had a breaking stress of about 100 kg. per sq. mm., the ability to flow under stress was developed completely in the short period of 0.003—0.005 sec. in the dynamic tests, and higher values for the ultimate tensile strength and the work utilised in producing deformation at fracture were obtained than in the corresponding static tests. The reduction of area was also greater.—J. W. D.

High-chromium steel; Heat treatment of —. H. J. French and Y. Yamauchi. *Chem. and Met. Eng.*, 1920, 23, 13—16.

SAMPLES of chromium steel containing 12.4—13.2% Cr and 0.27—0.29% C were quenched in oil from various temperatures. With increasing quenching temperature both the Brinell and Shore hardness increase until 1066° C. (1950° F.) is reached. Maximum hardness is generally obtained by quenching from or above this temperature, although in some cases with higher temperatures the hardness decreases owing to retention of solid solution. The best combination of strength and ductility is obtained by quenching from 955° C. (1750° F.), which condition coincides with maximum tensile strength. Ductility as measured both by elongation and reduction of area decreases rapidly as the quenching temperature increases. It attains a very low value at 1010° C. (1850° F.), above which temperature it remains practically constant. Maximum ductility does not correspond with maximum tensile strength or proportional limit. Tempering previously quenched samples for short periods at temperatures up to 427° C. (800° F.) decreases brittleness. Tempering above 427° C. causes marked decrease in strength and hardness. Ductility increases to a greater extent in tempered samples quenched from 955° C. The most rapid change in tensile properties and hardness occurs in tempering between 427° and 538° C. (800°—1000° F.). The structure of samples tempered for short periods tends to persist even when tempering is effected at temperatures comparatively close to the lower critical range.

—J. W. D.

Corrosion of iron and steel. W. D. Richardson. *Amer. Inst. Chem. Eng.*, June, 1920. [Advance copy.] 94 pages.

WITH ordinary varieties of iron and steel oxygen determines the beginning of corrosion, and the rôle of oxygen appears to be that of a depolariser, since it combines not only with hydrogen at the corrosion cathodes, but also with ferrous iron at the corrosion anodes. Corrosion will start in the absence of oxygen, but it soon drops to a negligible rate. Corrosion produced by salt solutions depends principally on the amount of oxygen in solution. Oxygen causes much more rapid corrosion than air, but may also produce a protective layer of rust not produced in air, thus reducing corrosion below that in air under similar conditions. Rust accelerates corrosion by acting as a cathode, but may also reduce corrosion by adhering and acting as a pro-

tector. This is more noticeable with cast iron than with the purer forms of iron. Carbon dioxide only plays a minor rôle in the corrosion of iron. Experiments carried out on the corrosion of 12 common varieties of iron and steel under 8 different conditions (in still and agitated water and brine) for 1000 days indicated that pure iron corrodes relatively slowly under non-oxidising conditions or with limited supply of oxygen and relatively rapidly in presence of oxygen or air. Cast iron, on the other hand, corrodes relatively more in the absence of air or oxygen, or with limited supply, and relatively less in the presence of air or oxygen compared with purer irons. Solution pressure, while fundamental in corrosion phenomena, is not as a rule the determining factor. This is shown by aluminium corroding not only less than iron, but also less than copper under similar conditions.—J. W. D.

Iron and steel; Rates of solution of — in non-oxidising and oxidising acids. W. D. Richardson. *Amer. Inst. Chem. Eng.*, June, 1920. [Advance copy.] 12 pages.

RELATIVELY pure iron dissolves at a low rate in non-oxidising acids such as N/1 sulphuric and hydrochloric acids, while less pure irons such as cast iron, white iron, and malleable iron dissolve at a relatively high rate. Cast steel dissolves at a rate intermediate between the two. In an oxidising acid such as N/1 nitric acid, the rates of solution of the metals are reversed, pure iron dissolving at a higher rate than cast iron, steel again being intermediate. Difficulties were experienced in producing exactly similar conditions during solution, the temperature of 16° C. used with the sulphuric acid rising to 39° C. with nitric acid. The results obtained, however, appear to indicate a general rule. Air agitation of the acids accelerates the rate of solution, but not to the extent anticipated.

—J. W. D.

Corrosion; Suggested basis for an index of — for iron and steel. W. D. Richardson. *Amer. Inst. Chem. Eng.*, June, 1920. [Advance copy.] 8 pages.

THE index of corrosion (C) recommended represents the relation between the loss per sq. cm. per hr. in N/1 nitric acid (O), and the similar loss in N/1 sulphuric acid (H). The index cannot yet be expressed as a simple ratio $C=O/H$ owing to the small amount of data available. It has to be considered as really consisting of two indices written together as a ratio for convenience, but subjects for separate consideration. H the index for one metal ought to be compared with H the index for another, and similarly the two O indices.

—J. W. D.

Tungsten, molybdenum, and vanadium; Estimation of — in their ferro-alloys. Mitt. Chemikerausschuss Ver. deuts. Eisenhüttenleute. *Stahl u. Eisen*, 1920, 40, 857—858.

Tungsten in ferro-tungsten.—0.5 g. of the finely-powdered alloy is roasted in a platinum crucible, the residue fused with 6 g. of sodium potassium carbonate, and the melt leached with water. The residue is filtered off, washed, and again fused etc. as before. The united filtrates are boiled with ammonium carbonate and alcohol to precipitate silica, alumina, and manganese oxide; the solution is allowed to stand for 2 hrs., then filtered and the precipitate washed with very dilute sodium carbonate solution. The filtrate is just neutralised with nitric acid, using methyl orange as indicator, boiled to expel carbon dioxide, and treated hot with a slight excess of mercurous nitrate, then with ammonia, drop by drop, until the precipitate becomes permanently grey. The precipitate is filtered off, washed with dilute mercurous nitrate, and ignited. The residual tungsten trioxide is heated

successively with hydrofluoric acid and ammonium chloride till of constant weight. If chromium is present, the weighed oxide is fused with alkali carbonate, the melt leached, and the chromium determined in the solution with thio sulphate. *Molybdenum in ferro-molybdenum*.—1 g. of the finely-divided alloy is dissolved in nitric acid (sp. gr. 1.2), any residue being fused with sodium potassium carbonate and the solution of the melt added to the main solution, which is then evaporated to fumes with 5 c.c. of strong sulphuric acid. The mass is treated with water, a considerable excess of ammonia added, and the liquid set aside for 2 hrs. in a warm place. It is then cooled, diluted to 1 l., 200 c.c. filtered through a dry paper and saturated with hydrogen sulphide. The light red solution is just acidified with dilute sulphuric acid (1:5) and allowed to stand for 2 hrs.; the precipitate is filtered off, washed first with hydrogen sulphide water containing sulphuric acid, then with dilute alcohol, dried, ignited apart from the paper, and the residue weighed as MoO_3 . *Vanadium in ferro-vanadium*.—0.3 g. of the finely-powdered alloy is dissolved in 20 c.c. of nitric acid (sp. gr. 1.2), and the solution evaporated to fumes with 20 c.c. of concentrated sulphuric acid. After cooling, the residue is evaporated three times with 25 c.c. of concentrated hydrochloric acid, the last evaporation being prolonged until the sulphuric acid fumes strongly. The flask is kept covered with a clock-glass during these operations to prevent re-oxidation. The final liquor is diluted with 300 c.c. of air-free water containing 15 c.c. of phosphoric acid (1:3) and the solution titrated at $60^\circ\text{--}70^\circ\text{C}$. with permanganate, which should be standardised against ferro-vanadium of known vanadium content or against pure vanadic acid.—A. R. P.

Copper assay; Electrolytic —: a new modification. F. G. Hawley. Eng. and Min. J., 1920, 110, 162—165.

From 1 to 3 g. of the sample is mixed with 1 g. of sodium bisulphate, 1 g. of ammonium sulphate, and 5 c.c. of 70% sulphuric acid; 5 c.c. of nitric acid saturated with potassium chlorate is added, and, after standing for a few minutes, a further 10 c.c. The mixture is digested warm for a short time, then evaporated until strong fumes are evolved and the mass has become pasty. After cooling, 1 c.c. of water is added, followed immediately by 50 c.c. of dilute ammonia (ammonia 2 l., water 4 l., ammonium nitrate 100 g.); the liquid is well stirred, heated to boiling, cooled slightly, 5 c.c. of strong ammonia added, and the precipitate filtered off, washed once with water, once with ammonia (1:1), and once again with water. The filtrate is boiled to expel ammonia, cooled, and treated with 3 c.c. of sulphuric and 3 c.c. of nitric acid. Meanwhile the precipitate is dissolved off the filter with dilute nitric acid, the solution made ammoniacal, a few drops of 2% potassium cyanide added, and the warm solution, without filtration, electrolysed for $\frac{1}{2}$ hr. at 0.2 amp., using a rotating cathode. The beaker is then removed and replaced by that containing the bulk of the copper, the current is switched on in the opposite direction to re-dissolve the small amount of impure copper that was deposited from the iron precipitate, then switched back to the original direction and the electrolysis continued for 1—1½ hr. at 1—1.5 amp., and finished as usual. Hydrochloric acid should be used in dissolving oxidised ores, and, if molybdenum is present, a small amount of a soluble lead salt should be added before the ammonia solution.—A. R. P.

Brasses; Special —. L. Guillet. Comptes rend., 1920, 171, 172—173. (Of J., 1906, 510, 813; 1913, 90, 913; 1920, 157A, 269A.)

BRASSES containing cobalt are in no way superior to those containing nickel, and consequently have

no industrial importance. When chromium is introduced into brasses, a portion of it enters into solution in the constituents of the brass, but another portion forms a special constituent, probably metallic chromium, which diminishes the value of the alloy. Silver has no very pronounced action on the structure of a copper-zinc alloy, but gold, on the contrary, gives a fictitious copper content (J., 1906, 540) markedly greater than the real content. —W. G.

Brasses; Special —. L. Guillet. Mem. Soc. Ing. Civils de France, 1920, 73, 154—182.

A FULL account of investigations, the chief results of which have been published previously (J., 1906, 540, 813; 1913, 90, 913; 1914, 358; 1918, 338A; 1920, 157A, 269A; also preceding abstract).

Zirconium. Marden and Rich. See VII.

Anode corrosion of lead. Brown and others. See XI.

PATENTS.

Steel melting plant. E. and W. Waring. E.P. 147,244, 7.2.19.

A MIXTURE of iron ore and coal is passed through a producer chamber on a conveyor and delivered with lime to a cupola from which the material is conveyed to a furnace, the gases from which pass through the cupola to a combustion chamber in which a part of the conveyor and a boiler are situated. The gases from the producer are conveyed to tuyères in the producer chamber and furnace.—J. W. D.

Iron; Production of —. A. Jackson. E.P. 147,999, 25.4.19.

REFINED iron containing Si 0.3—0.6%, total C 1.5—2.5%, P 0.3—0.6%, and less than 0.1% S, is prepared by melting in a cupola a mixture of steel scrap and selected pig iron in suitable proportions according to analysis.—J. W. D.

Ferro-alloys; Production of —. H. L. Suhman and W. B. Ballantine. E.P. 145,925, 9.5.19.

CASTINGS resistant to rust and corrosion are produced by reducing a pure compound of iron and chromium (e.g., oxides) with a thermo-reducing agent containing white iron, or forge scale containing silicon, or calcium silicide, so that silicon is introduced into the alloy during reduction.

—J. W. D.

Chromium-containing [iron] alloys; Process of producing —. F. A. Fahrenwald. U.S.P. 1,346,187, 13.7.20. Appl., 25.8.19.

FERROUS metal ores are mixed with just sufficient carbon to reduce all the metallic constituents, and the mixture is first heated for a long time to a temperature sufficiently high to carry out the reduction without reducing the silica in the charge; finally, the heat is raised until the reduced metal melts and the unreduced constituents are slagged off.—A. R. P.

Firearm and [iron and copper] alloys for making same. F. A. Fahrenwald. U.S.P. (A) 1,346,188, (b) 1,346,189, (c) 1,346,190, and (d) 1,346,191, 13.7.20. Appl., (A) 25.8.19, (n) 15.10.19, (c) (d) 15.12.19.

(A) An alloy of one or more iron group metals with one or more chromium group metals. (b) An alloy of approximately 36% Ni and 64% Fe. (c) An alloy of copper and not less than 25% Ni. (n) An alloy of iron with at least 30% of one or more metals at the iron group.—A. R. P.

Metallic powders; Production of —. E. Odam. E.P. 129,631, 21.6.19. Conv., 10.7.18.

GRANULATED metal, waste metal cuttings, etc., are reduced to powder by causing them to flow under

the action of centrifugal force into a conduit which directs the material between balls and the races in which they run. The material is passed several times successively through the device before being discharged.—J. W. D.

Crucible and like furnaces. J. A. Gaskill. E.P. 132,266, 3.9.19. Conv., 3.9.18.

A CRUCIBLE for melting steel, iron, or other metals, reducing refractory ores, or annealing and heat treating metal articles, is mounted on supports within a refractory combustion chamber, closed at the bottom by hinged doors, and at the top by a cover having a central vent opening. The combustion chamber is surrounded by a jacket into which the air for combustion passes tangentially at the bottom, and is preheated while passing round the combustion chamber to the outlet at the top. Two burners for fuel gas or oil project tangentially into the combustion chamber at opposite sides near the bottom. The side wall of the combustion chamber projects inwards at its middle zone, so that the combustion products are deflected into contact with the crucible and are then allowed to expand before escaping at the top.—W. F. F.

Regenerators for smelting furnaces and the like. P. Kühn. E.P. 138,103, 20.1.20. Conv., 4.5.18.

THE regenerators are provided with gratings in two compartments inclined to one another, the floor of the compartment nearer the furnace sloping downwards to the rear. This arrangement prevents clogging of the gratings when coal dust is used as fuel.—J. W. D.

Furnaces for the heat treatment of metal articles. A. Hardy, J. H. Hunt, and M. Wigglesworth. E.P. 145,958, 5.6.19.

GAS from a producer passes into a horizontal longitudinal flue below the horizontal heating chamber. This flue is provided with inclined outlets arranged alternately on opposite sides and the gas on leaving each outlet meets a stream of preheated air. The burning gas from each outlet passes through separate transverse flues, extending completely around the heating chamber. The hot gas then passes through tortuous passages adjacent to the passages through which the air supply for combustion is passed to preheat it.—W. F. F.

Alloy; Metal — for the armourings of electric cables. D. Stenquist. E.P. 136,143, 9.10.19. Conv., 5.12.18.

THE alloy is composed of lead and 0.01 to 0.1% of magnesium.—B. N.

Metal alloy. A. E. Farmer. E.P. 145,956, 4.6.19. THE alloy consists of copper, 53 lb., aluminium 0.25 lb., tin 0.25 lb., spelter 28 lb., and nickel 25 lb. The copper and 23 lb. of the spelter may be replaced by 75 lb. of 70–30 cartridge metal.—W. F. F.

Aluminium castings; Production of —. Metall-industrie Schiele und Bruchsal. E.P. 137,325, 2.1.20. Conv., 18.2.18.

IN the production of aluminium castings in metal moulds the surfaces of the moulds and their metal or sand cores are coated before each pouring with a varnish containing either aluminium or an aluminium alloy, and then slowly heated to 400° C. to burn off organic matter and leave an even coating of aluminium which prevents oxidation and ensures clean castings. The moulds and cores are then fitted together, heated to 550°–700° C., and the aluminium cast in them in the usual manner.—A. R. P.

Castings; Process of making —. Z. Jeffries, Assr. to Aluminium Castings Co. U.S.P. 1,347,481, 20.7.20. Appl., 24.9.17.

INTRICATE castings of aluminium alloys or other

metals having high coefficients of expansion are prepared by casting the molten metals into a permanent mould formed of metal having approximately the same coefficient of expansion as the metal of the casting, and a high coefficient of thermal conductivity compared with iron.

—J. W. D.

Ores; Concentration of —. L. A. Wood, and Minerals Separation, Ltd. E.P. 145,852, 23.3.19.

ORES containing copper sulphide and/or carbonaceous material, and/or molybdenite may be separated from other ingredients such as iron, zinc, or lead sulphides by the froth flotation process using frothing agents such as amyl and butyl alcohols, camphor, furfural, phenol, cresol, aniline, toluidine, picoline, lutidine, or aqueous extracts of commercial oils, such as wood tar or eucalyptus oil. The differential separation effected by these substances is improved by the addition of alkali silicates, hydroxides, or carbonates.—W. F. F.

Ores; Concentration of — and apparatus therefor. Minerals Separation, Ltd. From E. H. Emerson. E.P. 145,870, 1.4.19.

THE ore pulp is mixed with a mineral-frothing agent, and is supplied to the top of a separating vessel placed at the top of an inclined trough. The pulp is delivered into a funnel having a wide stem projecting downwards into the vessel, and the mixture is aerated and agitated by a jet of water and air which is projected downwards into the stem of the funnel. The froth carrying the mineral particles overflows at the top into the inclined trough. The residue passes from the bottom of the separating vessel to the top of a second separating vessel, and so on through a series of vessels arranged at progressively lower levels along the inclined trough. The mixing jet for each vessel is produced by a water-air injector, and the jets are directed against a transverse baffle at the centre of each separating vessel.—W. F. F.

Flotation process. R. Gahl, Assr. to Pneumatic Process Flotation Co. U.S.P. 1,346,819, 20.7.20. Appl., 10.12.18.

ADDITIONAL sand substantially free from slimes is introduced into the normal pulp and the mixture subjected to froth flotation.—J. W. D.

Corrosion of metallic surfaces; Composition for preventing —. C. A. Allison. From Naigai Kagaku Seihin Kabushiki Kaisha. E.P. 146,676, 15.5.19.

A COMPOSITION for preventing oxidation of metallic surfaces immersed in liquids consists of borax, 8–15 pts., dextrin 2–3 pts. An addition of 1% of the above to ink made from tannic and sulphuric acids prevents corrosion of pens. The composition is also applicable for protecting metal articles immersed in disinfecting solutions such as mercuric chloride, and for preventing corrosion of steam boilers.—W. F. F.

Rust-preventing treatment. W. R. Swan, Assr. to J. J. Woolverton. U.S.P. 1,346,473, 13.7.20. Appl., 8.1.20.

IRON and steel articles are heated in a sealed chamber, the chamber is then partially evacuated, and the vapour of a non-corrosive substance admitted.—A. R. P.

Electrolysing cobalt solutions. E. B. Cutten. U.S.P. 1,344,869, 29.6.20. Appl., 20.10.19.

COBALT is deposited by electrolysing a solution of cobalt sulphate, which is maintained neutral by the action of a reagent that forms an insoluble and innocuous precipitate with the liberated sulphuric radical.—B. N.

Metals [copper]; Process of extracting — from their ores. W. E. Greenawalt. U.S.P. 1,315,846, 6.7.20. Appl., 13.12.15. Renewed 15.9.19.

COPPER ores are leached with an acid solution to dissolve the copper; the rich solution is drawn off and electrolysed to deposit the copper and regenerate the acid. The gangue is washed, the washings treated with hydrogen sulphide, and the precipitated copper sulphide digested with the electrolyte to reduce the metallic salts therein to their lower state of oxidation.—A. R. P.

Nickel ores; Process of treating —. M. H. Caron, Assr. to Clevenger and Caron. U.S.P. 1,346,175, 13.7.20. Appl., 19.5.19.

NICKEL silicate ores are subjected to a preparatory reducing roast, the reduced ore being cooled under non-oxidising conditions.—A. R. P.

Metal from ores etc.; Apparatus for extracting —. H. H. Godfrey. U.S.P. 1,347,026, 20.7.20. Appl., 4.8.19.

THE apparatus consists of an ore concentrating shaking table inclined longitudinally and transversely, covered with a cathode, and an anode spaced above the cathode.—J. W. D.

Steel alloys and process of making the same. G. Honhorst (Legal Rep. of W. C. Honhorst), and J. Sofge. E.P. 138,327, 20.1.20. Conv., 8.12.17. SEE U.S.P. 1,313,894 of 1919; J., 1919, 778 A.

Steel; High-speed —. J. O. Arnold. U.S.P. 1,345,732, 6.7.20. Appl., 9.11.18. SEE E.P. 132,082 of 1918; J., 1919, 826 A.

Ferrochromium; Manufacture of —. W. L. Turner. U.S.P. 1,346,728, 13.7.20. Appl., 7.1.18. SEE E.P. 123,105 of 1917; J., 1919, 224 A.

Open-hearth furnaces. D. McLain and F. Carter. E.P. 146,020, 10.10.19. SEE U.S.P. 1,331,804 of 1920; J., 1920, 302 A.

Zinc; Treatment of zinc solutions preparatory to recovery of — by electro-deposition. D. Avery and R. T. D. Williams, Assrs. to Electrolytic Zinc Co. of Australasia Proprietary, Ltd. U.S.P. 1,347,200, 20.7.20. Appl., 25.7.18. SEE E.P. 131,702 of 1918; J., 1919, 827 A.

Metallurgical furnace. S. M. Howell. Reissue 14,914, 13.7.20. of U.S.P. 1,337,703. 20.4.20. Appl., 5.6.20. SEE J., 1920, 457 A.

Metals; Process of treating —. W. E. Ruder, Assr. to General Electric Co. U.S.P. 1,346,062, 6.7.20. Appl., 10.7.18. SEE E.P. 125,046 of 1919; J., 1919, 327 A.

Lead and zinc; Extraction of — from their ores. F. E. Elmore. U.S.P. 1,346,642, 13.7.20. Appl., 1.4.19.

SEE E.P. 131,353 of 1918; J., 1919, 827 A.

XI.—ELECTRO-CHEMISTRY.

[*Electrical precipitation.*] **Motion of droplets and particles in the field of the corona discharge.** R. C. Tolman and S. Karrer. Chem. and Met. Eng., 1920, 22, 1203—1206.

By photographing the form of a water jet arranged within a precipitation chamber containing a central wire between two parallel sheet metal plates, the jet being discharged parallel to the central wire, it is shown that there are four methods whereby particles in the field of a corona discharge may ob-

tain charges of electricity, namely, by induction, by conduction of electricity from the earth, by the adsorption of ions present in the corona field, and by actual contact with an electrode. The motion of the particles is determined by the charge received and by the potential gradient of the field. The space charge in the major part of the corona discharge is of the same sign as the central wire. It is believed that the adsorption of ions having the same charges as the central wire is the underlying process in the action of Cottrell precipitators.

—J. S. G. T.

Lead; Anode corrosion of — in sodium hydroxide solutions. O. W. Brown, C. O. Henke, and L. T. Smith. J. Phys. Chem., 1920, 24, 367—378.

THE amount of corrosion of lead anodes in the electrolysis of sodium hydroxide solutions rapidly increases with increasing temperature; thus using a solution of 152 g. of sodium hydroxide per l. the amount of corrosion is 4.5% of the theoretical at 20° C., 98.3% at 60° C., and theoretical at 75° C. At all temperatures the corrosion increases, with increasing concentration of the sodium hydroxide, to a maximum which it reaches with 152 g. per l. and then falls with further increase in concentration. Thus at 60° C. the following fractions of the theoretical amount of corrosion are found with the concentration given: 35 g. per l., 5.7%; 71 g., 49.3%; 110 g., 96.4%; 152 g., 98.3%; 192 g., 58.5%; 304 g. per l., 37.7%. The current efficiency increases rapidly with increase in temperature, this being especially marked for solutions containing 152 g. per l. and a current density of 4 amps. per sq. dm. There is a 100% corrosion when the bath tension is low (0.2—0.3 volt), and a low corrosion when the voltage is high (3.2 volts). When the anode is corroding properly the discharge potential is about 0.2 volt, but when it ceases to corrode the discharge potential rises to -1.317 volt.—J. F. S.

Salts; Electrical conductivity of mixtures of fused and solid —. C. Sandonnini. Gaz. Chim. Ital., 1920, 50, I., 289—321.

THE conductivities of various binary mixtures of salts, some of which merely mix while others form compounds, have been measured. For the fused mixtures the equivalent conductivity—in which allowance is made for the slight change in specific gravity occurring on mixing—is always less than the value calculated from the law of mixtures; the divergences are the more marked for those pairs of salts with which compounds exist in the fused mass, but the conductivity isotherms in general exhibit no singular points corresponding with the composition of such compounds, although for KCl—CaCl₂ this is approximately the case. (*cf.* J.C.S., Sept.) —T. H. P.

PATENTS.

Electrolytic cell. L. W. Chubb, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. (A) 1,344,752 and (B) 1,344,753, 29.6.20. Appl., (A) 3.2.17, (B) 7.3.19.

(A) A FILM-FORMING conductor for electrolytic cells is composed of metallic titanium. (B) An electrode for electrolytic cells comprises a compound, e.g., an oxide, of titanium.—B. N.

Electric furnaces. A. M. Teixeira. E.P. 147,988, 23.4.19.

SEE U.S.P. 1,313,761 of 1919; J., 1919, 830 A.

Catalysis. E.P. 126,279. See I.

Hypochlorites. E. P. 148,095. See VII.

Resistance material. U.S.P. 1,346,874. See VIII.

Electric cables. E.P. 136,143. See X.

XII.—FATS; OILS; WAXES.

Linseed oil; Hexabromide test of the purity of —. Amer. Soc. Testing Materials. Circ. No. 99. Educat. Bureau, Paint Manufacturers' Assoc., U.S.A., July, 1920. 16 pages.

In Bailey's modification of Steele and Washburn's method (J., 1920, 197 A) the linseed oil fatty acids are prepared in an atmosphere of carbon dioxide. 1 g. is dissolved in 25 c.c. of ether, which has been purified, dried, and saturated with linolenic hexabromide at 0°–2° C., in a weighed centrifuge tube (1 in. diam. × 5 in. long), which is then placed in a freezing mixture at about –5° C. (crushed ice and dilute hydrochloric acid). Freshly-prepared bromine solution (5 c.c. in 25 c.c. of glacial acetic acid) is added, drop by drop, until a deep red coloration is produced. The tube is left for about 14 hrs. in an ice-chest, then centrifuged, cooled in an ice-bath, and the ether decanted. The deposit is washed with 20 c.c. of the prepared ether at 0° C. and the tube again centrifuged, and this washing and decantation repeated twice more. Finally the tube is warmed in water at 60° C. until most of the ether has evaporated and attached to a vacuum pump showing a pressure of 30–40 mm., and after 15 mins. is removed from the water bath, allowed to stand for 15 mins. and weighed. The results are somewhat lower and less concordant than those given by Steele and Washburn's method. (Cf. J., 1920, 417 A.)—C. A. M.

Tung oil; Standard specifications for the purity of raw —. Amer. Soc. for Testing Materials. Circ. No. 100. Educat. Bureau, Paint Manufacturers' Assoc., U.S.A. July, 1920. 12 pages.

It is proposed that raw tung oil shall answer to the following requirements:—Sp. gr. 15.5°/15.5° C., 0.943–0.939; acid value, 6 (max.); saponif. value, 195–190; unsaponif. matter, 0.75% (max.); n_D^{25} = 1.520–1.515; iodine value (Hübl 8 hrs.), 165 (min.); and heating test, 12 mins. (max.). The following modification of Browne's heating test is prescribed:—Test tubes for the oil should be 15 cm. by 16 mm., and be closed by a cork through which a glass rod 3 mm. in diam. can move easily. A beaker (800 c.c.), 15 cm. high and 10 cm. in diam., is filled to 7.5 cm. with cottonseed or soya bean oil, and in this is suspended a thermometer at 1.5 cm. from the bottom. The oil is heated, and at 293° C. (560° F.) two test-tubes containing respectively 5 c.c. of the tung oil and 5 c.c. of a control sample are introduced so that the bottom of each tube is level with the lowest part of the thermometer bulb. The source of heat is removed for about 45 secs. and then replaced, and the temperature maintained at 282° C. (540° F.) as constantly as possible. After 9 mins. the glass rods are raised at intervals of ¼ min., and a note taken of the time required for each sample to set firmly. The average time of setting of pure American pressed tung oil was 9½ mins., whilst the same oil with 5% of soya bean oil required 10½ mins. to set.—C. A. M.

Yeast fat; Nature of —. J. S. MacLean and E. M. Thomas. Biochem. J., 1920, 14, 483–493.

CRUDE yeast fat is a brown liquid with a high iodine value and a low saponif. value, and has a large proportion (25–45%) of unsaponifiable matter. It may contain as much as 20% of a sterol, apparently identical with ergosterol, partly in the free state and partly as fatty acid esters. Palmitic, oleic, and linolic acids have been identified with certainty in yeast fat, and there is evidence for the presence of lauric and possibly arachidic acids. (Cf. J.C.S., Sept.)—W. G.

Cockle-burr oil. A new seed oil. L. B. Rhodes. J. Amer. Chem. Soc., 1920, 42, 1507–1508.

THE burrs were of the species *Xanthium echinatum*.

The kernels freed from the black shucks amounted to 30.69% of the burrs, and contained moisture, 6.95–7.22%; crude protein, 40.34–40.53%; oil, 29.78–29.89%; crude fibre, 2.47–2.58%; ash, 5.42%. When cold pressed they gave a light yellow sparkling oil, with a pleasant odour and a nutty flavour. It had sp. gr. at 15.5° C. 0.9251; $n_D^{15.5}$ = 1.4773; Zeiss butyrometer reading at 20° C. 1.4771, 40° C. 1.4691; solidif. pt. –18.0° C.; iodine value, (Hanus) 140.8; saponif. value 190.2; Reichert-Meissl value 0.233; Hehner value 89.7; acetyl value 10.6; m.pt. of fatty acids 19.0° C. The oil is a drying oil. The kernel meal and the press cake were both highly toxic to guinea-pigs.—W. G.

"Stearic" acids; Explanation of uniform composition of —. B. H. Nicolet. J. Ind. Eng. Chem., 1920, 12, 677–679.

COMMERCIAL "stearic" acids were found to agree within 0.5° in solidif. pt. (55.18°–55.66° C.) after allowing 0.2° C. for each 1% of oleic acid calculated from the iodine value. In the case of mixtures of pure stearic and palmitic acids the solidif. pt. curve falls continuously from 100% stearic acid to a point B (55° C. 50% palmitic acid), then rises to a maximum at C (55.4° C. 55% palmitic acid), falls to D (about 72% palmitic acid), and finally rises normally to 100% palmitic acid. It is concluded that in accordance with the behaviour of solid solutions the commercial "stearic" acids examined have been derived from mixtures containing between 53 and 72% of palmitic acid in their saturated fatty acids, so that their composition would fall within the section of the curve from B to D, and on pressing would approach the composition corresponding to the maximum point C, and thereafter show no further alteration.—C. A. M.

Fatty acids; Synthesis of — from paraffin wax and other petroleum fractions. K. Löffl. Chem.-Zeit., 1920, 44, 561–562.

In the catalytic oxidation of petroleum hydrocarbons (cf. J., 1920, 459 A, 521 A) the best yields of fatty acids are obtained with lead or mercuric compounds, which apparently dissolve completely in the products of oxidation. The best results are obtained by oxidising at 115°–120° C., under a pressure of 3 atm. and with agitation for about 7 hrs., in the presence of water, which is an essential factor. Alkalis have not the catalytic influence claimed by Schaal and by Franck (J., 1920, 395 A). The oxidation products (98–120%; av. 106–110% on the original material) consist of about 7–20% of water, 25–40% of lower fatty acids, with small amounts of aliphatic aldehydes and ketones, and 78–50% of higher fatty acids and 10–15% of unsaponifiable matter. They may be fractionated by distillation (up to 200° C.) with superheated steam and then by distillation with steam *in vacuo*. The unsaponifiable hydrocarbons may be separated by fractional precipitation with alcohol, alcohol-ether, and alcohol-ethyl acetate. The fatty acids mixed with 10–20% of tallow or coconut oil fatty acids yield satisfactory soaps.—C. A. M.

Fats; Comparison of different methods for determining the iodine value of —. T. Sundberg and M. Lundborg. Z. Unters. Nahr. Genussm., 1920, 39, 87–95.

THE Hanus method gives results which agree with those found by the Hübl method, whilst considerably higher values are found by the Wijs method and slightly lower by Winkler's method. In the case of fats or oils with high iodine values the Hübl method gives results which are nearest to the calculated values, e.g., linolic acid, 181.1 (calculated), 194.3 (Hübl), and 201.1 (Wijs).—W. P. S.

Cottonseed oil. Daniels and Loughlin. See XIXA.

PATENT.

Linseed oil substitute. U.S.P. 1,347,074. See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Air in paint and varnish containers; Composition of —. H. A. Gardner and T. M. Rector. Paint Manufacturers' Assoc., U.S., and National Varnish Manufacturers' Assoc. Circ. 103, Aug., 1920. 2 pp.

The oxygen in the air contained in the space above the liquid in a sealed can of paint or varnish is found to be completely absorbed by the drying oils in a period of two weeks.—A. de W.

PATENTS.

Phenolic condensation product; Process of producing a —. Method of preserving infusible phenolic condensation products from discoloration. L. V. Redman, A. J. Weith, and F. P. Brock, Assrs. to Redman Chemical Products Co. U.S.P. (a) 1,345,694 and (b) 1,345,695, 6.7.20. Appl., (a) 16.9.18 (Renewed 12.5.19), (b) 24.7.19.

(A) To obtain a phenolic condensation product without the aid of a condensing agent, a solution of formaldehyde and a phenolic substance largely in excess of the formaldehyde is boiled until the formaldehyde is substantially eliminated from the supernatant aqueous layer which forms, the aqueous layer is discarded, the viscous gummy lower layer is concentrated by further heating, then mixed with a solution of formaldehyde in lesser proportion than in the first step, poured into moulds, caused to set at a temperature below 100° C., removed from the moulds, and hardened to an infusible and substantially insoluble condition by prolonged heating below 100° C. (b) An infusible and practically insoluble phenolic condensation product of the transparent order is preserved in a medium which is inert thereto when exposed to the action of sunlight. —A. de W.

Linseed oil substitute and process of making same. R. C. Bone, Assr. to M. A. Esteve. U.S.P. 1,347,074, 20.7.20. Appl., 23.3.18.

A MIXTURE of 60 pts. of kerosene and 40 pts. of lubricating oil is mixed with 10 pts. of rosin, the mixture heated to boiling for approximately 50 mins., agitated, 2½ pts. of manganese hydroxide added with stirring and the mixture boiled for about 15 mins., the supernatant liquid withdrawn, about 1 pt. of red-rod oil added, and the mixture allowed to rest for about 12 hrs.—A. de W.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; New elasticity testing machine for soft (vulcanised) —. A. Schob. Gummi-Zeit., 1920, 34, 995—997.

A METAL bob supported pendulum-fashion, with an effective weight of 200 g., falls through a vertical height of 25 cm., and at the bottom of its swing strikes a disc of the rubber, 6 mm. thick and approximately 36 mm. diam., supported vertically against the heavy base of the machine. The extent of the recoil of the pendulum is read from a graduated arc and indicates the relative elasticity of the material of the disc. The test pieces are dusted with tale before use and the temperature is kept as near 20° C. as possible. Results are quoted illustrating the effect of different temperatures and of different thicknesses of material.

—D. F. T.

PATENT.

Vulcanising dissolved caoutchouc; Process for —. S. J. Peachey. E.P. 146,731, 2.8.19.

RUBBER in solution in e.g. benzene, naphtha, carbon bisulphide, etc., can be vulcanised at ordinary temperatures by treatment with sulphur in the presence of nitrosobenzene or similarly constituted nitroso-hydrocarbons of the cyclic series.—A. de W.

XV.—LEATHER; BONE; HORN; GLUE.

Tan liquors; Determination and control of acidity in —. W. R. Atkin and F. C. Thompson. J. Soc. Leather Trades' Chem., 1920, 4, 143—152.

A RAPID method is described for determining the actual hydron concentration of tan liquors, by the use of bromophenol blue, which changes colour from $pH=2.8$ to $pH=4.6$. The colour observed through two test-tubes, one containing the tan liquor and the other the standard acid solution and indicator, is matched through a yellow screen against two other tubes, one containing water and the other the indicator, to which is added increasing amounts of tan liquor until the matching is complete. The quantity of tan liquor in the first tube is increased to correspond with that in the last one, and thus allowance is made for the colour of the tan solutions. A simple calculation shows the acid present in the amount of tan liquor necessary to match the standard. For control purposes the tan liquor can be titrated with a standard solution of acid until its acidity reaches that required as shown by the test. Figures are given for various tan liquors and comparisons are made with figures obtained by Procter's lime water method.—D. W.

Leather; Estimation of hide substance in —. P. Chambard. J. Soc. Leather Trades' Chem., 1920, 4, 159—161.

DISCREPANCIES in hide substance determinations by the Kjeldahl method have been attributed to incomplete combustion of organic matter following the use of an insufficient quantity of potassium permanganate and also the difficulty of obtaining an average sample. The author recommends that the leather should be very finely cut up, and 2.8 g. heated with 25 c.c. of sulphuric acid, 12 g. of pure potassium bisulphite, and 0.6 g. of anhydrous copper sulphate. The mixture is boiled for 2½ hrs. With 9 different samples of leather the method gave higher figures than those obtained by oxidising with permanganate.—D. W.

Glues and gelatins; Properties and constitution of —. R. H. Bogue. Chem. and Met. Eng., 1920, 23, 5—12, 61—66, 105—109, 154—158, 197—203.

THERE is no variation in the viscosity of solutions of normal glues with time up to 90 mins. Vigorous agitation of a glue or gelatin solution results in a lowering of the viscosity as also does prolonged heating. Dehydration produces a reversion of "soluble" gelatin to "insoluble" collagen. At temperatures well above the melting point the viscosity increases nearly as a logarithmic function of the concentration and varies inversely as the temperature. Addition of formaldehyde, chloral hydrate, and alums increases the viscosity. Alums have no effect upon the gel strength, but the other substances decrease the strength. Nearly all electrolytes produce a lowering in gel strength, but their effect upon viscosity may be continually to increase, continually to decrease, or first to increase and later to decrease the viscosity. It is probable that the colloid aggregates of protein molecules are united into a "streptococcic thread" which would account more adequately than the older theories for the various properties of gelatin. If the gel strength remains constant the viscosity varies as the melting point, and if the normal viscosity remains constant the gel strength varies as the

melting point. There appears to be no relation between the proximate constitution of glues and their gel strength and viscosities except that the water content of the air-dry glue is proportional to the gel strength. The water-retaining capacity of glues and gelatins is therefore controlled by the same factors as determine the gel strength. This varies directly as the protein nitrogen content and indirectly as the proteose and peptone nitrogen in all glues and gelatins. The amino-acid nitrogen is greater in bone glue than in hide glues, and tends slightly to increase with decrease in gel strength. The melting point and therefore the viscosity of a glue is determined normally by the balance between the protein content and that of its hydrolysed products, but at any given uniform gel strength the melting point and viscosity will depend upon the ratio of large to small sized molecules within the protein group. As the boiling of a glue progresses it is probable that some foreign substances as chondridin, keratin, mucin, etc., become hydrolysed and enter the solution, causing variations in the different grades. Hide and bone glues differ from each other in chemical constitution. Fish glue corresponds more closely in its composition to low-grade bone glue than to any other. Fish glue and isinglass differ from animal glues in their low content of proline, oxyproline, and tryptophane. The strength of a glue in service is a function of the gel strength and of the viscosity, and directly proportional to the melting point. The melting point method for evaluation of glues is recommended as yielding a true index of actual strength in service. A very full bibliography on the subject is given.

—D. W.

Gelatin solutions; Acidity of ash-free and of commercial — H. E. Patten and T. O. Kellems. *J. Biol. Chem.*, 1920, 42, 363—366.

The isoelectric point of ash-free gelatin lies at $p_H = 4.8$, corresponding to a hydrogen-ion concentration of 1.59×10^{-5} , and that of commercial gelatin at $p_H = 5.64$, corresponding to a hydrogen-ion concentration of 2.28×10^{-5} .—W. G.

PATENTS.

Tanning material, and method of producing the same. A. Koetzie, Assr. to Rohm and Haas Co. U.S.P. (A) 1,344,950, (B) 1,344,951, and (C) 1,344,952, 29.6.20. Appl., (A) 12.4, (B) (C) 6.6.19.

(A) SULPHOPHENOLCARBOXYLIC acid, (B) sulphocresol-carboxylic acid, or (C) chlorinated naphthalene-sulphonic acid is condensed with formaldehyde. The products are amorphous, soluble in water, precipitate glue and gelatin in acid solutions, and produce a substantially white, strong, pliable leather.

—D. W.

Leather; Method of making glazed kid — Method of seasoning leather. M. A. Zdanowski. U.S.P. 1,345,429 and (B) 1,345,430, 6.7.20. Appl., (A) 21.4.17, (B) 27.4.20.

(A) THE tanning solution is prepared by melting salt and aluminium sulphate before dissolving them in water. (B) The leather is seasoned with a mixture of water, Nigrosine, carbolic acid, blood albumin, wood alcohol, gelatin, and formaldehyde.—D. W.

Leather; Process of making — A. Rogers. U.S.P. 1,346,385, 13.7.20. Appl., 5.5.20.

HIDES and skins are subjected to a mineral tannage followed by treatment with shark liver oil.—D. W.

XVI.—SOILS ; FERTILISERS.

Protozoa in the soil; A method for estimating the number of active — D. W. Cutler. *J. Agric. Sci.*, 1920, 10, 135—143.

THE total number of protozoa in the soil is first found by a dilution method. A fresh portion of the

soil is then treated with sufficient 2% hydrochloric acid to neutralise the carbonate present and leave an excess of acid, and the sample is left overnight. By this means all the active forms are killed. A second count by the dilution method gives the number of cystic protozoa in the soil, and the number of active forms can then be found by difference.—W. G.

Arable soil; Measure of the ammonifying power of — R. Peretti. *Att. R. Accad. Lincei*, 1920, 29, i., 251—256.

THE best conditions for carrying out the solution method of determining the ammonifying power of arable soil are as follows:—10 c.c. of 1.5% peptone solution is treated in a test-tube with 5 c.c. of the solution obtained by steeping 50 g. of the soil in 500 c.c. of well-water. The tube is kept in a thermostat at 20°—25° C. for 4 days, after which the ammonia produced is estimated by distilling the contents of the tube in presence of magnesia. The mean of four separate determinations is taken.

—T. H. P.

Cyanamide in the soil; Mechanism of the decomposition of — G. A. Cowie. *J. Agric. Sci.*, 1920, 10, 163—176. (*Cf. J.*, 1919, 380 A.)

CYANAMIDE is first broken down to urea in soils by a purely chemical change, and then the urea is converted into ammonia by soil organisms. In sterile soils (previously heated to 120° C. or 135° C.) only the first change occurs and there is an accumulation of urea. In the case of an inert sand in which the cyanamide did not undergo decomposition, it was found that the addition of a zeolite, prehnite, resulted in the conversion of the cyanamide into urea. Cyanamide does not appear to break down in this manner in peat and fen soils; in these it gives rise to a relatively small production of urea under normal conditions.—W. G.

Humic acid. F. Fuchs. *Chem.-Zeit.*, 1920, 44, 551.

THE results obtained with humic acid prepared from peat by means of alkali indicated that it was a true acid, with molecular weight of about 680. It formed simple salts with different metals, and was precipitated by inorganic or organic acids. When treated with sodium sulphide or sulphite it yielded a soluble sulphite compound which reacted with aniline and other organic bases. (*Cf. J.C.S.*, Sept.)—C. A. M.

Plant growth; Action of chromium and manganese on — T. Pfeiffer, W. Simmermacher, and A. Rippel. *Bied. Zentr.*, 1920, 49, 259—263.

EXPERIMENTS on oats and barley treated with an ordinary fertiliser and with small proportions of chrome iron ore or potassium bichromate failed to reveal any beneficial effect of the chromium, and similar experiments on oats treated with manganese sulphate indicated that the effect of the manganese is at most very minute.—T. H. P.

Iron; Toxicity of — [to plants] and the antitoxic properties of copper with respect to ferrous salts. L. Maquenne and E. Demoussy. *Comptes rend.*, 1920, 171, 218—222.

FERROUS salts are much more toxic to young plants than ferric salts. Any condition which tends to facilitate the oxidation of ferrous salts will consequently diminish their toxicity. This explains the beneficial effect of copper sulphate in diminishing the toxicity of ferrous salts to plants, since the former has been shown to exert a catalytic effect on the oxidation of the latter.—W. G.

Manurial composts; Occurrence and nature of the plant growth-promoting substances in various organic — F. A. Mockridge. *Biochem. J.*, 1920, 14, 432—450.

LEAF-MOULD, fresh and well-rotted stable manure,

as well as well-manured fertile soil, contain, in varying proportions, water-soluble substances, which are effective as plant growth-promoting substances, as is shown by the effect of their aqueous extracts on the development of plants of *Lenina major*. In all cases the extract of rotted manure was more effective than that of fresh material. All these materials contain appreciable quantities of nucleic acid and its derivatives in various stages of decomposition, and the greater the bacterial decomposition of the material concerned, the greater is the resolution of the original nucleic acid into its free bases.—W. G.

XVII.—SUGARS; STARCHES; GUMS.

Glucosan. A. Pietet and P. Castan. *Comptes rend.*, 1920, 171, 243—245. (See *Gelis ibid.*, 1860, 51, 331.)

When dextrose is heated at 150°—155° C. under a pressure of 15 mm. it gives a crystalline anhydride, glucosan, m.p. 108°—109° C., which is very deliquescent, gives a tribenzoyl derivative, and a sodium bisulphite compound. (*Cf.* J.C.S., Sept.) —W. G.

XVIII.—FERMENTATION INDUSTRIES.

Brewing water; Zikes' method of testing — for organisms injurious to wort. R. Heuss. *Z. ges. Brauw.*, 1920, 43, 177—180.

In Zikes' method for the biological examination of brewery water (*Allg. Z. Bierbr. Malzfabr.*, 1915, 43, 237), different volumes of the water, ranging from 50 to 0.5 c.c., are mixed with equal volumes (*i.e.*, 50 to 0.5 c.c.) of sterile brewery wort previously concentrated to twice its normal extract-content. The mixtures, contained in a series of special U-shaped apparatus of corresponding capacities, each having one limb closed and the other plugged with cotton wool, are incubated at 25° C. and observed after 7 days. The closed limb, which is completely filled with the liquid, enables the development of anaërobic organisms to be detected, whilst film-formation and the growth of aerobic organisms may be observed in the plugged limb. This method, compared with Hansen's method, has the advantage of employing a much larger volume of the water, and it also ensures that the organisms are incubated in presence of wort constituents at the normal concentration of the latter. In comparative tests of a number of waters by Zikes' and Hansen's methods the former was found the more sensitive, revealing in some of the waters the presence of organisms which Hansen's method failed to show.—J. H. L.

Inversion of sucrose; Physico-chemical investigations of the diastatic —. A. Chaudun. *Ann. Chim.*, 1920, 43, 307—319.

A FULL account of work already published (*J.*, 1918, 525 A, 600 A; 1919, 550 A, 957 A).—W. G.

Fermentation of dextrose, galactose, and mannose by Lactobacillus pentoaceticus n.sp. W. H. Peterson, E. B. Fred, and J. A. Anderson. *J. Biol. Chem.*, 1920, 42, 273—287.

Dextrose, galactose, and mannose are fermented by *Lactobacillus pentoaceticus* (*J.*, 1920, 381 A) to give lactic acid, ethyl alcohol, and carbon dioxide together with a little acetic acid, which is probably produced by secondary fermentation of the lactic acid. Dextrose and galactose are fermented at approximately the same rate and to the same extent, but mannose is fermented more slowly and less lactic acid is produced than in the case of the other two sugars.—W. G.

Worts and beers; Changes in the acidity, surface tension, and colour of —, produced by ultrafiltration. W. Windisch and W. Dietrich. *Woch. Brau.*, 1920, 37, 231—233, 237—239.

A MALT wort, the corresponding beer, and the same beer after boiling for 20 mins. under a reflux condenser, were subjected to ultrafiltration in a series of Beechhold glacial acetic acid collodion filters (*cf.* *J.*, 1906, 1177; 1907, 993) of 1, 3, 4.5, 6, and 7.5% impregnation. The original surface tension of the beer was practically the same as that of the wort; in both cases ultrafiltration through filters of increasing fineness brought the surface tension nearer and nearer to that of water. The greater part of the colouring matters of the wort and beer was removed by the 3% or 4.5% filter; only a pale yellow colour remained, resembling that of cold water extracts of ground barley, and this colour was not removed by the finest filter used (7.5%). The acidity of the wort and beer (as determined by titration) was reduced by ultrafiltration, the maximum reduction, amounting to 25—35% of the original acidity, being effected by the 7.5% filter. The 6% filter gave anomalous results, having no more effect, and in some cases less effect, than the 4.5% filter.—J. H. L.

Beer foam. H. Lüers, K. Geys, and A. Baumann. *Z. ges. Brauw.*, 1920, 43, 185—187, 193—195, 201—203. (See also Windisch and Bermann, *J.*, 1920, 462 A.)

LARGE quantities of foam from beers of various types were collected and allowed to subside in closed vessels and the resulting liquids were analysed and compared with the original beers. The most significant differences observed were as follows:—In general the foam contained rather more total nitrogen but considerably less amide nitrogen (determined by "formol-titration") than the original beer. The foam also possessed a higher ester-content and total acidity than the beer, and a volatile acidity 2—10 times as great as that of the latter. The surface tension of the foam was somewhat lower than that of the beer. The collapsed foam contained numerous flecks of coagulated protein, due to the fact that colloidal substances in liquid films tend to change from the sol to the gel state, whereby the stability of the films is much increased. Colloidal particles of nitrogenous substances of a certain size are more favourable to foam formation than larger or smaller ones. Hence the formation of head on beer may be detrimentally affected by excessive proteolysis, or by filtration through too fine a medium which retains particles of the optimal size; on the other hand, chilling may in some cases diminish head-forming power by causing particles of the optimal size to coalesce into larger particles, but this process may usually be reversed by warming again.—J. H. L.

Moselle wines; Addition of sugar to, and reduction of acidity of —. Wellenstein and Seiler. *Z. Unters. Nahr. Genussm.*, 1920, 39, 1—30.

From inferior musts, made partly from frost-bitten grapes, and possessing abnormally high acidity, it was found possible to obtain marketable wines of fine flavour by suitable treatment, including warm storage, de-acidification with calcium carbonate, re-fermentation, especially in the early part of the year after vintage, and fining with isinglass and gelatin. Contrary to a widely held opinion, long storage at 15°—16° C. tended to improve the flavour, and also accelerated maturation and reduction of acidity. Addition of sugar solution did not always reduce the final acidity in proportion to the dilution brought about by the added solution. The conditions favourable to the spontaneous biological reduction of acidity are: warm

storage, omission or minimisation of sulphuring, and frequent rousing of the yeast after the main fermentation. Reduction of acidity is impeded by the presence of a high proportion of tartaric acid, and therefore de-acidification with calcium carbonate before fermentation or re-fermentation promotes a rapid biological destruction of acidity. De-acidification with pure, precipitated calcium carbonate, in the early months or autumn of the year after the vintage, followed by re-fermentation, was found to give excellent results, but de-acidification of unfermented musts proved detrimental to the character of the wines. De-acidification and sugaring of young wines should only be carried out when they have been transferred to casks free from tartar.—J. H. L.

Wine; Balance of the specific gravity of —. W. J. Baragiola and O. Schuppli. Z. Unters. Nahr. Genussm., 1920, 39, 313—335.

THE sp. gr. of an aqueous solution of the total solids of a wine was found to be 1.0072, as calculated from the sum of the sp. grs. of 25 separate constituents, whilst direct determination of the sp. gr. gave 1.0084. The difference, 0.0012, between the calculated and found sp. gr. would correspond with about 3 g. per l. of total solids consisting of unknown or undetermined constituents.—W. P. S.

Jerusalem artichokes; Production of alcohol from —. M. Rüdiger. Z. Spiritusind., 1920, 43, 203.

LABORATORY experiments indicated that a fair yield of alcohol could be obtained from Jerusalem artichokes in small distilleries. Tubers which had remained in the ground until the new year, and had thus deteriorated, gave a yield of 6.4 l. of alcohol per 100 kg. by fermentation of a mash of the raw pulped material without preliminary hydrolysis. By heating the mash for an hour at 55° C. before fermentation the yield of alcohol was increased by 0.4 l., but heating at 75° or 100° C. reduced the yield. Higher yields were obtained by heating with acid, but this would not be economical for small distilleries. The spirit obtained, carefully re-distilled once, was more palatable than spirit from beets.—J. H. L.

Sulphite cellulose waste liquor. McKee. See V.

Yeast fat. MacLean and Thomas. See XII.

Oralic acid. Bau. See XXIII.

Methyl alcohol. Gettler. See XXIII.

PATENTS.

Glycerol; Manufacture of — from sugar. Verein. Chemische Werke A.-G. E.P. (A) 138,099, 20.1.20, and (B) 138,329, 21.1.20. Conv., (A) 12.4.15, (B) 22.4.16.

(A) COMMERCIAL sucrose, dextrose, levulose or invert sugar is fermented by yeast in presence of one or more organic or inorganic substances of alkaline reaction, e.g., disodium phosphate, sodium or ammonium carbonate, or sodium bicarbonate. Catalysts such as manganese or iron sulphate may also be added. (B) The yeast may be employed for successive fermentations if after each alkaline fermentation, which is preferably conducted in presence of sodium sulphite together with a small quantity of a hydrosulphite or sulphonylate, the yeast is regenerated by a purifying fermentation in presence of dilute acid. The yeast thus treated produces a higher yield of glycerol in each successive alkaline fermentation, and it also yields a surplus which can be used for baking or fodder. (Cf. Connstein and Lüdecke, J., 1919, 691 A).—J. H. L.

XIXA. FOODS.

Flour; Pentosans as a basis for determining the grade of —. J. Gerum. Z. Unters. Nahr. Genussm., 1920, 39, 65—69.

THE pentosan content of a flour affords but little evidence as regards the proportion of the wheat grain present, since the amount of pentosans contained in different grades of flour varies only within narrow limits; a "94%" flour yielded 8.77%, and an "82%" flour 7.47% of phloroglucide. Better information is obtained from the starch and ash content of the flour.—W. P. S.

Wheat flour; Detection and determination of — in mixtures with rye and barley flour. J. Abel. Z. Unters. Nahr. Genussm., 1920, 39, 44—47.

RYE flour gives no gluten, and in mixtures with wheat flour it prevents the recovery of the calculated amount of gluten from the latter; mixtures containing more than 60% of rye flour give no gluten at all. Thus mixtures of wheat and rye flours containing 0, 10, 20, 30, 40, 50, and 60% of the latter gave respectively 9.0, 8.1, 7.2, 6.0, 4.5, 2.8, and 0.6% of dry gluten. Mixtures of wheat and barley flours containing respectively 0, 10, 80% of the latter gave 9.0, 8.9, 9.2, 9.1, 9.0, 8.3, 6.2, 3.5, and 0.1% of dry gluten. The method is applicable also to doughs which have been mixed with yeast for a day or even longer.—J. H. L.

Milk; Detection of added water in — by means of the refractometer. Utz. Milchw. Zentr., 1920, 49, 137—143.

THE refractometer value of milk serum affords more trustworthy evidence of the presence of added water than does the sp. gr.; the lowest value for genuine milk is 51.0 scale degrees. The milk serum is best prepared by the use of the mercury chloride reagent (Ambühl and Weiss; J., 1920, 464 A).

—W. P. S.

Milk; Acidity of ropy —. K. Freear and E. C. V. Venn. Biochem. J., 1920, 14, 422—431.

FROM two samples of ropy milk organisms were isolated which were either identical with or closely related to *Streptococcus Hollandicus*. When these were inoculated into milk they produced ropiness associated with a minimum acidity of 0.43% lactic acid, and the ropiness continued for varying periods of time accompanied by an acidity which, except on one occasion, never rose above 1%. In every case the ropiness diminished, and after sufficient time finally disappeared. From electrometric determinations it appeared that ropiness was found in milk with a pH range of 5.82 to 4.10. With diminishing ropiness no appreciable alteration in the pH value could be detected.—W. G.

Milk, blood, and other nitrogenous substances; Improvement and treatment of —. E. F. W. Rasch. Milchw. Zentr., 1920, 49, 61—67, 77—79.

TREATMENT of the milk, blood, etc., with bases (soda, potash, lime, ammonia) or basic salts is advocated in order to neutralise the acidity and to form albuminates etc.; such treated milk or blood is suitable for drying to a powder and for other purposes.—W. P. S.

Vitamine; Quantitative method for the determination of —. R. J. Williams. J. Biol. Chem., 1920, 42, 259—265.

A KNOWN amount of the solution to be tested is added to 100 c.c. of a stock culture solution containing both asparagine and ammonium sulphate, and the mixture is made up to 110 c.c. and sterilised. 1 c.c. of a freshly-prepared suspension of yeast containing 0.3 g. of fresh yeast per l. is added and the whole is incubated for 18 hrs. A little formalde-

hyde is added to stop the growth, any growth of wild yeast on the surface is skimmed off with a copper gauze scoop, and the yeast proper is collected in a Gooch crucible, washed with water and alcohol, dried at 103° C. for 2 hrs. and weighed. The weight of yeast produced minus that produced in a control is proportional to the amount of vitamin present. The results are expressed as the "vitamine number" of the material, which is the number of mg. of yeast produced by the addition of 1 g. of the material.—W. G.

Vitamine studies. V. Antiscorbutic properties of raw beef. R. A. Dutcher, E. M. Pierson, and A. Biester. *J. Biol. Chem.*, 1920, 42, 301—310.

The addition of water extracts of raw lean beef to a basal ration deficient in antiscorbutic ingredients had no effect on the time of onset of scurvy or the length of life of guinea-pigs. The beef extract contained no deleterious ingredients, since on the further addition of orange juice to the diet there was no scurvy and the animals were in excellent condition.—W. G.

Antiscorbutic property of vegetables. II. Experimental study of raw and dried potatoes. M. H. Givens and H. B. McClugage. *J. Biol. Chem.*, 1920, 42, 491—515.

Ten grams of raw white potato per day was sufficient to protect guinea-pigs against scurvy for the duration of the experiment (129 days). There is no appreciable reduction of the antiscorbutic potency of the potato by boiling it in water for 15 mins., but if the cooking is prolonged to 1 hr. the material can no longer ward off scurvy. Various methods of drying potatoes were tried, but all caused a reduction in the antiscorbutic factor, which was still further reduced by boiling the product with water for 15 mins. Potatoes baked at 204° C. and then dried at 35°—40° C. gave the most satisfactory results, in that 2.5 g. of material daily afforded the guinea-pigs protection against scurvy.—W. G.

Vitamine; Occurrence of water-soluble — in some common fruits. T. B. Osborne, L. B. Mendel, and A. J. Wakeman. *J. Biol. Chem.*, 1920, 42, 465—489.

The fresh juices of the edible parts of the orange, lemon, and grape-fruit contain water-soluble B vitamin, being about equivalent in this respect volume for volume with cow's milk. Grape juice contains less of this vitamin. These juices may be suitably dehydrated on starch without losing their efficiency. The edible portions of apples and pears furnish some water-soluble B, but from a comparative standpoint they cannot be regarded as rich in this factor. Prunes are somewhat richer than apples and pears. From preliminary experiments it is doubtful whether lemon or grape-fruit juice contains more than traces, if any, of the fat-soluble vitamin, which, however, appears to be present in orange juice.—W. G.

Fat-soluble growth-promoting substance in lard and cottonseed oil. A. L. Daniels and R. Loughlin. *J. Biol. Chem.*, 1920, 42, 359—362.

Born lard and cottonseed oil apparently contain appreciable amounts of the fat-soluble growth-promoting substance.—W. G.

Fat-soluble factor of cabbage and carrot; Extraction of the — by solvents. S. S. Zilva. *Biochem. J.*, 1920, 14, 494—501.

Alcohol extracts the fat-soluble factor from both cabbage and carrots. In addition, such an extract from carrots contains the antineuritic and, to a lesser extent, the antiscorbutic factors. An amount of such extract equivalent to 10—12 g. of fresh carrots given daily is sufficient to promote normal

growth in rats subsisting on a diet lacking the fat-soluble factor. An ethereal extract of the alcoholic extract equivalent to 25 g. of fresh carrots promotes recovery and renews growth in rats declining in weight owing to a deficiency in fat-soluble factor.—W. G.

Apples; Odeorous constituents of —. Emanation of acetaldehyde from the ripe fruit. F. B. Power and V. K. Chesnut. *J. Amer. Chem. Soc.*, 1920, 42, 1509—1526.

The odorous constituents from the parings of different varieties of apples consist essentially of the amyl esters of formic, acetic, and caproic acids, with a very small amount of the caprylic ester. In addition there is a considerable proportion of acetaldehyde and probably some free acids. The aqueous distillate from fresh parings contains exceedingly small amounts of methyl and ethyl alcohols and furfural, the latter probably being a product of some chemical change during distillation. There is no evidence of the presence of amyl valerate. Acetaldehyde is present in the exhalations of ripe apples.—W. G.

Lupins; Loss of nutritive matters and alkaloid in the treatment of — by Löhnert's method for removing bitter substances and by the method of cold aqueous extraction. W. Dietrich and H. Jankon. *Woch. Brau.*, 1920, 37, 203—205, 211—212.

EXTRACTION of seeds of the yellow lupin in a continuous current of cold water for 72 hrs. removed only 26% of the alkaloid originally present. This treatment is therefore useless. Löhnert's method was carried out as follows:—500 g. of the seeds, without previous steeping, was boiled with 2 l. of water for 2 hrs.; the water was then replaced by 1 l. of fresh cold water, which was renewed twice within the 3 days following. This treatment removed 84% of the alkaloid originally present. Löhnert's process, like Kellner's, yields a satisfactory fodder rich in protein. Since neither of these processes, however, removes the alkaloid entirely, horses should not be given more than 1 kg. (dry substance) of the treated lupins per head per day, as they are very sensitive to alkaloids.—J. H. L.

Amino-acids etc. Foreman. See XXIII.

Arginine. Kochler. See XXIII.

PATENTS.

Meat and other alimentary products; Preservation of —. G. Bernard and E. Schmitt. E.P. 144,531, 3.10.19.

MEAT, fish, or other foodstuff, is chilled at or below 0° C., but not frozen, and afterwards dehydrated *in vacuo* whilst its temperature is maintained at about 15° C. Dehydration may be effected in a vacuum chamber provided with a water jacket which serves to control the temperature. After dehydration, oxygen may be admitted to the vacuum chamber, in order to render the surface of the meat more or less impervious to moisture.—J. H. L.

Tea leaf; Process for firing or drying —. C. G. L. Judge and G. H. Hilliard. E.P. 145,872, 1.4.19.

THE prepared leaf is submitted to the action of hot air under pressure.

Margarine; Method of crystallising —. A. C. A. Zeuthen and M. Larsen. U.S.P. 1,345,657, 6.7.20. Appl., 3.4.17.

SEE E.P. 110,872 of 1917; J., 1917, 1286.

Cocoa; Process of roasting — [by indirect heating], and apparatus for the purpose. L. A. Lévy. E.P. 148,082, 20.10.19.

XIXB.—WATER PURIFICATION; SANITATION.

Toxicity of organic compounds to wireworms; Influence of chemical constitution on the —. F. Tattersfield and A. W. R. Roberts. *J. Agric. Sci.*, 1920, 10, 199—232.

THE substances were tested in the form of vapour diluted with air. Aromatic hydrocarbons and their halides are, on the whole, more toxic than aliphatic hydrocarbons and their halides. When introduced singly into the benzene ring the substituents which influence toxicity most may be arranged in descending order of effectiveness as methylamino, dimethylamino, hydroxy, nitro, amino, iodine, bromine, chlorine, methyl groups. This order is, however, modified if another group is present. Chlorine and hydroxy groups together give rise to highly poisonous substances. The association of chlorine and nitro groups together in chloropicrin gives rise to one of the most toxic substances tested. Compounds with irritating vapours usually have high toxic values, e.g., allyl isothiocyanate, chloropicrin, benzyl chloride, but their toxic values are not closely correlated with their vapour pressures or rates of evaporation. In the case of compounds of the same chemical type there is a fairly close relationship between toxicity and vapour pressure, rate of evaporation, and volatility. In a series of such compounds decrease in vapour pressure and in volatility is associated with increased toxicity. Chemically inert compounds boiling above 170° C. are generally uncertain in their poisonous effect on wireworms. Nearly all organic compounds boiling above 215° C. are uncertain in their action, while those boiling above 245° C. are non-toxic, though these limits depend to some extent on the resistance of the insect, the length of exposure, and the temperature at which the experiment is carried out.—W. G.

Adsorption by charcoal. Lowry and Hulett. See VII.

Diphenylamine-sulphuric acid reagent. Haun. See XXIII.

PATENT.

Sand-filters. N. M. Barron. E.P. 146,577, 3.4.19.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Glycerophosphates; Detection of inorganic phosphate in —. J. L. Lizius. *Pharm. J.*, 1920, 105, 100—101.

WHEN 0.1 g. of a glycerophosphate is treated with 10 c.c. of dilute nitric acid and 10 c.c. of 10% ammonium molybdate solution, the development of a turbidity indicates the presence 0.1%, or more, of inorganic phosphate. A more sensitive reagent (Scott and Plimmer, *J. Physiol.*, 1906, 35, 120) is prepared by adding 80 c.c. of 10% ammonium molybdate solution to 12 c.c. of hydrochloric acid (sp. gr. 1.16) and then introducing, successively, 20 g. of ammonium chloride and 10 c.c. of saturated potassium persulphate solution. Five c.c. of this reagent is mixed with 0.1 g. of the glycerophosphate; a turbidity forms within a few mins. if as little as 0.001% of inorganic phosphate is present. The U.S.P. test is untrustworthy, possibly due to the fact that an excess of molybdate reagent is not used.—W. P. S.

Acetylsalicylic acid; Determination of free acetic acid in —. A. N. Smith. *Pharm. J.*, 1920, 105, 90—94.

ONE gram of the sample is spread in a thin layer

on the surface of a piece of "robe muslin" tied over the top of a funnel of 3.5 in. diam.; the stem of the funnel fits through a hole in a ground-glass plate and the whole is covered with a bell-jar. A tube from the top of the bell-jar is connected with a vessel containing water (previously boiled), and this in turn with a water-pump by which a current of pure air is aspirated through the apparatus. The air enters through the stem of the funnel, passes through the layer of acetylsalicylic acid and then bubbles through the water; the acetic acid is absorbed and is determined by titration. Free salicylic acid does not volatilise under these conditions.—W. P. S.

Diazo compounds; New catalytic elements for the transformation of —. A. Korczynski, W. Mrozinski and W. Vielau. *Comptes rend.*, 1920, 171, 182—184.

NICKEL and cobalt salts may be used equally well in place of copper salts for certain of the changes brought about by the Sandmeyer reaction. Thus nickel salts may be used for the preparation of nitriles, and cobalt salts for the preparation of thiocyanates from the corresponding diazonium salts. Zinc and iron salts showed no sign of any catalytic action for these changes. (*Cf.* J.C.S., Sept.)—W. G.

Nitriles; Catalytic hydration of —. A. Mailhe. *Comptes rend.*, 1920, 171, 245—247.

NITRILES may be converted into the corresponding acids by passing their vapours along with steam over thoria at 420° C.—W. G.

Catalytic oxidation of alcohols by the use of metallic oxides and of finely divided metals. J. B. Senderens. *Ann. Chim.*, 1920, 13, 266—283.

NONE of the oxides tried (nickel, copper, vanadium, molybdenum, and zinc oxides) was satisfactory as catalyst in the oxidation of alcohols to aldehydes or acids. Of the metals tried in the form of turnings or powder, silver was the only one which gave at all satisfactory results.—W. G.

Formaldehyde; Degree of alkalinity necessary for the phloroglucinol test for —. P. J. Hanzlik. *J. Biol. Chem.*, 1920, 42, 411—413.

THE minimum alkalinity necessary for a positive reaction in the phloroglucinol test for formaldehyde is equivalent to $pH=12.13$, or $N/100$ sodium hydroxide. The optimum alkalinity is $pH=13.0$ or $N/10$.—W. G.

Cineol; Determination of — in eucalyptus oils. T. T. Cocking. *Pharm. J.*, 1920, 105, 81—82.

THREE grams of the eucalyptus oil is mixed with 2.1 g. of o-cresol and the solidifying pt. of the mixture is determined. Reference to a graph, constructed from the results obtained with mixtures containing known amounts of cineol, gives the percentage of cineol corresponding with the solidif. pt. found.—W. P. S.

"Cresineol," a compound of cineol and o-cresol. T. T. Cocking. *Pharm. J.*, 1920, 105, 81.

CINEOL and o-cresol combine in molecular proportions to form a crystalline product termed "cresineol." m. p. 55.2° C., sp. gr. at 60°/60° C. 0.9661, $n_D^{20}=1.4846$; this substance is soluble in organic solvents generally and is decomposed into its constituents by alkali hydroxides.—W. P. S.

See also pages (A) 596, *Mercuric oxycyanide* (Jones), *Action of finely-divided gases* (Zenghelis); 611, *Fluorometry* (Desha), *Organic acids etc.* (Foreman), *Oxalic acid* (Ban), *Methyl alcohol* (Gettler).

PATENTS.

Camphor; Process of oxidising iso-borneol to —. R. L. Andreau, Assr. to E. L. du Pont de Nemours and Co. U.S.P. 1,347,071, 20.7.20. Appl., 12.11.17.

ISOBORNEOL is oxidised to camphor by means of mixed nitric and sulphuric acids.—A. de W.

Tuberculosis, leprosy, and other diseases; Substance for treatment of —. R. S. Paschall. E.P. 116,078, 5.4.18. Conv., 17.11.17.

SEE U.S.P. 1,250,345 of 1917; J., 1918, 106A.

Mercurio-amino combinations; Process of producing —. A. Eckmann, Assr. to Schweiz. Serumund Impfinstitut. U.S.P. 1,347,083, 20.7.20. Appl., 29.6.17.

SEE G.P. 307,893 of 1916; J., 1919, 28A.

Catalysis. E.P. 126,279. See I.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENT.

Colour photography. Natural Colour Pictures Co., Assees. of W. F. Fox. E.P. 143,180, 14.10.19. Conv., 12.5.19.

PRINTS are made in register from two complementary colour-sensation negatives on opposite sides respectively of a transparent base sensitised on both sides. Both prints are toned to a colour corresponding to that through which one of the negatives was taken, then the positive printed through that negative is toned in a solution which will change the tone already obtained to its complementary. For example, if a red and a green filter have been used in taking the negatives, the first toning bath may be a uranium ferri cyanide solution and the second toning bath, applied to the positive from the "red" negative, may be a ferric ammonium oxalate solution. In the second toning operation, the solution may be applied to one side by a suitable rolling or brushing device, or the side not required to be toned may be protected by a waterproof coating.—B. V. S.

XXIII.—ANALYSIS.

Melting point curves; Estimation of impurities by means of —. W. P. White. J. Phys. Chem., 1920, 24, 393—416.

FREEZING points are more accurately determined by immersing the thermometer in the substance than by the capillary tube method. If in addition the form of the melting point curve is observed, an indication of the amount of impurity present is obtained, which is independent of all previous knowledge or uncertainty as to the melting point of the pure substance and even of the absolute accuracy of the thermometer. Small tubes diminish local temperature differences and are often a superior substitute for a stirring rod. A small thermocouple may also with advantage be used instead of a thermometer.—J. F. S.

Fluoremetry. Quantitative analysis by comparative fluorescence. L. J. Desha. J. Amer. Chem. Soc., 1920, 42, 1350—1363.

A NEW method of micro-analysis, of the same order of sensitiveness as colorimetry and nephelometry, is described. This should prove generally applicable to the estimation of minute quantities of substances, which are either fluorescent themselves or may be rendered so by the addition of a suitable reagent. Ultraviolet rays from a quartz mercury lamp, from which most of the visible radiations

have been removed by means of a colour filter, are used to excite fluorescence in solutions of such substances contained in the comparison tubes of the Kober nephelometer. The intensity of the fluorescent light thus produced, as observed in the eyepiece of the instrument, is equalised in the usual manner by altering the heights of the exposed columns. For sufficiently dilute solutions the curves obtained by plotting the scale readings against concentrations are quite regular. Calibration curves for solutions containing 0.5—2.0 mg. per l. of quinine sulphate in N/1 sulphuric acid and for solutions of 2—4 mg. per l. of Anilino Iodococin in N/10 alkali are given.—J. F. S.

Methyl orange as indicator. V. Macri. Boll. Chim. Farm., 1920, 59, 193—196.

CHLORINE in either acid or alkaline solution destroys methyl orange and bromine acts similarly, whereas iodine is without effect. Hydrosulphurous acid destroys methyl orange, but not in presence of formaldehyde; the destruction caused by certain alkali sulphides depends on the presence of accidental impurities. Of oxygen-yielding compounds, hydrogen peroxide is without action on methyl orange, but ozone readily destroys it. In the alkalimetric titration of percarbonate the indicator is destroyed immediately any free alkali carbonate is completely neutralised and the percarbonate begins to undergo decomposition. The indicator is readily destroyed by nitrous acid but serves in titrating cyanic, thiocyanic, and carbonylferrocyanic acids; with hydrocyanic acid it is insensitive and with ferrocyanic acid uncertain. Certain bases, notably ferrous and manganous oxides, destroy it, the yellow colour persisting when a solution of a ferrous or manganous salt, precipitated by addition of alkali, is re-acidified. This phenomenon, due to the reducing action of the two hydroxides, is not observed if hydrogen peroxide is added prior to the alkali. Both the free and the combined acid in solutions of ferrous and manganous salts may hence be determined by titration. With ferrous salts it is necessary to allow for the free acid entering into combination in accordance with the equation: $2\text{FeSO}_4 + n\text{H}_2\text{SO}_4 + \text{O} = \text{Fe}_2(\text{SO}_4)_3 + (n-1)\text{H}_2\text{SO}_4 + \text{H}_2\text{O}$; with manganous salts, however, manganic salts are not formed under ordinary conditions. Stannous chloride also destroys the indicator, but only in acid solution, and here, too, when hydrogen peroxide is used, the free acid combining to form the stannic salt must be taken into account. Copper salts behave like those of tin, and cobalt salts like those of iron and manganese. The mercurous oxide precipitated from the nitrate by addition of alkali does not destroy the coloration but carries down the colouring matter with it. In the titration of commercial and fuming sulphuric acid methyl orange is sometimes destroyed owing to the impurities present, and the same is observed with alkali hydroxides, even with those sold as pure. Ethyl alcohol is markedly alkaline towards methyl orange, whereas methyl alcohol is without effect.—T. H. P.

Methyl red; Preparation of —. L. Desvergnès. Ann. Chim. Analyt., 1920, 2, 209—210.

A MIXTURE of anthranilic acid, 137, water, 1000, crushed ice, 300 g., and hydrochloric acid (sp. gr. 1.18), 222 c.c., is stirred and kept below 5° C., while a mixture of sodium nitrite, 59, and water, 150 g., is added. The whole is stirred for a further 20 mins. and then added to a mixture of dimethylaniline, 121, water, 200 g., and hydrochloric acid (sp. gr. 1.15), 90 c.c. After a few mins., a mixture of fused sodium acetate, 165, and water, 500 g., is added. The purple red crystals which separate when the mixture is set aside for some hrs. are collected, washed with cold water, dried at 50° C., and recrystallised from alcohol. The yield of the crude product is about 43% of the theoretical amount calculated on the anthranilic acid.—W. P. S.

Sodium thiosulphate, arsenious acid, potassium bromate, potassium iodate, etc. solutions; Standardisation of — by means of standard sulphuric acid solution. L. Bertiaux. *Ann. Chim. Analyt.*, 1920, 2, 207—208.

THE equivalent quantity of iodine liberated from a mixture of potassium iodate and iodide by a definite volume of standard sulphuric acid solution may be used for the standardisation of thiosulphate or arsenious acid solutions. The latter solutions then serve for the standardisation of bromate or iodate solutions.—W. P. S.

Titration with the hydrogen electrode. W. D. Treadwell and L. Weiss. *Helv. Chim. Acta*, 1920, 3, 433—446.

A HYDROGEN electrode is described which is highly stable and readily renewed, and serves for the titration of both strong and weak acids.—T. H. P.

Potassium; Determination of — and its separation from sodium by means of sodium cobaltinitrite. P. Wenger and C. Hémen. *Ann. Chim. Analyt.*, 1920, 2, 198—199.

POTASSIUM cobaltinitrite precipitate obtained in the usual way has the composition $K_2NaCo(NO_2)_6$, and the author recommends a determination of the cobalt as a measure of the potassium present. The precipitate is dissolved in hydrochloric acid, the solution treated with sodium hydroxide, the precipitated cobalt hydroxide collected, reduced to metallic cobalt, and weighed. Alternatively, the cobalt may be determined electrolytically in the hydrochloric or sulphuric acid solution of the precipitate.—W. P. S.

Lead; Determination of — in acid solution. N. Evers. *Pharm. J.*, 1920, 105, 85—86.

THE sulphide colorations obtained in the determination of small quantities of lead are not comparable unless the solutions have the same hydrogen ion concentration; the author recommends neutralisation of the test and comparison solutions before the addition of hydrogen sulphide. Bromophenol-blue is used as indicator. Iron does not give a coloration under these conditions, and the interference of copper may be prevented by the addition of cyanide. If both iron and copper are present, the original solution should be treated with alum and ammonia, the aluminium hydroxide precipitate (this also contains the lead whilst the copper remains in solution) collected, dissolved in dilute acid, and this solution used for the determination of the lead.—W. P. S.

Manganese; Estimation of — in biological material, together with data on the manganese content of human blood and tissues. C. K. Reiman and A. S. Minot. *J. Biol. Chem.*, 1920, 42, 329—345.

FOR the preliminary conversion of manganese in the blood or tissue into manganese sulphate Bertrand's method (*J.*, 1911, 650) of fusion with potassium bisulphate is modified, the fusion being conducted in quartz beakers at 500°—600° C. The beakers are liable to contain manganese and must first be tested by a preliminary fusion. For the conversion of the manganese sulphate into potassium permanganate, treatment with potassium persulphate is recommended, Bertrand's modification (*loc. cit.*) of Marshall's method (*Chem. News*, 1901, 83, 76) being further modified.—W. G.

Manganese; The use of phenolphthalein and diphenylamine in the persulphate method for the determination of —. D. H. Wester. *Rec. Trav. Chim.*, 1920, 39, 600—602. (*Cf. J.*, 1920, 469 A.)

THE author was unable to obtain accurate results by the use of phenolphthalein in alkaline solution

as a comparison liquid, or by the use of diphenylamine for intensifying the colour as recommended by Tillmanns and Mildner (*J.*, 1914, 709).—W. G.

Barium sulphate; Rapid identification of the two ionic elements of —. G. Deniges. *Bull. Soc. Chim.*, 1920, 27, 560—564.

A FULLER account of work already published (*cf. J.*, 1920, 469 A).—W. G.

Diphenylamine - sulphuric acid reagent [for nitrates]; Preparation of —. F. Haun. *Z. Unters. Nahr. Genussm.*, 1920, 39, 355—356.

SULPHURIC acid which yields a reaction for nitric acid with the brucine test is unsuitable for the preparation of diphenylamine reagent. Occasionally a blue coloration, which develops in a reagent made with sulphuric acid free from nitric acid, may be due to the presence of traces of ferric salts; if the acid is heated to boiling the ferric salts are reduced and no longer interfere. By adding a small quantity of permanganate to the acid before it is heated, the reduction of the ferric salts may be followed, since the permanganate is reduced at the same time and a colourless acid results. The presence of manganese in the reagent does not interfere and in some cases accelerates the reaction between the reagent and nitrates.—W. P. S.

Hydrocyanic acid; Determination of —. R. L. Morris. *Pharm. J.*, 1920, 105, 83—85.

IN applying Liebig's method to the determination of free hydrocyanic acid, the latter must be neutralised previously and only a slight excess of alkali must be present. Borax solution may be used in place of alkali hydroxide for the neutralisation (Guerin, *J.*, 1905, 1258), and an excess of borax does not interfere. The iodometric method proposed by Fordos and Gelis (*cf. Guerin, loc. cit.*) tends to give low results. In the B.P. (1914) method, based on Deniges' modification of Liebig's process (*cf. J.*, 1894, 664), to obtain accurate results it appears to be necessary to add about four times the prescribed quantity of potassium iodide.—W. P. S.

Recording [gas] calorimeters; Differential air thermometers and —. H. A. Madsen and A. Herber. *Gas J.*, 1920, 151, 338—339.

THE principle of the differential air thermometer is applied to the construction of a recording gas calorimeter. The long tube connecting the two bulbs of the differential air thermometer is bent into a ring or spiral which is balanced on friction wheels. The lower part of the ring is filled with mercury. One bulb is exposed to the hot products of combustion arising from a flame due to the combustion of gas at a controlled rate, the air supply for combustion and the supply of air to be heated being separated. The latter supply is automatically controlled by means of a damper. A pen is affixed to a lever attached to the horizontal portion of the tube connecting the bulbs, and serves, after calibration of the instrument, to record the calorific value of the gas on a drum.—J. S. G. T.

Gas analysis apparatus accurate to 0.001% mainly designed for respiratory exchange work. A. Krogh. *Biochem. J.*, 1920, 14, 267—281.

TWO improvements are introduced into the usual apparatus. Three separate gas burettes are used, the first for moving the air to and from the absorption pipettes, the second for measuring the air after the absorption of carbon dioxide, and the third for measuring the air after the absorption of oxygen. The mercury is raised and lowered in the burettes by means of air pressure.—W. G.

Carbon dioxide in flue gases etc.; Continuous electrolytic determination of — and automatic regulation of the draught in accordance with the analysis. K. von Haken. *Z. angew. Chem.*, 1920, 33, 188.

The gases are passed through an ordinary purifying apparatus, and enter the electrolytic apparatus through a valve controlled by a sensitive differential manometer charged with two immiscible liquids of different sp. gr., and regulated by a float. They are then mixed with a standard solution of sodium hydroxide to remove the carbon dioxide, and passed through a second differential manometer, which regulates the pressure in the mixing vessel in the reverse direction. The solution of sodium bicarbonate produced flows into the electrolytic vessel which has a regulated overflow, and is electrolysed with an alternating current, the current of the secondary coil being conducted through a milli-ampere-meter or similar device, which shows directly the amount of CO_2 in the gas. The method is based on the difference in the conductivity of the original sodium hydroxide solution and of that containing the dissociated salts and acid salts after the absorption of the CO_2 , and is sensitive to fractions of 0.1%. The draught is regulated by an automatic shutter, the aperture of which varies with the variation in the amount of CO_2 .—C. A. M.

Organic substances; Incineration of — prior to analysis for the mineral elements which they contain; application to the analysis of blood. A. Desgrez and J. Meunier. *Comptes rend.*, 1920, 171, 179—182.

For the preparation from organic material of an ash suitable for subsequent analysis it is advisable to conduct the incineration in two stages. The dry material is first ignited and allowed to burn at as low a temperature as possible. In this way a carbonised material is obtained which on further incineration readily becomes incandescent and burns to a white ash. If excess of alkali salts is present these should be extracted with water before the final incineration. By means of this method the presence of copper, manganese, and lithium in blood was detected.—W. G.

Capillary active and inactive modifications of the higher homologues of the fatty acid series and their relation to titration with capillary active substances as indicators. W. Windisch and W. Dietrich. *Woch. Brau.*, 1920, 37, 215—217, 225—228. (*Cf. J.*, 1920, 128A.)

UNDECYLIC, nonyllic, and capric acids may exist in capillary inactive as well as capillary active forms. In a state of molecular dispersion, and in a relatively coarse suspensoid state, the acids are inactive, but in an intermediate colloidal state they are active. Solutions of the pure fatty acids might be used as indicators in place of their sodium salts, but the latter are preferable in most cases, because in solution the free fatty acids become inactive in the course of a few days, i.e., the surface tension of their solutions gradually approximates to that of pure water, probably owing to the colloidal particles increasing in size beyond the optimum.—J. H. L.

Amino-acids, organic acids, and organic bases; Rapid volumetric methods for the estimation of —. F. W. Foreman. *Biochem. J.*, 1920, 14, 451—473.

The method, which is applicable to the estimation of organic acids, and amino-acids except arginine, is as follows:—(1) 5—10 c.c. of the aqueous solution is titrated with $N/10$ -aqueous sodium hydroxide. (2) 10 vols. of 97% alcohol is added to 5—10 c.c. of the aqueous solution and the mixture is titrated with $N/10$ -alcoholic potassium hydroxide. (3) To the liquid from titration (2) 12.5 c.c. of neutral 13% aqueous formaldehyde is added for every 50 c.c.

of alcohol present and the titration is continued to the same end-point. Phenolphthalein is used in all cases as the indicator. Titration (1) gives useful information when dibasic amino-acids are present. Titration (2) gives the correct estimation of a number of amino-acids. In titration (3) the carboxyl groups of all the amino-acids in an amino-acid mixture, except that of arginine, are estimated. The method is based on the fact that ammonia, primary, secondary, and tertiary amines and basic methylene derivatives of secondary amines do not form ionisable compounds with phenolphthalein in aqueous-alcoholic solution providing the concentration of alcohol is above 80%, and also that, under similar conditions, the amino- or imino-groups of certain amino-acids show no basicity to phenolphthalein. For the estimation of volatile bases and amino-acids in "alcoholic extracts" (Foreman and Graham-Smith, *J. Hyg.*, 1917, 16, 144) to an aliquot portion of the extract the amount of alcoholic alkali necessary for neutralisation as determined by titration (3) is added and the mixture is distilled with steam free from carbon dioxide for about 5 mins., the distillate being collected in standard acid. The excess acid is titrated, using alizarin as indicator, and thus a measure of the volatile bases is obtained. The residual liquid in the distillation flask is now free from alcohol and, owing to hydrolysis in the water of the alkali salts of the amino-acids, is alkaline. It is titrated with $N/10$ -acid and thus a value is obtained for the amino-acids present in the original "alcoholic extract." (*Cf. J.C.S.*, Sept.)—W. G.

Arginine; Modification of the Van Slyke method for determining —. A. E. Koehler. *J. Biol. Chem.*, 1920, 42, 267—268.

VAN SLYKE's method (*J.*, 1911, 1135) is modified by drawing a slow current of air through the liquid during the whole of the digestion with alkali, the apparatus for absorbing the ammonia being a simple gas wash-bottle.—W. G.

Oxalic acid; Determination of —. III. A. Bau. *Woch. Brau.*, 1920, 37, 201—203, 209—211, 217—219. (*Cf. J.*, 1918, 524 A; 1920, 349 A.)

THE extraction of materials with water or dilute hydrochloric acid, for the determination of oxalic acid, should be carried out in an atmosphere of carbon dioxide to prevent the decomposing action of iron salts in presence of air. A further reason for this procedure is furnished by Stachelin's discovery of the wide distribution, in vegetable materials, of an oxydase capable of decomposing oxalic acid in presence of air (*Biochem. Zeits.*, 1919, 96, 1). Liquids of yellow colour, such as beer or urine, containing oxalic acid, may be exposed to daylight for months without any destruction of oxalic acid occurring. For the preservation of standard oxalic acid solutions Junk's method (*J.*, 1919, 481 A) is probably the best, but the solutions should be kept in yellowish-brown bottles and standardised every three months.—J. H. L.

Methyl alcohol; Critical study of methods for the detection of —. A. O. Gettler. *J. Biol. Chem.*, 1920, 42, 311—328.

A CRITICAL study of 58 tests for the detection of methyl alcohol, 46 being based on the oxidation of the alcohol to formaldehyde and the subsequent detection of this compound, and twelve applied directly to the alcohol after suitable isolation. Of the first series the author recommends five colorimetric tests which can be used to detect one part in 200,000, and two tests in which crystalline products are obtained, these being less sensitive but used for confirmation. The second group of tests require considerably more alcohol and only two are recommended. Full details of typical pro-

cedures for the detection of methyl alcohol in liquors and in tissues are given, and there is a very full bibliography. (*Cf.* J.C.S., Sept.)—W. G.

Ion concentration in ultra filtrates and other protein-free solutions; Determination of the —.
R. Brinkman and E. van Dam. *Proc. K. Akad. Wetensch.* Amsterdam, 1920, 22, 762—771.

THE method depends on the addition of an ion which forms a sparingly soluble salt with the ion, the concentration of which is to be measured. The appearance of a milkiness denotes when the limit of solubility of the sparingly soluble salt is passed. The method gives results correct to 2—3 mg. of calcium per litre.—J. C. D.

See also pages (A) 591, *Tar in gas* (Stone and Prince); 593, *Phenolsulphonic acids* (Desvergnès); 594, *Diazo compounds* (Knecht and Thompson); *Chlorine* (Rona and Michaelis); 595, *Ammonia* (Denigès); 596, *Manganese salts* (Denigès); 598, *Glass to metal joints* (McKelvy and Taylor); 600, *Tungsten etc.*; 601, *Copper* (Hawley); 604, *Linseed oil, Tung oil, Iodine value* (Sundberg and Lundborg); 605, *Tan liquors* (Atkin and Thompson); *Leather* (Chambard); 600, *Protozoa* (Cutler); *Arable soil* (Perotti); 607, *Brewing water* (Heuss); 608, *Flour* (Gerum, Abel), *Water in milk* (Utz), *Vitamine* (Williams); 610, *Glycerophosphates* (Lizius), *Acetylsalicylic acid* (Smith), *Formaldehyde* (Hanzlik), *Cineol* (Cocking).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Alexander (Best, Inc.). Oxidation of finely-divided material. 23,317. Aug. 9.
Atherton. 24,495. *See* XXIII.
Atkinson, and Stein and Atkinson. 24,250. *See* VIII.
Automatic Telephone Manufacturing Co., and Roseby. Arrangements for obtaining high temperatures. 24,331. Aug. 21.
Bennett. 24,892. *See* II.
Bregcat. Means for effecting intimate intermingling of fluid streams. 24,696. Aug. 25.
Budill. Air-filters. 23,876. Aug. 16.
Charles. Utilising waste steam to produce power. 23,708. Aug. 13.
Crawford. Lubricating compound. 23,588. Aug. 12.
Eberts. Drying apparatus. 24,174. Aug. 19.
Elektro-Osmose A.-G. Purifying, dehydrating, or concentrating aqueous solutions of colloids etc. 24,945. Aug. 28. (Germany, 11.9.19.)
Fothergill. Apparatus for removing gases from fluids. 23,993. Aug. 17.
Grace. Centrifugal separators. 23,371. Aug. 9.
Grière. Evaporators. 23,457. Aug. 10.
Griscom-Russell Co. Evaporator feed and overflow regulator. 23,539. Aug. 11. (U.S., 9.2.20.)
Griscom-Russell Co. Combined evaporator and feed-water heating system. 23,877. Aug. 16. (U.S., 26.1.20.)

Griscom-Russell Co. High-heat-level evaporator systems. 23,895, Aug. 16; and 24,291, Aug. 20. (U.S., 26.1.20.)

Hardinge. Mills for grinding and disintegrating. 24,280. Aug. 20. (U.S., 11.9.19.)

Jones and McKean. Heat-exchange apparatus. 24,115. Aug. 19.

Kay. Removing water from rotary driers. 23,586. Aug. 12.

Kestner. Filling-material for Glover towers etc., and means for producing same. 24,052. Aug. 18.

Klingenberg. Filter for separating impurities from gas or steam. 24,873. Aug. 27. (Germany, 4.9.19.)

Lohmann. Mixing and kneading. 23,881. Aug. 16. (U.S., 16.8.19.)

Massy. 23,672. *See* II.

Mather. Stills. 24,095. Aug. 18.

Mauss. Vacuum filters. 24,079. Aug. 18.

Mellersb-Jackson (Fuller-Lehigh Co.). Cylindrical kilns. 24,373. Aug. 21.

Morton and Morton. Gas-fired kilns. 24,890. Aug. 28.

O'Dell (Canadian-American Finance and Trading Co.). Distillation process. 24,481. Aug. 23.

O'Dell (Canadian-American Finance and Trading Co.). Mechanism for diffusing gases and vapours through liquids. 24,482. Aug. 23.

Oldham. Grinding mills. 24,206. Aug. 20.

Orcutt. Grinding-machines. 23,784-7. Aug. 14.

Pell. Grinding etc. mills. 24,468. Aug. 23.

Porteus. Air-purifying apparatus. 24,216. Aug. 20.

Ratel. Furnace. 24,923. Aug. 28. (France, 28.8.19.)

Sharples. Refining liquids. 23,355. Aug. 9.

Smits. Method of heating apparatus. 23,615. Aug. 12. (Germany, 5.5.19.)

Spuhr. Steam-drier. 24,298. Aug. 20.

Stein et Cie. 23,987. *See* X.

Thompson (Soc. Anon. Fours et Proc. Mathy). Regenerative furnaces. 23,497. Aug. 11.

Von Codelli and Von Köppen. Cooling apparatus. 24,584. Aug. 24.

Wiegand. Continuously-acting evaporation. 23,492. Aug. 11.

Wilhelm. Surface apparatus for effecting transfer of heat. 24,378. Aug. 21. (Germany, 19.9.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

17,254 (1918). Vergniaud. *See* VIII.
2764 (1919). Gregory. *See* II.
10,662 (1919). Cox. Appliances for filtering or, and mixing oils and other liquids. (149,038.) Aug. 18.
10,979 (1919). Lee, Flockton, and Manlove, Allott, and Co. Roller mills. (149,048.) Aug. 18.
11,092 (1919). Boberg, and Techno-Chemical Laboratories. Drying processes, and apparatus therefor. (149,055.) Aug. 18.
11,289 (1919). Harter. Temperature exchanging apparatus. (127,565.) Aug. 25.
12,111 (1919). Merz and McLellan, Michie, and Weeks. Retorts and the like. (149,733.) Sept. 1.
12,174 (1919). Dressler Tunnel Ovens, and Dressler. Tunnel ovens and like structures. (149,805.) Aug. 18.
12,175 (1919). Dressler Tunnel Ovens, and Dressler. *See* II.
12,304 (1919). Brealey. Centrifugal disintegrating machines. (149,751.) Sept. 1.
12,323 (1919). Robinson and Son, Robinson, and Stevenson. Apparatus for separating solid particles from air employed for carrying off said particles from machinery, or processes in which they are produced or given off. (149,089.) Aug. 18.
12,479 (1919). Halliwell. Apparatus for indicating or recording continuously the specific gravity of any fluid. (149,448.) Aug. 25.

12,602 (1919). Cloudsley. Drying-apparatus. (149,774.) Sept. 1.
 12,652 (1919). Johnson (Allan). Apparatus for distilling and fractionating liquids. (149,776.) Sept. 1.
 13,540 (1919). Wood and Hall. Condensers for refrigerating plant. (149,797.) Sept. 1.
 14,210 (1919). Carpmals (Davis). Filters. (149,808.) Sept. 1.
 15,843 (1919). Smallwood. Device or apparatus for conveying or transporting goods, particularly as a device for charging furnaces. (149,195.) Aug. 25.
 18,048 (1919). Wellman Smith Owen Engineering Corporation, and Kemp. *See* X.
 20,305 (1919). Ehrhart. Surface condensers. (131,595.) Aug. 18.
 20,612 (1919). Morton. Mixing or heating liquids or semi-liquid substances. (149,136.) Aug. 18.
 25,206 (1919). Denoel. Decantation apparatus. (149,551.) Aug. 25.
 27,427 (1919). Ehrhart. Surface condensers. (131,852.) Aug. 25.
 32,370 (1919). Smith and Stephenson. Heat-exchange apparatus. (149,882.) Sept. 1.
 13,058 (1920). Chantaine. Recuperators or regenerators for furnaces. (143,244.) Aug. 25.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Angold and Railing. Electric arc lamps. 23,479. Aug. 11.
 Bamber and Parker. Means for supplying air and steam to producer-gas plants. 23,648. Aug. 12.
 Barrele. Manufacture of coke for metallurgical purposes. 24,270. Aug. 20.
 Barrett Co. Manufacture of coke. 23,455. Aug. 10. (U.S., 10.12.19.)
 Bennett. Mechanically-operated grates for gas-producers and furnaces. 24,892. Aug. 28.
 British Thomson-Houston Co. (General Electric Co.) Electric incandescent arc devices. 24,546. Aug. 24.
 Brown. Regenerative coke-ovens. 23,545. Aug. 11.
 Charpy. Coke-oven. 23,809. Aug. 14. (France, 10.9.19.)
 Coke and Gas Ovens, Ltd., and Smee. Gas burners of coke-ovens. 23,622. Aug. 12.
 De Beaufort and Claude. Manufacture and working of luminous tubes. 24,190. Aug. 19. (France, 29.8.19.)
 Edser. Fuel. 23,825. Aug. 14.
 Farbw. vorm. Meister, Lucius, u. Brünig. 23,446. *See* XX.
 Filippo, Lely, and N. V. Philips Gloeilampen-fabrieken. Electric discharge lamps. 24,835. Aug. 27. (Holland, 27.8.19.)
 Fischer. Process for converting combustible substances into soluble organic compounds. 23,354. Aug. 9. (Germany, 10.6.19.)
 France. Coal etc. washing apparatus. 24,195. Aug. 19. (Belgium, 20.8.19.)
 France. Filament electric lamps. 24,018. Aug. 18.
 General Electric Co., and Ryde. Electric incandescent lamps. 24,660. Aug. 25.
 Heenan. 23,507. *See* XII.
 Hilger. Operating gas-producers. 23,869. Aug. 16. (Germany, 11.8.19.)
 Jackson (Sperry Gyroscopo Co.). Arc lamps. 23,552. Aug. 11.

Keillor. Combination plant for manufacture of coal gas, blue-water gas, and coal-tar or oil-gas. 23,700. Aug. 13.
 Massy. Appliances for conversion of fuel energy for heating or power. 23,672. Aug. 13.
 Morton and Morton. 24,890. *See* I.
 Noding. 24,217-9. *See* VII.
 O'Dell. 24,480. *See* III.
 Parr and Pearson. Process for desulphurising carbonaceous material. 24,432. Aug. 23.
 Roberts. Coking coal. 24,338. Aug. 21.
 Stephens (Canadian-American Finance and Trading Co.). Process for volatilising and decomposing hydrocarbons. 23,357. Aug. 9.
 Stephens (Canadian-American Finance and Trading Co.). Process for treating hydrocarbons. 23,646. Aug. 12.
 Stephens (Canadian-American Finance and Trading Co.). Process for saturating unsaturated compounds. 23,647. Aug. 12.
 Turner. Electric generator lamps. 24,607. Aug. 25.
 Wells. Gas producers. 23,992. Aug. 17.

COMPLETE SPECIFICATIONS ACCEPTED.

6139 (1914). British Thomson-Houston Co. (General Electric Co.). Filaments for incandescent electric lamps and methods of making the same. Sept. 1.
 2764 (1919). Gregory. Means for generating and storing combustion products under pressure for use in turbines and other engines. (149,367.) Aug. 25.
 11,420 (1919). Marks (U.S. Industrial Alcohol Co.). Process of producing liquid fuel and product thereof. (149,398.) Aug. 25.
 11,773 (1919). Doherty. Process and apparatus for producing water-gas and for carbonising coal. (132,488.) Aug. 25.
 12,175 (1919). Dressler Tunnel Ovens, Ltd., and Dressler. Treatment of coal and other materials capable of being distilled, vaporised, or decomposed by heat. (149,086.) Aug. 18.
 12,317 and 26,640 (1919). Brown and Pickston. *See* XXIII.
 12,393 (1919). Markle. Fuel and process of forming the same. (126,972.) Sept. 1.
 12,396 (1919). Bentley and Appleby. Gas-producers. (149,759.) Sept. 1.
 12,585 (1919). Wells. Process and apparatus for carbonising wood and other carbonaceous material. (149,773.) Sept. 1.
 12,621 (1919). Armstrong. Manufacture of fuel, and apparatus therefor. (149,449.) Aug. 25.
 13,225 (1919.) Milton. Gas producers. (149,790.) Sept. 1.
 27,370 (1919). Ulmann. Process and apparatus for the treatment of peat-mass and for the production of turf or peat briquettes. (149,869.) Sept. 1.
 29,003 (1919). Pintsch A.-G. Porous charges for containers for the storage of acetylene or like explosive gases dissolved in liquids. (135,511.) Aug. 25.
 14,775 (1920). British Thomson-Houston Co. (General Electric Co.). Manufacture of incandescent electric lamps. (149,605.) Aug. 25.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

Anderson and Meikle. Process of treating mineral tars other than coal tar. 23,569. Aug. 12.
 Barrett Co. 23,454. *See* XIII.
 Commin. Covering of pitch composition. 23,296. Aug. 9.
 Fischer. 23,422. *See* XIII.
 O'Dell. Hydrogenating bituminous substances. 24,480. Aug. 23.
 Schroeter, and Tetralin Ges. Hydrogenation of naphthalene. 23,530. Aug. 11.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

Carpmael (Farbenfabr. vorm. F. Bayer und Co.). Manufacture of soluble chromium lakes of azo dyes. 23,551. Aug. 11.

Cassella und Co. Manufacture of dye-stuffs. 24,877. Aug. 27. (Germany, 27.8.19.)

COMPLETE SPECIFICATION ACCEPTED.

25,916 (1919). Imray (Soc. of Chem. Ind. in Basle). Manufacture of azo-dyestuffs and intermediate products therefor. (149,553.) Aug. 25.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Bearder. Manufacture of cellulose-acetate filaments. 24,594. Aug. 25.

Billings. Fibrous product, and process for treating eucalypti barks and bass fibres for use in manufactures. 23,716. Aug. 13.

Brougham (Technochemia A.-G.). Manufacture of artificial silk etc. 24,277. Aug. 20.

Denington and Perry. 24,746. *See* XVIII.

Duvinae and Muth. 23,960. *See* VII.

Jackson (Tanners Products Co.). Manufacture of felt. 23,434. Aug. 10.

Meldia Manufacturing Co., and Melsom. Manufacture of flexible waterproof and gas-proof material. 23,364. Aug. 9.

Raitt. Isolating or extracting cellulose or paper pulp from fibrous vegetable materials. 23,864. Aug. 16.

Whittaker. Manufacture of paper. 24,266—7. Aug. 20.

COMPLETE SPECIFICATIONS ACCEPTED.

4451 (1914). Frankel & Runge, and Colombek. Manufacture of waterproof material. Sept. 1.

18,938 (1917). Wood and Russell. Paper-making machines. (149,359.) Aug. 25.

13,226 (1919). Pilkington, and Mandleberg and Co. Proofing of fabrics. (149,791.) Sept. 1.

31,190 (1919). Gillet et Fils. Treating vegetable fibres. (136,569.) Aug. 25.

31,790 (1919). Great Northern Paper Co. Paper-making. (136,839.) Aug. 18.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Brandwood and Brandwood. Dyeing, bleaching, etc. textile fibres etc. 24,423. Aug. 23.

De Blicquy and Callebaut. Dye-vats etc. 24,823. Aug. 27.

Farbw. vorm. Meister, Lucius, und Brüning. Producing fast dyeings on cotton etc. 24,303. Aug. 20. (Germany, 30.5.14.)

Winslow. Apparatus for dyeing yarn in cop, cheese etc. form. 24,330. Aug. 21.

COMPLETE SPECIFICATION ACCEPTED.

11,958 (1919). McMyn. Application of dyestuffs containing the sulphonamide group to dyeing and printing. (149,428.) Aug. 25.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Alby United Carbide Factories, and Mitchley. Production of calcium carbide. 23,425. Aug. 10.

Amsterdamsche Superfosfaatfabriek, and Hirschel. Manufacture of phosphoric acid. 24,193. Aug. 19. (Holland, 24.6.20.)

Duvinae and Muth. Process for preparing compounds of alumina for sizing paper etc. 23,960. Aug. 17. (Germany, 18.8.19.)

Heinemann, and Hoesch & Co. Process for recovery of pure caustic alkalis from impure lyes. 23,818. Aug. 14.

Holden and Pandrich. Utilisation of nitre cake and waste liquor from the ammonia-soda process. 24,937. Aug. 28.

Kestner. 24,052. *See* I.

Macfarlane and Spink. 24,529. *See* XI.

Nöding. Methods of obtaining hydrogen. 24,247. 24,249. Aug. 20.

Nöding. Retort furnace for generation of hydrogen. 24,248. Aug. 20.

Noef. Recovery of sulphur from sulphuretted hydrogen and ammonium sulphide and gases containing such. 24,387. Aug. 23.

Norske Aktieselskab for Elektrokemisk Industri Norsk Industri-Hypotekbank. Process for production of alumina from aluminium nitrate solutions. 24,847. Aug. 27. (Norway, 19.9.19.)

Tyrer. Extraction of alumina 24,579. Aug. 24.

COMPLETE SPECIFICATIONS ACCEPTED.

3754 (1919). Freeth and Munro. Removal of calcium and magnesium from rock salt, brine, or solutions of rock salt in water. (149,707.) Sept. 1.

12,494 (1919). Morel. Process for the manufacture of alumina. (149,769.) Sept. 1.

12,642 (1919). Freeth and Cocksedge. Manufacture of ammonium nitrate. (149,095.) Aug. 18.

12,698 (1919). Hood. Preparation of aluminium nitrate. (149,783.) Sept. 1.

12,699 (1919). Hood. Conversion of powdered aluminous materials into granular form. (149,453.) Aug. 25.

25,688 (1919). Hedstrom. Production of compounds of radium. (149,552.) Aug. 25.

VIII.—GLASS; CERAMICS.

APPLICATIONS

Atkinson, and Stein and Atkinson. Drying china clay etc. 24,250. Aug. 20.

COMPLETE SPECIFICATIONS ACCEPTED.

17,254 (1918). Vergniaud. Apparatus for drying crucibles. (149,703.) Sept. 1.

12,025 (1919). Smith. Treatment of clays, and manufacture of articles and materials therefrom. (149,440.) Aug. 25.

30,586 (1919). Sato. Manufacture of decorative glass, porcelain, and the like. (149,567.) Aug. 25.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Berry. Manufacture of artificial stone, cement, etc. 24,469. Aug. 23.

Boyerolle. Manufacture of cement from slag. 23,277. Aug. 9. (Belgium, 7.3.19.)

Commin. 23,296. *See* III.

Imperial Trust for the Encouragement of Scientific and Industrial Research, and Schryver. Manufacture of plastic material for flooring etc. 23,640. Aug. 12.

Vaughan. Cement. 24,649. Aug. 25.

COMPLETE SPECIFICATIONS ACCEPTED.

2341 (1914). Leger. Composition for use in the construction of walls, linings, panels, roof coverings, coach bodies, or the like. Aug. 18.

12,438 (1919). Fasting. Rotary kilns for burning cement and the like. (149,764.) Sept. 1.

16,136 (1919). Steiger. Manufacture of tiles, flags, sheetings, or the like of cement and fibre. (149,120.) Aug. 18.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.**APPLICATIONS.**

- Ball. Production of powdered metals. 23,738. Aug. 13.
 Barrelle. 24,270. *See* II.
 Bengough. Coating metals with lead. 23,721. Aug. 13.
 Boveroulle. 23,277. *See* IX.
 British Thomson-Houston Co. (General Electric Co.). Arc-welding compositions. 24,261. Aug. 20.
 Brunskill. Treatment for prevention of surface corrosion in aluminium alloys. 24,035. Aug. 18.
 Chambers and Fisher. Treating iron and iron alloys. 23,745. Aug. 13.
 Chesterfield and Harden. Applying lead linings to steel etc. pipes. 24,582. Aug. 21.
 De Lavandeyra. Method of heat-treating alloys and operating heat-treating furnaces. 24,585. Aug. 24. (U.S., 3,320.)
 Gamlen. Process for reducing iron ore. 24,551. Aug. 24.
 Hale. Metal, and method of manufacturing same. 23,494. Aug. 11.
 James. Manufacture of tin, terne, etc. coated plates etc. 24,936. Aug. 28.
 Jones. Treatment of impure zinc etc. 24,886. Aug. 28.
 Krupp A.-G. Grusonwerk. Process of separating slags containing iron from coke etc. residues. 24,343. Aug. 21; and 24,471. Aug. 23. (Germany, 26.8 and 11.10.19.)
 Manchester Furnaces, Ltd., and Russell. Heat-treatment furnaces. 24,670. Aug. 25.
 Marino. 24,659. *See* XI.
 Mond (Metallbank u. Metallurgische Ges.). Process for separation of metals from alloys. 24,047. Aug. 18.
 Perkins. Treatment of complex sulphide ores. 23,487, 24,924. Aug. 16 and 28.
 Stein et Cie. Crucible smelting-furnaces. 23,987. Aug. 17.
 Wills. Alloy steel. 24,524. Aug. 21. (U.S., 9.6.17.)
 Wood and Wood. Cupolas or melting or heating furnaces. 24,102. Aug. 19.

COMPLETE SPECIFICATIONS ACCEPTED.

- 243 (1919). Wilson. Treatment of arsenious ores. (149,366.) Aug. 25.
 4161 (1919). Jensen. Preservation of metals and metallic articles. (149,371.) Aug. 25.
 13,420 (1919). Deziel. Process of heating metal objects. (149,793.) Sept. 1.
 18,048 (1919). Wellman Smith Owen Engineering Corp., and Kemp. Continuous reheating furnaces. (149,509.) Aug. 25.

XI.—ELECTRO-CHEMISTRY.**APPLICATIONS.**

- Barfield and Wild. Electric furnaces. 23,537. Aug. 11.
 Bernard. Process of manufacturing insulators. 24,024. Aug. 18.
 British Thomson-Houston Co. (General Electric Co.). 24,261. *See* X.
 Brown. Electric batteries. 24,320. Aug. 21.
 Haglund. Electrolytic tanks with diaphragm cells. 24,854. Aug. 27. (Norway, 17.9.19.)
 John. Means of cooling electrodes. 24,243. Aug. 20.
 Macfarlane and Spink. Apparatus for production of ozone. 24,529. Aug. 21.
 Marino. Electrolyte for electro-deposition of metals and metallic alloys. 24,659. Aug. 25.
 Mauri. Three-phase electric furnaces. 24,198. Aug. 19.

- Morris. Electrolysis of acetone solutions. 24,210. Aug. 20.
 Oldham (G., J., & O.), and Oldham and Son. Galvanic batteries. 24,314. Aug. 20.
 Seward. Electrolytic apparatus. 24,456. Aug. 23.
 Withers (Renault). Electric furnaces. 24,789. Aug. 26.

COMPLETE SPECIFICATIONS ACCEPTED.

- 20,746 (1918) and 5894 (1919). F.I.A.T. Fabbrica Ital. Automobile Torino. Electrical furnaces. (121,485 and 134,810.) Sept. 1.
 710 (1920). Booth Electric Furnace Co. Electric furnace doors. (137,540.) Aug. 25.
 7417 (1920). August. Electric muffle furnaces. (149,893.) Sept. 1.

XII.—FATS; OILS; WAXES.**APPLICATIONS.**

- Firth. 23,815. *See* XIX.
 Heenan. Treatment of oil. 23,507. Aug. 11.
 Hicks. Soap. 23,464. Aug. 10.
 Norris. Liquid soap. 23,567. Aug. 12.

COMPLETE SPECIFICATIONS ACCEPTED.

- 10,662 (1919). Cox. *See* 1.
 11,871 (1919). Longbottom. Soap. (149,728.) Sept. 1.
 12,287 (1919). Mellersh-Jackson (Twitchell Process Co.). Process for splitting fats and oils in the manufacture of glycerin and fatty acids. (149,748.) Sept. 1.
 12,598 (1919). Nordiske Fabriker De-No-Fa Aktieselskap. Process for polymerisation of unsaturated fatty acids at low temperature. (127,814.) Sept. 1.
 15,076 and 30,653 (1919). Marlborough. Cleaning-composition. (149,111.) Aug. 18.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.**APPLICATIONS.**

- Barrett Co. Production of resins from naphthas. 23,454. Aug. 10. (U.S., 28.8.19.)
 Carpmal (Bayer and Co.). 23,551. *See* IV.
 Fischer. Production of resinous bodies from phenols and oxygen. 23,422. Aug. 10. (Germany, 24.5.19.)
 Imperial Trust for Encouragement of Scientific and Industrial Research, and Schryver. Manufacture of coating-compositions. 23,641. Aug. 12.
 Iverson and Roberts. Pigmental enamel or varnish. 23,623. Aug. 12.
 Lilienfeld. Manufacture of plastic masses. 23,867. Aug. 16. (Austria, May, 1920.)

COMPLETE SPECIFICATION ACCEPTED.

- 18,959 (1918). Iverson and Roberts. Composition impervious to oils, spirits, water, and the like. (149,365.) Aug. 25.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.**APPLICATION.**

- Paterson and Smith. Manufacture of caoutchouc etc. froth. 24,418. Aug. 23.

XV.—LEATHER; BONE; HORN; GLUE.**APPLICATIONS.**

- Elektro-Osmose A.-G. Tanning etc. 23,903. Aug. 16. (Germany, 13.10.19.)
 Long. Process for treating vegetable ivory. 23,697. Aug. 13.
 Manley. Adhesive paste. 24,591. Aug. 25.
 Ross and Walker and Sons. Treatment of skins. 24,505. Aug. 24.

XVI.—SOILS; FERTILISERS.

APPLICATION.

Molassine Co., and De Whalley. Artificial manure or fertiliser. 24,933. Aug. 28.

COMPLETE SPECIFICATION ACCEPTED.

3990 (1920). Norsk Hydro-Elektrisk Kvaestof-aktieselskab. Nitrogenous fertilisers. (139,462.) Aug. 25.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATION.

Denington and Perry. 24,746. *See* XVIII.

COMPLETE SPECIFICATION ACCEPTED.

5512 (1919). Lenders. Method of manufacturing starch and conversion products thereof, and the products obtained therefrom. (149,374.) Aug. 25.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Bargate and Walkey. Preparation of alcohol. 23,849. Aug. 16.

Denington and Perry. Process of converting cellulosic and ligneous material into sugars. 24,746. Aug. 26.

Warwick. Apparatus for fermentation of beer etc. 23,828. Aug. 16.

COMPLETE SPECIFICATIONS ACCEPTED.

2786 (1916). Weizmann. Fermentation processes for the production of acetone and butyl alcohols. (149,355.) Aug. 25.

5512 (1919). Lenders. *See* XVII.

12,020 (1919). Dupire. Process for the manufacture of yeast from fermented musts, and more particularly the fermented musts of molasses and sugar-beet pulp. (149,438.) Aug. 25.

21,915 (1919). Anderschou. *See* XIX.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Barton. Cleansing and/or disinfecting baths etc. 23,804. Aug. 14.

Challis. Preserving eggs. 24,117. Aug. 19.

Elektro-Osmose A.-G. Method of killing micro-organisms. 24,191. Aug. 19. (Germany, 19.8.19.)

Firth. Treatment of soya beans for recovery of oil and production of synthetic milk. 23,815. Aug. 14.

Humphrey. Production of fermentable flours and bread-making mixtures. 24,856. Aug. 27.

Lyman. Manufacture of food for animals, poultry, etc. 23,486. Aug. 11.

Magrath. Water-softening apparatus. 24,662. Aug. 25.

Marshall and Sutcliffe. Method of boiling meat for production of food for animals. 23,663. Aug. 13.

Spear. Preservation of food etc. 24,370. Aug. 21.

COMPLETE SPECIFICATIONS ACCEPTED.

16,477 (1918) and 8961 (1919). Higginbottom. Apparatus for separating and purifying wheat and other cereals and their products. (149,020.) Aug. 18.

3091 (1919). Turner. Scouring of grain. (149,368.) Aug. 25.

9854 (1919). Kershaw. Treatment of refuse in refuse-destructors and the production and treatment of fluid slag obtained therefrom. (149,033.) Aug. 18.

16,592 (1919). Rogers and Frémelin. Milk-sterilising apparatus. (149,500.) Aug. 25.

21,915 (1919). Anderschou. Process of treating waste brewers' yeast to render it suitable for baking purposes. (149,533.) Aug. 25.

22,844 (1919). Hieber. Process of treating fruit juices. (149,537.) Aug. 25.

30,772 (1919). McColl, McColl, and Dryden. Installation of boiling pans for the manufacture of confectionery. (149,172.) Aug. 18.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Cockerton, and Gentosan, Ltd. Manufacture of compressed tablets from aspirin etc. 23,513. Aug. 11.

Elektro-Osmose A.-G. Process for making specific vaccines. 24,192. Aug. 19. (Germany, 19.8.19.)

Elektro-Osmose A.-G. Manufacture of specific vaccines. 24,302. 24,351. Aug. 20 and 21. (Germany, 23.8.19.)

Farbw. vorm. Meister, Lucius, und Brüning. Manufacture of methane. 23,446. Aug. 10. (Germany, 15.4.20.)

Fischer. 23,354. *See* II.

Morris. 24,210. *See* XI.

COMPLETE SPECIFICATIONS ACCEPTED.

15,775 (1919). U.S. Industrial Alcohol Co. Continuous process for the manufacture of esters. (130,968.) Aug. 18.

20,223 (1919). Chem. Fabr. vorm. Sandoz. Process for isolation of alkaloids. (134,197.) Aug. 25.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Aves and Duke. Moving-picture films. 24,931. Aug. 28.

Batty and Whyte. Photographic processes. 24,760. Aug. 26.

Guimoin-Sanson. Kinematograph films. 24,459. Aug. 23. (France, 9.6.20.)

Meissner. Process of renovating kinematograph films. 23,631. Aug. 12. (Germany, 5.2.20.)

Schwartz. Photographic printing. 24,208. Aug. 20.

COMPLETE SPECIFICATION ACCEPTED.

11,036 (1915). Sosna and Biedebach. Photographic developing processes. Aug. 18.

XXII.—EXPLOSIVES; MATCHES.

APPLICATIONS.

Explosives Trades, Ltd. (Wardenburg). Process for treating gun-cotton etc. 24,313. Aug. 20.

Silberrad. Propellants. 23,564. Aug. 11. (France, 21.8.19.)

XXIII.—ANALYSIS.

APPLICATIONS.

Atherton. Carbon dioxide recorders. 24,495. Aug. 24.

Frink. Determining viscosity of highly-viscous material. 24,264. Aug. 20.

COMPLETE SPECIFICATIONS ACCEPTED.

12,317 and 26,640 (1919). Brown and Pickston. Apparatus for automatic and continuous analysis and recording of gases. (149,752.) Sept. 1.

I.—GENERAL; PLANT; MACHINERY.**PATENTS.**

Evaporating, condensing, and cooling apparatus; Tubular —. E. Barbet et Fils et Cie. E.P. 117,070, 28.5.18. Conv., 21.6.17. Addn. to 114,838 (J., 1919, 750 A).

THE tubes are so spaced that the sheaths may be dispensed with, the desired velocity of the vapour circulating through the space between the tubes being obtained by using a mechanical blower or a fan.—W. H. C.

Cooling and liquefaction of gaseous fluids; Process and apparatus for —. O. Imray. From E. A. W. Jefferies and F. E. Norton. E.P. 148,588, 30.1.19.

By taking advantage of the latent heat of vaporisation, the heat transference is carried out with only small differences of temperature in the exchangers, thus approaching nearer to the principle of reversibility and so effecting considerable economy and enabling the preliminary compression of the working fluid to be dispensed with. (Reference is directed under Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 106,675; J., 1917, 858.) (See also Cottrell, J., 1919, 126 T.) —W. H. C.

Furnaces; Muffle and semi-muffle and like —. C. F. Priest. E.P. 148,630, 26.4.19.

THE combustion chamber is placed adjacent to but not beneath the furnace chamber, and the combustion gases pass first through flues in the side walls of the furnace chamber, then through flues in the front wall, then beneath the floor and up flues in the back wall, and lastly through flues in the roof to the stack. In the case of the semi-muffle furnace the furnace gases pass in part directly over the material under treatment.—W. H. C.

Grinding or disintegrating ores or other materials; Method of —. H. W. Hardinge. E.P. 148,637, 29.4.19.

THE material is fed into a rotating, conical tube-mill, provided with a removable lining, perforated with holes which register with one or several perforations in the shell at that part of the mill where the greatest peripheral force and grinding effect exist. The grinding is effected by balls of various sizes, which arrange themselves in layers, and the ground material is discharged through the perforations as soon as it is fine enough to pass. —W. H. C.

Drying solid liquid-containing bodies; Arrangement for —. J. W. A. Elling, Assr. to A/S. Myrens Verkstad. U.S.P. 1,347,501, 27.7.20. Appl., 21.2.19.

THE material is placed between flexible bags which extend into a drying chamber and communicate with another chamber supplied with the heating medium. The drying chamber is maintained at a lower pressure than the heating medium chamber. —W. H. C.

Dryer; Tunnel —. Internationale Ges. für Trockenanlagen m.b.H. G.P. 322,019, 6.6.18.

THE cars in which the material is carried through the dryer are divided into compartments by transverse partitions. Separate channels are provided in each for the conveyance of air supplementary to the drying air proper, and when the cars are pushed together these form a continuous passage-way through which the supplementary air flows to mix with the drying air. Openings for air are also provided in these channels at the front end of each car, and these lead to the separate compartments of the

car, so that the supply of supplementary air can be accurately controlled.—J. S. G. T.

Filtering; Method of and apparatus for —. N. B. Down, Assr. to Braden Copper Co. U.S.P. 1,318,159, 3.8.20. Appl., 2.1.20.

A SUBSTANTIALLY dry filter-cake is obtained by the use of a curved filtering surface. When the cake is of sufficient thickness the unfiltered liquid is withdrawn and a gaseous medium is passed through the cake. The curvature of the surface is such that any cracks that may form as the material dries do not extend through to the filtering surface. —W. H. C.

Filter; Drum —. H. Hencke. G.P. 318,843, 7.6.16.

THE filter consists of a cylindrical revolving drum mounted on trunnions; the channels leading from the several compartments of the drum to the suction chamber can be closed by means of taps at any desired part of the periphery of the drum, so that the suction can be interrupted, if necessary, while the drum is revolving and solutions of different concentration withdrawn from any section. The suction and pressure chambers are divided into several compartments to admit of the pressure or suction being applied successively to any desired number of compartments of the drum. The material being filtered can also be transferred successively from one compartment to another to be washed and the various solutions collected separately. —A. R. P.

Filter for removing liquids from gases and vapours. E. Christianus. G.P. 319,936, 15.6.19.

THE basket of the filter consists of a system of fine wires woven between spiral steel bands which can be compressed or expanded in order to adjust the capacity of the filter to the amount of gas to be treated.—A. R. P.

Filter-masses; Process and apparatus for washing out loose —. H. Reisert Ges. G.P. 321,815, 25.6.18. Addn. to 311,895 (J., 1919, 961 A).

TWO or more adjacent filter beds can be treated as described (*loc. cit.*) whereby a diminished supply of water to one filter bed is accompanied by an increased air supply to the same and *vice versa*, whilst a diminished water supply and increased air supply to one filter bed is accompanied by an increased water supply and diminished air supply to the other bed and *vice versa*.—J. S. G. T.

Contents of liquids; Apparatus for the recovery of the —. C. P. Grimwood. U.S.P. 1,348,409, 3.8.20. Appl., 15.7.16.

THE liquid is concentrated in a vessel having a conical bottom connected by a downwardly extending leg with a boot which is provided with a conveyor for the removal of the crystals or precipitates that form as the liquid becomes more concentrated and drop by gravity into the boot. A supply of unconcentrated liquid, introduced into the boot, rises into the leg and washes the descending solids. —W. H. C.

Electrical purification of gases; Apparatus for —. Siemens-Schuckertwerke G.m.b.H. G.P. 318,896, 6.12.18. Addn. to 318,432 (J., 1920, 355 A).

THE pit for the collection of the precipitated dust is divided into several compartments with sloping walls and arranged transversely to the direction of the current. An electrode is fixed at the junction of the dividing walls and this arrangement serves to prevent the current from disturbing the dust that has fallen into the pits.—A. R. P.

Electrical purification of gases; Apparatus for —.
Siemens-Schuckertwerke G.m.b.H. G.P. 322,188,
5.12.18.

THE transverse dimensions of the electrical fittings inserted in the gas stream are smaller than those of the gas stream. For this purpose a number of electrodes facing one another are inserted in succession in various parts of the gas stream, the size of the electrodes relative to the cross section of the channel increasing in the direction of the gas stream. This arrangement facilitates the removal of material precipitated upon the electrodes.

—J. S. G. T.

Crystallisation of salts from hot solutions; Apparatus for the —. Deutsche Solvay Werke A.-G.
G.P. 319,751, 17.12.18. Addn. to 296,635.

THE salt is detached from the various ledges and grids in the crystallising vessel by means of hammers, the tails of which are engaged by hooks or eccentric discs on an endless chain or band running through the apparatus. The vibration detaches the crystals, which fall to the bottom, where they can be collected.—A. R. P.

Treating materials; Process for — for purification purposes, either mechanical or chemical. T. Steen. G.P. 319,708, 10.11.17.

THE material together with the liquid with which it is to be treated is made to flow by means of air-lifts through a series of tanks, in which separation of the material takes place, whilst the liquid is returned to the first tank. Further quantities of liquid are introduced at one or several places below the air-lift device, so that a current of liquid is produced in the opposite direction to that of the material, which current passes from tank to tank through overflows and carries with it impurities, which are discharged from the first tank. Different liquids or liquids at different temperatures may be introduced at different parts of the system.

—J. H. J.

Packing material for gas washers and coolers. F. Trappmann. G.P. 321,768, 22.8.19.

THE packing consists of a series of overlapping inclined screens, each composed of a number of strips crossing diagonally. The upper surfaces of the diagonal strips form deflecting surfaces upon which the descending washing liquid is atomised, while its fall is hindered. The liquor collecting upon the diagonal strips runs off at the several points of intersection, and is thus uniformly distributed over the whole of the washing area of the screens.—J. S. G. T.

Separation of the constituents of gaseous mixtures [; Electrical —]. F. Skaupy. G.P. 321,769, 8.4.15.

By passage of direct current through a mixture of two gases, e.g., neon and argon, the one gas, argon, becomes concentrated at the cathode, and the other, neon, at the anode. In the case of a mixture of three gases, e.g., neon, nitrogen, and mercury vapour, one gas is concentrated at the anode, another at the cathode, and the third in the region between the two electrodes.—J. S. G. T.

Tube-mill. H. W. Hardinge. U.S.P. 1,348,642, 3.8.20. Appl., 12.9.16.

SEE E.P. 123,705 of 1918; J., 1919, 242 A.

Evaporating installations; Apparatus for regulating and maintaining a desired level of liquid in —.
E. A. Barbet. U.S.P. 1,349,114, 10.8.20. Appl., 26.3.17.

SEE E.P. 105,543 of 1917; J., 1918, 173 A.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; A fundamental study of Japanese —. C. Iwasaki. Technol. Rep. Tôhoku Imp. Univ., 1920, 1, 101—135.

THE majority of Japanese coals are anthracites, semi-anthracites, and semi-bituminous. High-grade bituminous coal is scarce, but a low grade is abundant, its composition averaging 40% of volatile matter, 48% of fixed carbon, and 12% of ash. Lignite, with high ash and water content, is also abundant. Thin sections of the coals were prepared and examined microscopically by reflected and transmitted light, and also with the help of X-rays. Two components are distinguished in coal masses, "wood," and "fundamental matter," their relative quantities varying in coals from different localities. The "wood" is abundant in the margin of the coal field but scanty in the central part of it. The "fundamental matter" is that portion of the coal rich in "resin" and ash. It is composed of spherules, resin, cork, pollens, leaves, stems of grasses, etc., all plant remains, cemented by sapropel formed by the total decomposition of plant substances.—W. P.

Coals; Air-dried condition and hygroscopicity of fossil — in relation to the numerical value of their calorific power. R. Kempf. Mitt. K. Materialprüf., 1920, 37, 178—227. Chem. Zentr., 1920, 91, IV., 202.

HYSTERESIS phenomena interfere to a large degree in the attainment of equilibrium between fossil fuels and a moist atmosphere. If fossil coals are exposed to an atmosphere half saturated with water vapour until constant in weight, they will take up varying weights of water according to whether they have been exposed previously to a moist or to a dry atmosphere. This result is attributed to the colloidal nature of the coals. Results of analysis and calorific value should not, therefore, be referred to air-dried coal of unknown moisture content, but to material of known moisture content.—A. G.

Coal; Examination of — with regard to its gasification in producers. H. Strache. Mitt. Inst. Kohlenvergasung, 1919, 1, 35—37.

THE method proposed by the author in 1911 (J., 1911, 1245) and subsequently improved by Hiller (J. Gasbeleucht., 1916, Feb. 26), is eminently suitable for the examination of coals which are to be gasified in producers. The ash content of the "raw coke" is of much more importance than the ash content of the raw coal; e.g., with a coal of composition — moisture 33.1%, ash 17.3%, fixed carbon 14.7%, volatile matter 34.9%, the ash content of the coke would be 54.0%. The water content is of great importance also, since the efficiency of the producer is largely influenced thereby; but the main factor is not the percentage of moisture in the coal, but the ratio moisture: fixed carbon. The Hiller method gives not only the percentage of tar, but the calorific value of the raw coal and of the tar-free coal. The calorific values in the case cited above are as follows, calculated on raw coal: — Coke, 14.7% at 8137 cal., 1196 cal.; tar, 10.3% at 8500 cal., 876; gas (determined directly), 285; total, 2357 cal. The calorific value of the tar is 876 cal., whence the calorific value of the tar-free coal is 1481 cal. A factor is thus determined of 2357:1481—i.e., 1.59. This factor gives a good indication of the bituminous content of the coal. The fusibility of the ash can be determined by the method of Waidner and Burgess (Phys. Rev., 1906, 22, 357).—A. G.

Coals; Calculation of the efficiency of complete gasification from the analysis of —. H. Strache and A. Gross. Mitt. Inst. Kohlenvergasung, 1919, 1, 75—78, 86—89.

THE calculations are based on the carbonisation of coal and the gasification of the resulting coke to either producer-gas (semi-water-gas) or to water-gas, the coal-gas and producer-gas being afterwards mixed, and the calorific value of the mixture determined. Knowing the volume of the gas produced, the total heat units in the gas can be calculated and the efficiency determined from the calorific value and weight of coal used. Losses occur as carbon in the clinker, carbon in the flue gases, radiation and conduction losses, and finally as loss depending on the specific heat of the issuing gas and as undecomposed steam. The authors discuss the distribution of the heat required for drying and carbonising the coal and the heat evolved in the producer portion of the plant. The efficiency of such a system using raw peat as fuel is 88.6%, or 83% on the tar-free fuel.—A. G.

Lignite; Carbonisation of —. Pfeiffer. J. Gasbeleucht., 1920, 63, 420—422.

VERTICAL retorts, operated in the usual manner, are not suitable for the carbonisation of lignite. In the first trials, which were carried out with mixtures of coal and lignite, the coke delagrated violently and was dangerous to the operatives. Not more than 15% of lignite could be safely mixed with the gas coal. Later experiments were tried using lignite alone in an installation of ten vertical retorts. On withdrawing the coke, which was all under 7 mm. mesh, it burst into flame to a height of 20 m. and caused great damage. This latter problem has been solved by quenching in enclosed conduits. The coke can be burnt in household grates when admixed with 30 g. of "Camo cub" (a by-product containing sodium nitrate) per cwt. of coke. From 1 ton of lignite, 250 kg. of coke and 241.5 cb.m. of gas were produced. The cost of the gas worked out at 27.4 pf. per cb.m., as compared with 29.68 pf. per cb.m. for ordinary coal gas.—A. G.

Gas; Reduction of the carbon dioxide in — from wood carbonised in horizontal retorts. Henke. J. Gasbeleucht., 1920, 63, 422—423.

THE high percentage of carbon dioxide in the gas produced by carbonising young, freshly cut pine logs was reduced by passing the gas from the bottom and middle tiers of retorts through the top tier, the latter being kept full of glowing wood charcoal, which was replenished when necessary from the bottom retorts. The carbon dioxide content was reduced from 16.4—18.6% to 5.4—9.8%, whilst the carbon monoxide increased from 22—27% to 34.4—41.8%. The liquor produced was sent to waste, as its content of ammonia was very small, and of pyroligneous acid only 0.5%. The viscous tar fetched only the same price as coal tar.—A. G.

Coke; Hygroscopicity of by-product —. W. A. Selvig and B. B. Kaplan. J. Ind. Eng. Chem., 1920, 12, 783-784.

FOUR samples of pulverised by-product coke were dried at 105° C., and exposed for 1 to 24 hrs. to the laboratory air (average humidity 20—27%). From 0.14 to 0.54% of moisture was absorbed in 7 hrs. To prevent analytical error from this source, the moisture in the laboratory sample should be determined and allowed for in calculating the results on a dry basis.—C. A. M.

Heat of combustion of benzene, toluene, aliphatic alcohols, cyclohexanol, and other carbon compounds. T. W. Richards and H. S. Davis. J. Amer. Chem. Soc., 1920, 42, 1559—1617.

THE heats of combustion of the following substances

have been redetermined, and the values expressed in 18°-calories per g.: sucrose, 3943; benzoic acid, 6320; naphthalene, 9614; benzene, 10,014; toluene, 10,155; tertiary butylbenzene, 10,434; cyclohexanol 8882; di-isamyl, 11,339; methyl alcohol, 5326; ethyl alcohol, 7101; propyl alcohol, 8033; *n*-butyl alcohol, 8615; isobutyl alcohol, 8590.—J. F. S.

Acetylene; Combustion of — in motors. F. Haber. Z. Elektrochem., 1920, 26, 325—329.

THE use of acetylene in place of benzene or petrol is an economic possibility if the increased production of carbide is rendered possible by improved output of coal. The utilisation of acetylene is notably better than that of benzene, since the former is completely, the latter very incompletely, burnt in motor engines. The usual engines with a pre-compression of 1.4:5 can be used. It is, however, necessary to provide sparking plugs in which the metallic portions extending into the combustion chamber are as well cooled as possible; insulating material of low heat conductivity should only be used in the more remote parts of the plug. With pure acetylene, use of an excess of air cannot be avoided in practice, and in consequence the efficiency of the machine is not quite so good as with benzene. A mixed fuel in which another combustible substance in addition to acetylene is used, with the object of depressing the inflammability of the latter, appears particularly promising.—H. W.

Gasoline from natural gas. II. Use of charcoal in determining the gasoline content of natural gas. R. P. Anderson and C. E. Hinckley. J. Ind. Eng. Chem., 1920, 12, 735—738.

HIGHLY activated gas-mask charcoal (8- to 14-mesh) is an efficient absorbent of gasoline. The gas is passed through two glass tubes about 0.8 in. diam., each containing about 8 in. of the charcoal. Variations from 20 to 50 cb. ft. per hr. in the speed make little difference in the case of gas containing 0.15 gall. of gasoline per million cb. ft. For the amount of charcoal specified, the vol. of gas used in a test may vary from a fraction of 1 cb. ft. to 30 cb. ft. or more. The vol. of gasoline removed by the first tube should be between 5 and 10 c.c., but the second should not contain more than a small fraction of 1 c.c. Drying the gas or the charcoal increases the yield by 5 to 8%, and variations in the temperature make but little difference. The gasoline may be recovered by covering the charcoal with mineral oil and distilling. Only 60% is recovered by dry heat *in vacuo*. A portable apparatus for field use is described.—C. A. M.

Bitumens; Extraction of — from mineral aggregates. M. R. Walczak and H. I. Rice. J. Ind. Eng. Chem., 1920, 12, 738—740.

BITUMENS, and especially tar bitumens (sp. gr. about 1.2), may be separated from mineral aggregates (sp. gr. about 2.70) by the use of a solution of a practically inert salt having a sp. gr. intermediate between the two substances. Two kg. of the material—e.g., bituminous concrete—is introduced into a vertical copper still, the lid of which is screwed down, and is connected with a flanged glass tube, about 1.5 in. diam., fitted by means of a cork into a reflux condenser. Sufficient sodium carbonate solution (sp. gr. 1.27—1.28) is added to make the surface of the liquid just visible, and the flask heated until the bitumen accumulates as a layer in the upper portion of the tube. The reflux condenser is washed with 10 c.c. of the sodium carbonate solution, and then closed, and the still rapidly heated, so as to expel the liquid bitumen through an overflow tube, in the cork, which has meanwhile been opened. The material is collected in a large test-tube, centrifuged for 15 min., the upper bituminous layer decanted, and its consistency determined. The addition of about 100 g. of

sodium bicarbonate to the sodium carbonate solution will promote the separation of bitumens which adhere to the mineral aggregate.—C. A. M.

PATENTS.

Coal; Coking of — with recovery of by-products. E. Fleischer. G.P. 298,085, 9.12.16.

A COMBUSTIBLE gas, as free as possible from steam or carbon dioxide, is introduced above 900° C. into the bottom of a furnace, which is charged above with coal, whilst at the same time air and steam are blown into the furnace at a higher level in such quantities that the temperature of the gases which are then burned does not exceed 600°–800° C. Good yields of coke and distillation products are thus obtained.—C. A. M.

Coal; Conversion of — into soluble organic compounds. F. Fischer. G.P. 306,471, 20.5.16.

COAL or a mixture of coal and other substances is treated with ozone, avoiding excessive heating, until the coal is largely converted into substances soluble in water. The aqueous solution when evaporated leaves a hygroscopic residue which has a strong odour of caramel, and is soluble in alcohol but nearly insoluble in ether.—C. A. M.

Coal; Decomposition of —. Rütgerswerke A.-G. G.P. 320,056, 10.8.18.

COAL is softened and decomposed when heated with a small amount of coal tar oils of high b. pt., soluble oils and part of the coal being dissolved to form a tar-like fluid. The insoluble portions may be separated by filtration, sedimentation, etc., after diluting the mass with more of the solvent, or with other organic liquids, such as oils containing carbolic acid, or tar oils of b. pt. 250°–360° C. —C. A. M.

Gas; Apparatus for making —. D. E. Campbell. E.P. 148,027, 29.5.19.

A HYDROCARBON oil is projected at high speed against a breaker plate, air being admitted at the same time or alternately with the oil. The mixture of air with the gas thus produced passes on through a chamber in which oil globules are separated. Means are provided for returning this oil to the jet, and the gas is stored in a gasometer which actuates the air- and oil-nozzle valves.—A. G.

Gas producers. J. F. Wells. E.P. 148,057, 22.8.19.

THE producer is divided into upper and lower compartments by a transverse plate, having an aperture furnished with a bell or cone extending into the lower compartment. The upper compartment acts as a fuel hopper, the fuel being fed into the lower chamber by a vertical feed-rod on which are teeth arranged helically. The producer chamber is provided with an inclined grate and water seal.—A. G.

Ammonia; Process for obtaining — from gases obtained by dry distillation. Gebr. Hinselmann. G.P. 320,415, 1.10.12.

THE gas is cooled in a scrubber before being led into an acid bath, and the hot condensed liquor, after separation of tar, is stirred with milk of lime to produce ammonia gas without the use of steam and with the employment of only a small amount of external heat.—L. A. C.

Peat fuel; Process for manufacturing a —. H. P. K. T. Neilsen. E.P. 125,083, 4.4.19. Conv., 30.3.18.

SEE U.S.P. 1,317,936 of 1919; J., 1919, 890 A.

Peat; Treatment of — for fuel and apparatus therefor. K. E. Edgeworth. U.S.P. 1,347,940, 27.7.20. Appl., 12.9.18.

SEE E.P. 118,903 of 1917; J., 1918, 645 A.

Combustion of fuel. W. A. Bone, J. W. Wilson, and C. D. McCourt, Assrs. to Surface Combustion, Inc. U.S.P. 1,349,040, 10.8.20. Appl., 5.11.10. Renewed 29.12.19.

SEE E.P. 29,430 of 1909; J., 1910, 1448.

Gas-producer. E. H. Crush. U.S.P. 1,349,497, 10.8.20. Appl., 29.12.19.

SEE E.P. 141,771 of 1918; J., 1920, 439 A.

Fuel; Liquid —. E. C. R. Marks. From U.S. Industrial Alcohol Co. E.P. 144,052, 7.5.19.

SEE U.S.P. 1,271,114–5 of 1918; J., 1918, 539 A.

Petrol or gasoline; Process for making —. C. R. Burke. U.S.P. 1,344,253, 22.6.20. Appl., 8.12.10. Renewed 18.5.20.

SEE F.P. 432,762 of 1911; J., 1912, 117.

Pulverised-fuel mixture; Controlling proportions of —. W. E. Harvey. U.S.P. 1,348,561, 3.8.20. Appl., 7.3.19.

Viscous oils. G.P. 319,799. See III.

Dyes. E.P. 148,617. See IV.

Hydrogen. E.P. 147,235. See VII.

Extracting copper. G.P. 309,160. See X.

Coal. E.P. 147,254. See XXIII.

II.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Kelp; Potash from —. III. Preliminary examination of kelp distillates. G. C. Spencer. J. Ind. Eng. Chem., 1920, 12, 786–792.

LOW-TEMPERATURE distillation of kelp (243°–305° C.) yielded 1.1% of crude tar. The dried tar (45–65% of the crude tar) when fractionally distilled yielded 34.2% of oil, 10.6% of aqueous distillate, and 55% of pitch (sp. gr. 1.11; m.p. 103° C.). The oil was separated into 8.5% of oil soluble in ammonia and acids, 10.8% of tar acids, and 14.9% of residual oil. The aqueous portion yielded 0.1% of ammonia. High temperature kelp tar (800°–900° C.) yielded similar products and a residual pitch (sp. gr. 1.17; m.p. 130° C.) containing 3.5–5.15% nitrogen, and yielding Prussian blue when fused with iron turnings and caustic alkali. The first distillates contained variable amounts of oils and phenolic substances. The tar acids had sp. gr. 1.024–1.041. When mixed with the residual oil, alkali solution, and rosin they gave a disinfectant $\frac{1}{4}$ times as toxic to a wood-destroying fungus as coal tar creosote. The aqueous distillate (sp. gr. 1.01–1.065) yielded 0.76% ammonia, and left 3.66–7.93% of residue, which, when distilled, gave a main fraction of reddish colour, somewhat less viscous than glycerol, with sp. gr. 1.3431 at 23° C., n_D^{20} = 1.5013, and composition $C_{10}H_{18}O_6$. It appeared to be a hydroxyl compound of the glycol or glycerol series. It might be of value as a glycerin substitute for mechanical purposes.—C. A. M.

Decolorising carbon. Bock. See XVII.

III.—TAR AND TAR PRODUCTS.

Coal tar and pitch; Free carbon in —. R. Hodurek. Mitt. Inst. Kohlenvergasung, 1919, 1, 9–10, 19–21, 28–30.

THE quantity of pitch produced by the distillation of any tar may be represented by the formula $x = 100k/K$, where k is the free carbon content

of the tar and K that of the pitch; in practice the value of x is always too high. Methods for the estimation of free carbon in tar and pitch by extraction with various solvents are all open to the objection that the results vary according to the solvents used, the carbon separated being of a different degree of purity in each case. The carbon separated out by various solvents is always more or less contaminated with bituminous substances, which the solvent is not able to dissolve or which may even be precipitated from the tar. Such bituminous substances (C_{II}) are precipitated from tar by alcohol, methyl alcohol, benzol, petroleum, benzine, toluol, xylol, pyridine, ether, acetone, and acetic acid, but not by aniline, quinoline, phenol, cresols, nitrobenzene, naphthalene, and anthracene oils. The precipitated bituminous substances are soluble in anthracene oil. They are fusible and possess great binding power, and their evaluation is therefore important if the pitch is to be used for briquetting. For determining the true free-carbon content (C_I), $C_I + C_{II}$ is first estimated by treatment with benzol. The C_{II} in the filtered tar is then estimated in a similar manner and the difference gives C_I , the true "free" or "suspended" carbon. In a sample of tar from Zeche Schwerin the content of C_I (true free carbon) determined by the author's method was 1.50%, whereas results ranging from 3.74 to 6.80% were obtained by other known methods. In general, gas tars contain more C_I than C_{II} , whilst coke-oven tars contain more C_{II} than C_I . During the distillation of tar to pitch (amounting to 57% of the tar) the C_{II} increased by 4.22%, whilst the free carbon C_I increased only by 0.37%. On further distillation to yield 48%, 40%, and 30% of pitch respectively, the C_I increased to 6.68%, 16.80%, and 48.70%, whilst the C_{II} increased to 24.90%, 37.40%, and then diminished to 26.80%. With increase in temperature, the bituminous substances which are at first formed during distillation undergo decomposition with the production of more free carbon. Hence the formula for the calculation of the pitch yield should be $x = 100k/K + C$, where C is a constant depending on the process used, the type of apparatus adopted, and the finishing temperature of distillation.—A. G.

o-Chlorodinitrotoluenes. I. G. T. Morgan and H. D. K. Drew. Chem. Soc. Trans., 1920, 117, 784—793.

THE product of the nitration of *o*-chlorotoluene with nitric-sulphuric acid contains four isomerides in the following proportion: 3.5-dinitro-2-chlorotoluene 55—60%, 5.6-dinitrochlorotoluene 18—20%, 4.5-dinitrochlorotoluene 18—20%, and 4.6-dinitrochlorotoluene 1—2%. The 4.5-dinitro-2-chlorotoluene, m.p. 88.5° C., is more readily obtained as the main product of the nitration of 2-chloro-4-nitrotoluene, whilst about 12% of the 4.6-dinitroisomeride, m.p. 49° C., is formed in the same reaction. The further nitration of 2-chloro-6-nitrotoluene gives a 90% yield of the 5.6-dinitro compound. The dinitro-2-chlorotoluenes contain labile acid radicles displaceable by amino groups. In this reaction addition of ammonia to the aromatic compound precedes substitution, and the first phase of the reaction is accompanied by the development of intense colorations.—G. F. M.

Heat of combustion of carbon compounds. Richards and Davis. See IIa.

Extracting bitumens. Walczak and Rice. See IIa.

Oxidation of toluene. Nakao. See VII.

Phenol and naphthols. Luce. See XXIII.

PATENTS.

Benzol light oils; Recovery of crude products from —. F. Tschudy. U.S.P. 1,348,606, 3.8.20. Appl., 5.7.17.

BENZOL light oils are distilled continuously at the highest temperature required to vaporise the low-boiling homologues. The high-boiling constituents condensed from the vapour by partial fractionation are redistilled in a separate vessel, and the undensified low-boiling constituents are further fractionated by dephlegmation in successive stages.

—L. A. C.

Oils; Manufacture of viscous — readily converted into water-soluble products. Tetralin, G.m.b.H. G.P. 319,799, 4.12.17.

Viscous oils which can be readily sulphonated to yield water-soluble products are prepared by treating hydrogenated naphthalenes with alkyl halides or acid chlorides with or without the addition of condensing agents. Tetrahydronaphthalene forms with benzyl chloride and zinc chloride at 120° C. an oil with b.p. 200°—202° C. (20 mm.), and viscosity 3.2° (Engler) at 50° C.; with chlorinated xylol, an oil of viscosity 5°—6° (Engler) at 20° C.; with benzoyl chloride and a little phosphorus pentoxide at 280°—300° C., an oil with b.p. 228°—230°, and with chlorinated tetrahydronaphthalene and a little phosphorus pentoxide, an oil with b.p. (15 mm.) 240°—243° C., viscosity 35.5° (Engler) at 50° C., sp. gr. (15° C.) 1.104, and flash point about 200° C. From decahydronaphthalene, benzyl chloride, and a little phosphorus pentoxide at 170°—190° C., a very viscous, dark green oil is obtained.—L. A. C.

Alkylamino compounds [alkylaminoanthraquinones]; Manufacture of aromatic —. F. W. Atack and W. N. Haworth. E.P. 147,964, 22.1.19.

AMINOANTHRAQUINONES are alkylated by treatment with a dialkyl sulphate in solution in a high-boiling neutral medium, such as nitrobenzene or tetrachloroethane, in presence of a mild alkali such as sodium carbonate. Thus 20 pts. of 1-aminoanthraquinone may be dissolved in 216 pts. of nitrobenzene and 20 pts. of sodium carbonate added, the mixture being heated to boiling under a reflux condenser, whilst 17½ pts. of dimethyl sulphate is added during a period of about 2 hrs. A yield of about 87% of 1-methylaminoanthraquinone may be recovered from the reaction mixture. When 5 pts. of 2-aminoanthraquinone is treated in the same way with 6 pts. of dimethyl sulphate 2-dimethylaminoanthraquinone is the chief product.

—G. F. M.

Carbazole; Purification of —. W. Kirby, Assr. to South Metropolitan Gas Co. U.S.P. 1,348,722, 3.8.20. Appl., 9.12.19.

SEE E.P. 139,441 of 1919; J., 1920, 291 A.

Decomposition of coal. G.P. 320,056. See IIa.

Acid resins. G.P. 320,255. See XIII.

Resin from wood tar. G.P. 320,620. See XIII.

IV.—COLOURING MATTERS AND DYES.

Benzanthrone; Some properties of —. A. G. Perkin. Chem. Soc. Trans., 1920, 117, 696—708.

SOLUTIONS of benzanthrone in glacial acetic acid yield red or scarlet oxonium salts of the type $(C_{14}H_8O)_2HFeCl_4$ with ferric, stannic, and platinum chlorides. They are all readily dissociated on treatment with water. Benzanthrone dissolves in concentrated sulphuric acid with a deep red coloration, but no sulphate could be isolated. Hydroxy-

benzanthrone, however, prepared from 2-hydroxy-anthranol, sulphuric acid, and glycerol, gave scarlet needles of the sulphate when its acetyl compound in acetic acid was treated with concentrated sulphuric acid. Methoxybenzanthrone prepared by the action of methyl sulphate on the above hydroxy compound formed yellow needles, m.p. 198°–199° C., and gave not only oxonium sulphates, ferrichlorides, etc., but also a red hydrochloride and hydrobromide. The *o*-dihydroxybenzanthrone derived from desoxylizarin—the anthranol corresponding with alizarin—was prepared and purified by acetylation, and the subsequent decomposition of the diacetyl derivative, m.p. 199°–201° C. This dihydroxybenzanthrone, for which the name benzalizarin is suggested, forms orange-red plates, m.p. 309°–310° C. (with decomp.), and maroon-coloured oxonium salts with sulphuric and the halogen acids. It dyes mordanted wool shades similar to those given by alizarin, but of a slightly more orange tint. As benzalizarin readily gives a dimethyl derivative, it is concluded that it must be a 7,8-dihydroxybenzanthrone, and the monohydroxybenzanthrone above mentioned will accordingly be the 7-hydroxy compound. On oxidation in acetic acid solution with chromic acid benzanthrone gives anthraquinone-1-carboxylic acid in excellent yield.—G. F. M.

Sulphide dyes; Constitution of yellow —. J. K. Mazumder and E. R. Watson. Chem. Soc. Trans., 1920, 117, 830–834.

THE yellow sulphide dye obtained by the fusion of diformyl-*m*-toluylenediamine with sulphur, having the empirical formula $C_{11}H_{14}N_4S_{6.27}$, yields, on oxidation with alkaline permanganate, a substance of the composition $C_{11}H_{14}O_2N_4S_{3.68}$. On the assumption that this substance is a sulphonic acid, it is deduced that the dye complex formed from 12 mols. of diformyl-*m*-toluylenediamine contains 5 -S.SH groups and 5 -S.S.SH groups, all of which are converted into sulphonic groups on oxidation. The remaining sulphur atoms and half the nitrogen atoms would accordingly be involved in thiazole rings. The action of reducing agents, with the exception of phosphorus and hydriodic acid which resulted in the formation of two bases, gave no definite products.—G. F. M.

PATENTS.

Dyes; Manufacture of —. R. Maclaurin. E.P. 148,617, 24.4.19.

THE ammonia liquor, or the liquor obtained by washing with water the oils obtained in the low-temperature carbonisation of bituminous fuels, either with or without removal of free ammonia, is made sufficiently acid to decompose any sulphides, and is then filtered to remove tarry and precipitated matter. Sufficient ferrous sulphate is added to the liquor, in which any free acid has been neutralised, to throw down a slate-coloured precipitate which dyes wool in an alkaline bath a slate colour, fast to light, which by after-chroming is converted to a brown shade. The liquor remaining after removal of this precipitate is acidified and treated with sodium nitrate and ferrous sulphate, whereby a precipitate is formed which dyes iron mordanted wool a green colour.—L. A. C.

Dye. U.S.P. 1,348,431. See VI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cellulose acetate. H. J. H. Fenton and A. J. Berry. Proc. Camb. Phil. Soc., 1920, 20, 16–22.

VARIOUS substances have been tried as substitutes

for acetone as solvent. The authors find no objection to esters as dope constituents, and as far as weak acids are concerned, tensile strength determinations gave excellent results, fabrics doped with acetic acid as the principal solvent comparing most favourably with others. No general conclusion can be drawn as regards the chemical nature of a liquid and its solvent action on cellulose acetate, but there appears to be some relation between the dielectric constant and solvent action. For the determination of the acetyl group the authors prefer Ost's method of cold alkaline saponification (J., 1912, 713). Cellulose acetate differs considerably from cellulose in its power of adsorbing various dyestuffs.—W. G.

Cellulose phthalate; Preparation and properties of —. H. A. Levey. J. Ind. Eng. Chem., 1920, 12, 743–744.

CELLULOSE phthalate is obtained by heating cellulose (preferably modified by treatment with an acetic acid solution of chlorine) for 24 hrs. at 70° C. with phthalic anhydride and zinc chloride in the smallest possible quantity of diethyl phthalate, and washing and drying the ester *in vacuo* at not more than 70° C. The product is practically insoluble in all common solvents. (Cf. J.C.S., Oct.)—C. A. M.

Thymol from p-cymene. Phillips and Gibbs. See XX.

PATENTS.

Straw and the like; Method of treating — for making paper-pulp. E. E. Werner. U.S.P. 1,347,979, 27.7.20. Appl., 5.5.19.

STRAW or the like is treated with an excess of an alkaline solution, and, before further treatment, a portion of the solution is expressed to reduce the alkalinity of the material to a given degree.

—L. A. C.

Cellulose; Process of dissolving —. Z. Ostenberg, Assr. to International Cellulose Co. U.S.P. 1,348,731, 3.8.20. Appl., 1.11.16. (See U.S.P. 1,218,954; J., 1917, 450.)

CELLULOSE is dissolved in highly-concentrated hydrochloric acid in which a portion of the hydrogen chloride has been replaced by $\frac{1}{3}$ times its quantity of concentrated sulphuric acid below 50° C.

—L. A. C.

Elastic masses [celluloid substitute]; Preparation of —. Farbenfabr. vorm. F. Bayer und Co. G.P. 309,224, 11.12.17.

COLOURLESS elastic masses suitable for the manufacture of insulating material, billiard balls, artificial glass, optical lenses, etc., are obtained by polymerising methylene ketone, or homologous or analogous compounds. Methylene ketone (b. pt. 96° C.) is thus polymerised when allowed to stand for several weeks at 30° C.—C. A. M.

Cellulose acetates; Process for the manufacture of —. J. O. Zdanowich. U.S.P. 1,347,801, 27.7.20. Appl., 5.5.20.

SEE E.P. 139,232 of 1918; J., 1920, 483 A.

Distillation [of waste lyes from cellulose manufacture]; Process of dry —. E. L. Rinman. U.S.P. 1,347,713, 27.7.20. Appl., 6.11.18.

SEE E.P. 120,724 of 1918; J., 1920, 15 A.

Wood-pulp and cellulose manufacture; Removing water from the pulp sheet in —. Apparatus for extracting water from wood-pulp and the like. R. E. Wagner, Assr. to Aktiebolaget Karlstads Mekaniska Verkstad. U.S.P. 1,347,723–4, 27.7.20. Appl., 19.7.17 and 22.1.18.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Mordant or dye and process of making the same. G. M. Lopez and A. Quaglia. U.S.P. 1,348,431, 3.8.20. Appl., 24.6.19.

THE fruit of a tree of the genus *Guaiacum* is desiccated until volatile juices are removed and the gummy portion is dried, the product is then ground and screened, and the powder is compressed.

—L. A. C.

β-Naphthol preparations; Manufacture of — for the production of a bluish Para Red and other azo colours on the fibre. Chem. Fabr. Grünau Landshoff und Meyer A.-G., and C. Bochter. G.P. 320,178, 25.4.15.

β-NAPHTHOL for the production of azo colours on the fibre is mixed with about 1% of a substantive dye capable of coupling with *p*-nitraniline or other developers, such as Diaminenitrazol Violet, Para Violet, Parazol Violet, or the like. The brown shade of the developed dye is thus avoided, and the liability of the *β*-naphthol to sublime is diminished.

—L. A. C.

Dyeing, washing, scouring, sizing, bleaching, and mercerising yarn in hank form; Machine for —. J. S. and S. S. Lord. U.S.P. 1,348,724, 3.8.20. Appl., 11.4.19.

SEE E.P. 121,075 of 1918; J., 1919, 71 A.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric and nitric acids; Freezing points of mixtures of —. W. C. Holmes. J. Ind. Eng. Chem., 1920, 12, 781—783.

THE freezing point of sulphuric acid is at first lowered by the addition of nitric acid, the minimum (−18.2° C.) being reached with 5.4 HNO₃. On continuing the addition the freezing point rapidly rises to a maximum at +2.3° C. with 10.8 HNO₃. It is probable that a definite compound 5H₂SO₄, HNO₃ is then present. (Cf. J.C.S., Oct.)

—C. A. M.

Hydrochloric acid; Indirect method of determining specific heat of dilute solutions, with preliminary data concerning —. T. W. Richards and A. W. Rowe. J. Amer. Chem. Soc., 1920, 42, 1621—1635.

THE specific heats of dilute solutions of hydrochloric acid are:—HCl.10H₂O, 0.752; HCl.20H₂O, 0.849; HCl.25H₂O, 0.8776; HCl.50H₂O, 0.9320; HCl.100 H₂O, 0.9664; HCl.200H₂O, 0.9812, and HCl.400H₂O, 0.9905. (Cf. J.C.S., Oct.) J. F. S.

Sulphates of sodium; Action of alcohol on the —. G. S. Butler and H. B. Dunncliff. Chem. Soc. Trans., 1920, 117, 649—667.

DRY ethyl alcohol, and also methyl, *n*-butyl, and amyl alcohols, act on dry sodium bisulphate with the formation of an intermediate sulphate, Na₂SO₄.NaHSO₄, and liberation of sulphuric acid which is subsequently partially converted into alkyl hydrogen sulphate. Potassium bisulphate is not decomposed in this way. Moist alcohol completely decomposes sodium bisulphate, giving the intermediate salt and finally the normal sulphate, which is sparingly soluble in moist alcohol. Alcoholic sulphuric acid reacts with normal sodium sulphate in the cold (18° C.) with the formation of an acid sulphate, the amount of the interaction increasing with the strength of the alcoholic sul-

phuric acid employed. Sodium bisulphate is converted by alcoholic sulphuric acid into the intermediate sulphate, Na₂SO₄.NaHSO₄, if the strength is below 20%, whilst if it is 30% or more the salt Na₂SO₄.2NaHSO₄ is probably produced, but if so, this compound is slowly decomposed by extraction with pure dry ether and the intermediate sulphate remains. The intermediate sulphate, Na₂SO₄.NaHSO₄, is also produced by heating dry sodium sulphate with dry sodium bisulphate, and it constitutes nitre cake, either alone, or mixed with the bisulphate or normal sulphate, according as the acidity is equal to, greater than, or less than 18.7% of sulphuric acid.—G. F. M.

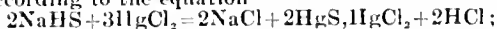
Potassium salts; Separation of — from the coke or ash obtained from cane molasses. G. E. G. von Stietz. Archief Suikerind. Nederl.-Indië, 1920, 28, 519—533. Int. Sugar J., 1920, 22, 467.

IF the coke or ash obtained from cane molasses be leached with a large amount of water (giving a liquor of sp. gr. about 1.07), the salt obtained on evaporation will be composed of:—K₂CO₃, 70—75; K₂SO₄, 15—20; KCl, 3—7; K₂SiO₃, 1—3; and insoluble matter, about 1%. If less water be used (so as to give a liquor of sp. gr. 1.38) the extract is purer, and contains about 90% K₂CO₃, and less than 1% K₂SO₄. Preferably, the material is extracted with an excess of water, and fractionally crystallised. From 100 tons of molasses 2.0% of pure potassium sulphate and about 4.6% of potassium carbonate of 90% purity are obtained.

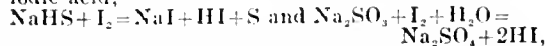
—J. P. O.

Hydrosulphides; Volumetric determination of — in the presence of sulphides, thiosulphates, and sulphites. A. Wöber. Chem.-Zeit., 1920, 44, 601.

SODIUM hydrosulphide reacts with mercuric chloride according to the equation



thiosulphate reacts similarly with the liberation of 2 mols. of sulphuric acid, whilst sulphide and sulphite yield neutral mixtures (cf. Sander, J., 1916, 307). If a solution containing a mixture of the sulphur compounds is treated with an excess of mercuric chloride solution, titration of the resulting acidity gives a measure of the hydrosulphide plus the thiosulphate (a); a similar titration after the hydrosulphide and sulphide have been eliminated by treatment with zinc carbonate gives the acidity due to thiosulphate (b); the difference between a and b is the acidity due to hydrosulphide. Sulphite and hydrosulphide react with iodine with the formation of an equivalent quantity of hydriodic acid,



and the iodometric method described by Sander (*loc. cit.*), with allowance for the hydrosulphide, may be used for the determination of the sulphite, sulphide, and thiosulphate.—W. P. S.

Ferric oxide; Carrying down of copper oxide and nickel oxide by precipitates of —. E. Toporescu. Comptes rend., 1920, 171, 303—305.

WHEN ammonia solution is added to a solution of a ferric salt containing in addition either a copper salt or a nickel salt a certain amount of copper oxide or nickel oxide is always carried down with the ferric hydroxide, the amount depending on the relative proportions of the two salts present and the amount of ammonia added. These oxides cannot be removed from the precipitate by washing or by treatment with strong ammonia.—W. G.

Sulphate of manganese peroxide; Preparation of — and oxidation of toluene by it. M. Nakao. J. Pharm. Soc. Japan, 1920, No. 459, 409—426.

THE best conditions for the preparation of sulphate of manganese peroxide (E.P. 17,981 of 1903; J.,

1904, 749) are as follows:—concentration of sulphuric acid, 55%; temperature, 55° C.; current density, 5–16 amp. per sq. m. The electrolyte in the positive compartment is agitated by a stirrer revolving 800 times per minute. After 20 hrs. the yield of the product begins to decrease. The maximum yield is about 20.8% in the whole liquid, which corresponds to 73.43% of that calculated from the manganous sulphate. Sulphate of manganese peroxide is stable in about 55% sulphuric acid, but unstable in the concentrated acid and above 80° C. By vigorously agitating excess of toluene with a sulphuric acid solution of sulphate of manganese peroxide at 50° C. (E.P. 17,952 of 1903; J., 1904, 910) a 61.45% yield of benzaldehyde was obtained. The manganous sulphate in the waste liquor may be used for the preparation of the peroxide.—K. K.

Phosphates of heavy metals; Determination of phosphoric acid in —. F. Seeligmann. Chem.-Zeit., 1920, 44, 599.

TEN g. of the powdered sample is heated at 90–95° C. for 15 mins. with 12 g. of sodium hydroxide solution (sp. gr. 1.383); the mixture is then diluted with four times its volume of water, boiled, cooled, diluted to 1 l. and filtered. An aliquot portion of the filtrate is acidified with acetic acid and titrated with uranyl acetate solution.—W. P. S.

Stannic chloride; Manufacture of —. S. Ochi. J. Chem. Ind. Japan, 1920, 23, 583–594.

By the action of dry chlorine on metallic tin below 114° C., a yield of 87% of stannic chloride is obtained. A 93% yield is obtained by acting on metallic tin with sulphur monochloride, then removing sulphur monochloride from the crude product by boiling with stannous chloride and distilling. By the action of chlorine upon tin at 100° C. in presence of sulphur monochloride (35% of the weight of tin) as catalyst an 88% yield is obtained. Stannic chloride may also be prepared in about 90% yield by the action of sulphur monochloride upon dry stannous chloride, stannous oxide, stannous sulphide, or stannic sulphide. Tin compounds in the residues may be recovered as sulphide and then as chloride by the action of sulphur monochloride. A mixture of stannic chloride and 34.6% of water forms a hydrate, $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$, m.p. ca. 60° C., which is soluble in water and convenient for use and transportation.—K. K.

Iodic acid and its anhydride; Preparation of —. A. B. Lamb, W. C. Bray, and W. J. Geldard. J. Amer. Chem. Soc., 1920, 42, 1636–1648.

IODINE pentoxide is best prepared, even on a manufacturing scale, by the oxidation of iodine to iodic acid by means of 24–26% chloric acid solution, evaporation of the solution, and dehydration of the iodic acid. A slight excess (3%) of chloric acid is used which destroys the hydrochloric acid formed in the process. The yield is theoretical. The chloric acid solution is prepared directly by the action of sulphuric acid on a solution of barium chlorate. (Cf. J.C.S., Oct.)—J. F. S.

Colloidal catalysts (platinum, palladium); Ageing of —. G. de Rocasolano. Comptes rend., 1920, 171, 301–303.

THE catalytic activity of electrosols of platinum and hydrosols of palladium in regard to the decomposition of hydrogen peroxide increases at first with age until it reaches a maximum and then decreases.

—W. G.

Potash from kelp. Spencer. See IIb.

Zirconia-silica. Washburn and Libman. See VIII.

Copper and ammonia. Yamasaki. See X.

Nitrates and nitrites. Luce. See XXIII.

PATENTS.

Sulphuric acid; Process for extraction of arsenic and other impurities from —. G. K. Davis. E.P. 146,598, 7.4.19.

SULPHURIC acid issuing from a denitration plant, at sp. gr. 1.600–1.750, is cooled and passed into an apparatus (E.P. 2113 of 1909; J., 1910, 79) in which it is rapidly saturated with hydrogen sulphide at a temperature not exceeding 18° C. Without allowing the arsenious sulphide to settle, the acid is then passed through a filter of the vacuum or pressure type. By this process decomposition of the acid by the hydrogen sulphide, with consequent solution of arsenious sulphide by the sulphurous acid, is avoided.—W. J. W.

Basic sulphate of chrome; Production of — and its application to tanning and other uses. J. Morel. E.P. 148,615, 22.4.19.

SOLUTIONS of alkali bichromate or normal chromate, without any addition of sulphuric acid or other reagent, are reduced with sulphurous acid gas to give a basic sulphate of chromium $\text{Cr}_2(\text{SO}_4)_2(\text{OH})_2$, which is used for chrome tanning.—D. W.

Salt; Process for purifying —. F. J. Wood, Assr. to Worcester Salt Co. U.S.P. 1,346,624, 13.7.20. Appl., 30.12.16.

BRINE flowing from the last of a series of vacuum pans is heated by a steam jet to precipitate impurities, filtered, and returned to the first pan and then to the succeeding pans, salt being precipitated in each of the pans.—W. J. W.

Pickle liquor; Treating waste —. A. T. Weaver, E. W. D. Laufer, and E. von Wintzingeroede, Assrs. to The American Steel and Wire Co. U.S.P. 1,348,462, 3.8.20. Appl., 3.8.16.

PICKLE liquor containing sulphates is sprayed into a furnace, and the evolved water, sulphurous and sulphuric acid gases are condensed in the presence of steam, metallic oxides being deposited on the floor of the furnace.—J. W. D.

Lime; Process of treating —. A. E. Horn. U.S.P. 1,348,494, 3.8.20. Appl., 12.6.19. Renewed 28.6.20.

QUICKLIME is treated with a quantity of water sufficient to produce a dry powder, and a fatty acid is added before the hydration is completed.—W. P. S.

Calcium oxide; Manufacture of compact blocks of — from calcium hydroxide sludge. Farbenfabr. vorm. F. Bayer und Co. G.P. 320,422, 27.3.17.

CALCIUM hydroxide sludge, produced, e.g., by slaking quicklime, or decomposing calcium carbide or crude calcium cyanamide, is partially freed from water, compressed into blocks, and calcined.

—L. A. C.

Magnesium carbonate; Preparation of light basic —. "Lipsia," Chem. Fabr. G.P. 302,413, 7.3.16. Addn. to 301,723 (J., 1920, 406 A).

AMMONIUM magnesium carbonate is precipitated from potash end-liquors by the addition of ammonium carbonate, and the precipitate, after washing, is converted into basic magnesium carbonate by boiling with water. A very light *magnesia alba*, free from potassium compounds and chlorides, is thereby produced.—L. A. C.

Nitrogen oxides and similar gases; Condensing —. H. Petersen. G.P. 302,533, 3.1.17.

THE moist gases are led into the top of a reaction tower filled with packing material, and pass out at the lower end. The time of contact of the gases with the liquor condensing on the packing is thereby increased.—L. A. C.

Ammonia; Production of — from ammoniacal liquor and milk of lime. Production of hydrogen sulphide from the waste liquor from ammonia generators. C. Otto und Co., G.m.b.H. G.P. (a) 303,832 (Addn. to 299,621), 23.6.17, and (a) 302,562, 3.7.17.

(A) AMMONIA is liberated from a mixture of ammoniacal liquor and milk of lime by means of steam in a reaction tower provided with ring packing (G.P. 299,621; J., 1920, 448 A). The ammoniacal liquor is led into the tower at a point about two-thirds of its height, and the milk of lime flows in at the top; the upper third of the tower then serves as a scrubber for the ammonia liberated in the lower portion. (n) Hydrogen sulphide is recovered from the waste liquor obtained in the above and like processes by passing the liquor down a similar tower and subjecting it to the action of a counter-current of steam and carbon dioxide.—L. A. C.

Gases containing sulphur compounds; Production of —. Harburger Chem. Werke Schön und Co., and W. Daitz. G.P. 307,081, 1.3.17. Addn. to 306,441

THE addition of magnesium sulphate to the mixture which is calcined to produce a mixture of calcium oxide and sulphide (J., 1920, 570 A) assists the production of calcium oxide, improves the quality of the magnesia sludge, and increases the proportion of sulphur dioxide to hydrogen sulphide in the gases.—L. A. C.

Hydrogen; Manufacture of —. J. Harger. E. P. 147,235, 2.4.18.

PRODUCER-GAS is enriched by passing it at 900°–1200° C. over briquettes of carbon, sodium carbonate, and iron, by which means it is freed from sulphur compounds, tar fog, and hydrocarbons, and its nitrogen content is reduced to 40–50%. The hot gas is passed through a furnace packed with iron round the outside of a vessel containing iron ore, to a heat-interchanger and thence to a purifier from which it is brought back through the heat interchanger into contact with the ore. Part of the spent gases from the reduction phase may be returned to the producer and part may be utilised for heating the briquette chamber. The combustion of part of the spent gases may be utilised for heating a water tube heater to supply steam, and water condensed from the gases in the oxidation stage may be converted into steam in the water heater.—W. J. W.

Sulphuric acid; Production of —. K. B. Quinan. U.S.P. 1,348,736, 3.8.20. Appl., 25.3.19.

SEE E.P. 130,712 of 1918; J., 1919, 717 A.

Nitric acid; Manufacture of concentrated —. P. A. Guye, Assr. to F. Gros et Bouchardy. U.S.P. 1,348,873, 10.8.20. Appl., 9.5.18.

SEE E.P. 131,335 of 1918; J., 1919, 763 A.

Nitric anhydride; Manufacture of —. P. A. Guye, Assr. to F. Gros et Bouchardy. U.S.P. 1,348,874, 10.8.20. Appl., 9.5.18.

SEE E.P. 131,334 of 1918; J., 1919, 763 A.

Magnesium; Separation of — from compounds and mixtures of magnesium and calcium compounds. H. W. C. Annable. U.S.P. 1,347,610, 27.7.20. Appl., 29.6.18.

SEE E.P. 117,483 of 1917; J., 1918, 506 A.

Separation of gases. G.P. 321,769. See I.

Ammonia. G.P. 320,415. See II.

VIII.—GLASS; CERAMICS.

Gas-fired [glass] furnace; New type of —. M. W. Travers. J. Soc. Glass Tech., 1920, 4, 205–225.

THE grate, fitted with Frisbie underfeed mechanism, is fixed below a conical space which tapers upwards to the "eye," the rate of firing being about 130 lb. of fuel per hr. for a furnace taking eight pots. Secondary air, after passing through a recuperator, enters at the sides of the conical space about 2 ft. 6 in. below the "eye," but owing to the gas and air tending to move up coaxially and combustion only taking place at the interface, little combustion takes place below the sieve. Although the furnace was not worked up to the full capacity, the actual output was over 1 ton of glass worked out for 1 ton of fuel consumed, while after nine months' working the furnace was in excellent condition. A rough estimate of the energy balance-sheet of the furnace shows 56 to 60% of the energy supplied to be utilised in the furnace chamber and lost by radiation and conduction from it.—H. S. H.

Optical glass; Casting of pots for use in experimental work on —. A. B. Taylor. J. Soc. Glass Tech., 1920, 4, 140–150.

A SLIP containing 3 pts. of kaolin, 2 pts. of grog (fired to cone 18 and passed through a 30-mesh sieve) and 0.2% (of the total dry weight) of sodium carbonate and sodium silicate together, was used to cast small experimental pots, 9 ins. in height. The pots lacked uniformity when the slip was poured between the core and the mould, or when introduced under pressure of a head of slip, even when an electric current was passed between the core and the mould. Pots showing a uniform distribution of grog were obtained when the slip entered the base of the mould under an initial pressure of 20–25 lb. per sq. in., which was reduced to 10 lb. per sq. in. after filling until the pot was removed, about 45 mins. later. Only small differences in density exist between a slip which is too thick and one which is too thin; too rapid a rise of slip in the mould leads to the inclusion of air bubbles.

—H. S. H.

[Glass] pot making; Art of —. C. O. Grafton. J. Amer. Ceram. Soc., 1920, 3, 653–662.

Soda-magnesia glasses; Density of —. S. English and W. E. S. Turner. J. Soc. Glass Tech., 1920, 4, 153–157.

THE substitution of magnesia for soda diminishes the density of a glass. Winkelmann and Schott's factors of 2.6 for the density of Na₂O, and 3.8 for MgO, would give an opposite result. Tilletson's new factors of 4.1 for CaO and 4.0 for MgO give poor agreement for the glasses examined. Factors of 2.20 for SiO₂, 3.47 for Na₂O, and 3.38 for MgO agree best with the experimental results. The relationship between density and molecular percentage composition is linear for both soda-lime and soda-magnesia glasses.—H. S. H.

Glass tubing; Discoloration produced by lead, antimony, and arsenic in lampworked —. F. W. Hodkin and W. E. S. Turner. J. Soc. Glass Tech., 1920, 4, 158–161.

THE minimum amount of arsenious and antimony oxides required to be present in the glass to cause discoloration lies between 0.02 and 0.05%, while 0.6% of lead oxide is required. The results are independent of the presence of nitre in the batch, while the presence of manganese dioxide, in amount sufficient normally to act as a decoloriser, does not hinder the appearance of discoloration.

—H. S. H.

Glass; Colouring and thermoluminescence of — produced by radium emanation. S. C. Lind. J. Phys. Chem., 1920, 24, 437—443.

ALL three types of radium rays have the same colouring effect on glass and other materials. The colour is discharged from glass and quartz by heating to 500° C. Violet-coloured glass is slowly decolorised by exposure to sunlight, whilst brown-coloured glass is changed to bright yellow by sunlight or ultra-violet light. Violet-coloured glass heated to 200° C. in the dark luminesces, but the colour is unaffected. Freshly-radiated glass luminesces at temperatures below that of boiling water, whilst glass which has been kept for 2—3 years after radiation only luminesces at 200° C.

—J. F. S.

British fireclays; Composition, drying and firing shrinkage, porosity, and density of — suitable for glass furnace refractory materials. E. M. Firth, F. W. Hodkin, and W. E. S. Turner. J. Soc. Glass Tech., 1920, 4, 162—205.

THE chemical compositions, the drying and firing shrinkages, porosity, apparent and actual densities of twenty-seven fireclays at a series of temperatures up to 1400° C. are recorded. It is not possible to correlate the physical properties either with the chemical composition of the clays or with their mineral compositions calculated by Washington's method from their chemical analyses (J., 1919, 105A). Little change of shrinkage occurs below 750° C., while usually a sudden expansion occurs at some temperature below 900° C. The variation of porosity with firing temperature is generally greater for highly aluminous clays than for the silicious clays. Clays with more than 2% of fluxes have frequently a wide porosity range and also a marked shrinkage above 750°. The silicious clays are usually easier to work than the aluminous clays. No relation exists between the amount of water required to produce a suitable working consistency of the ground clay and its chemical composition or the size of its particles. Only five of the clays fulfilled the requirements of the Provisional Specification for Glass Works Refractory Materials, one being Grade 1 and four being Grade 3. It would be better to specify a maximum limit for the iron oxide content and then to classify a clay further by its physical properties instead of by its chemical composition.—H. S. H.

Refractory materials; Tensile strength of —. M. W. Travers. J. Soc. Glass Tech., 1920, 4, 138—140.

THE failures of refractory materials in glass furnaces working at high temperatures are not due to the materials being under load, but are caused by the tensile stresses resulting from the shrinkage of supporting blocks. The ideal refractory for withstanding tensile stresses consists of a system of interlocking crystals, formed *in situ*, in a glassy matrix, rather than one consisting of a high percentage of very infusible material with a small quantity of vitreous bond.—H. S. H.

Zirconia-silica; Approximate determination of the melting point diagram of the system —. E. W. Washburn and E. E. Libman. J. Amer. Ceram. Soc., 1920, 3, 634—640.

THE melting points of mixtures of zirconia and silica were determined by heating one end of a small rod, 2 in. long and $\frac{1}{8}$ in. diam., in an oxy-acetylene flame and measuring the temperature of the droplets of fused material with an optical pyrometer. Temperature readings could be duplicated to about 100° C. The melting point of pure zirconia is about 2700° C.; that of natural zircon and of a mixture of zirconia and silica in molecular

proportions is about 2300° C. An eutectic between ZrO_2 and $ZrSiO_4$ occurs near 2300° C. For use as a refractory up to 2550° C. the presence of silica as an impurity in zirconia is not necessarily detrimental since the fluxing action can be eliminated by increasing the silica content to 33%. It is probable that refractories of considerable strength might be manufactured from zirconia bonded with zirconium hydroxide and precipitated silica in molecular proportions. Iron may be completely removed from zirconia by heating it at 300° C. in a current of phosgene. (Cf. J. Chem. Soc., Oct.)—A. B. S.

Graphite-ash and bond clays; Fusibility of mixtures of —. R. N. Long. J. Amer. Ceram. Soc., 1920, 3, 626—633.

THE ash from Alabama graphite has a higher fusion point than that from Ceylon graphite and is less active as a flux in mixtures of clay and ash. No formation of an eutectic could be detected in mixtures of graphite ash and bond clay. Graphites with a higher percentage of ash than those now in use might be employed with the more refractory clays without serious reduction in refractoriness.

—A. B. S.

Clays; Ultramicroscopic examination of some —. J. Alexander. J. Amer. Ceram. Soc., 1920, 3, 612—625.

FOURTEEN samples of clay were mixed with water and allowed to settle for 9 mins., and the supernatant fluids examined with an ultramicroscope. English china clay, Florida and North Carolina kaolins, Tennessee ball clay, and Illinois Kaolin Co.'s plastic clay were found to contain many actively colloidal particles, those in the English china clay and Laclede-Christy bond clay 69B being very active, those in Florida kaolin not quite so active, and those in the other clays having only a slow motion. Several of the clays contained iron salts and diffusible products of the hydrolysis of the clays; these coagulants probably balance the protective or deflocculant action of organic substances present. The United States is lacking in primary kaolins such as English china clay and no means are known for converting the abundant secondary kaolins into primary ones of the English china clay type. For some purposes Delaware kaolin, if properly treated, could replace English china clay.

—A. B. S.

Terra-cotta body; Effect of the size of grog in a —. H. E. Davis. J. Amer. Ceram. Soc., 1920, 3, 641—652.

TO develop sufficient strength in both dry and burned terra-cotta, a considerable proportion of fine grog should be used, as coarse grog in the absence of finer grog causes cracking. Fine grog is also a desirable ingredient of terra-cotta which is covered with a slip or engobe, as by its use a close-fitting, smooth-surfaced coating of great weather-resisting power may be obtained. The shrinkage and transverse strength are usually, but not always, increased by increasing the fineness of the grog.

—A. B. S.

Glaze study, involving some colours produced by nickel oxide. J. D. Whitmer. J. Amer. Ceram. Soc., 1920, 3, 663—670.

BLuish and greenish greys can be obtained by the use of nickel and magnesium oxides in glazes.

—A. B. S.

PATENTS.

Sheet-glass products; Manufacture of pressed —. F. L. O. Wadsworth. U.S.P. 1,346,571, 13.7.20. Appl., 21.11.13.

MOLTEN glass is spread on a table to form a sheet, one side is reheated to a uniform temperature, and the surface is then pressed.—W. J. W.

Kilns; Operation of chamber —. F. K. Meiser. G.P. 320,733, 20.6.19. Addn. to 316,535 (J., 1920, 366 A).

The supply of hot air is drawn in through the auxiliary air flue by means of the pressure gas supply, which acts as an injector.—L. A. C.

Glass; Apparatus for drawing —. H. K. Hitchcock, Assr. to Hitchcock Experiment Co. U.S.P. 1,319,201, 10.8.20. Appl., 5.8.18.

IX.—BUILDING MATERIALS.

Extracting bitumens. Walczak and Rice. See HA.

Wood-destroying fungus. Schmitz. See XVIII.

PATENT.

Concrete and like plastic materials; Method of curing —. L. A. Bellonby. U.S.P. 1,318,775, 3.8.20. Appl., 26.11.19.

See E.P. 126,813 of 1918; J., 1919, 466 A.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Chromium steels; Some physical constants of —. T. Matushita. Sci. Rep. Tôhoku Imp. Univ., 1920, 9, 243—250.

The investigations were carried out on chromium steels containing 0.6% C and up to 20% Cr in the slowly cooled and quickly cooled conditions. The thermal and electrical conductivities decrease to a minimum at 0.6% Cr, then increase to a maximum at 1.2% Cr, afterwards decreasing rapidly to 5% Cr and then slowly. The values for the slowly cooled specimens are always the greater. The variations can be explained from the structure of chromium steel and its change under heat treatment (Murakami, J., 1919, 257 A). In the annealed steels the moduli of elasticity and rigidity increase at first to a maximum, afterwards decreasing to a minimum at about 5% Cr, then increasing again with the higher concentrations of chromium. In the quickly cooled specimens the elastic constants have lower values. Thermal expansion curves are given, the coefficient decreasing with increasing chromium content. The α - β point first decreases to a minimum at 2% Cr, rising again to 5% Cr and afterwards varying little. The expansion coefficient-concentration curves have a minimum at 2% Cr.—T. H. Bu.

Molybdenum steels. G. W. Sargent. Amer. Soc. for Testing Materials, June, 1920. Chem. and Met. Eng., 1920, 22, 1190—1191.

DETAILS are given of a typical basic open-hearth melt in which about 6000 tons of a steel assaying 0.3% C, 0.64 Mn, 0.77% Cr, 0.35% Mo, and 0.12% Si was produced with an average yield of 93% of that calculated. Annealing the oil-quenched steel at progressively higher temperatures decreases the ultimate strength and elastic limit, which are close together, and increases the reduction in area and elongation. Quenching from 2000° F. (about 1090° C.) does not impair the physical properties or cause the characteristic grain growth of overheated steel. Low molybdenum steel (Mo 0.4, Cr 0.9, and C 0.32%) has a martensitic structure which reverts to fine short pearlitic filaments when the steel is drawn at 900°—1000° F. (480°—540° C.); the ferrite coalesces into rounded masses at 1400° F. (760° C.), at which temperature the steel is well annealed and works easily. A steel containing 0.39% C, 1.05% Cr, 0.17% V, and 0.87% Mo on oil quenching from 1600° F. (870° C.) exhibits a pearlitic structure which remains unchanged unless the steel is drawn

above 1400° F. (760° C.) when it is annealed. Thermal studies show that beyond 800° C. a compound is formed in the austenite which, on cooling, dissociates at the normal A₁ point with precipitation of Fe₃C and at 500° C. breaks up into a Mo-Fe compound which further dissociates and eventually on repeated reheatings gives a solid solution of molybdenum in the iron. Molybdenum up to 4% increases the tensile strength and hardness of cast iron.—A. R. P.

Iron-nickel and iron-cobalt alloys; Change of length by magnetisation in —. K. Honda and K. Kido. Sci. Rep. Tôhoku Imp. Univ., 1920, 9, 221—232.

The change in length of annealed rods of iron-nickel and iron-cobalt alloys in magnetic fields up to a maximum of 600 gauss was measured and its relation with the constituents of the alloys investigated. The 70% Co alloy shows the largest magnetic elongation of ferromagnetic substances yet investigated.—T. H. Bu.

Magnetic susceptibility; Variation of — during allotropic transformations and melting of some substances. T. Ishiwara. Sci. Rep. Tôhoku Imp. Univ., 1920, 9, 233—241.

The magnetic susceptibility of five weakly magnetisable substances (silver bromide, silver chloride, sulphur, zinc, and aluminium) was examined. Generally the susceptibility is constant over the range of temperatures observed, except with zinc, the susceptibility of which decreases one-half in a range of 600° (14°—61° C.). A change of susceptibility is generally found to accompany a change of state, but in the case of silver chloride and bromide such a discontinuity was not observed with the modified torsion balance apparatus used. (Cf. J.C.S., Oct.)—T. H. Bu.

Copper-zinc alloys; Magnetisability of —. R. H. Weber. Ann. Physik, 1920, 62, 666—672.

The magnetic susceptibility of the copper-zinc series was determined and the results plotted against the percentage of copper in the alloy. The compound CuZn₂, which is a greyish-white brittle alloy, has a susceptibility of at least -5.3×10^{-6} , and is, therefore, with the exception of bismuth, the most diamagnetic substance so far known.

—A. R. P.

Copper; Rate of dissolution of metallic — in aqueous ammonia. E. Yamasaki. Sci. Rep. Tôhoku Imp. Univ., 1920, 9, 169—220.

A MORE detailed account of work, the chief results of which have been published previously (J., 1911, 207). (Cf. J.C.S., Oct.)—T. H. Bu.

Brasses; Constitution of certain tin-bearing —. W. Campbell. Amer. Soc. for Testing Materials, June, 1920. Chem. and Met. Eng., 1920, 22, 1191.

INVESTIGATION of the effect of tin on the structure and properties of naval brasses containing 60—65% Cu, 0.5—1.5% Sn, 0—0.5% Fe, 0—1.0% Pb, 0.5% Al, and Zn remainder showed that the amount of tin which will cause the alloy to change from the ductile "naval brass" type to the less desirable "gun-metal" type is inversely proportional to the amount of copper present, decreasing from 2% Sn with 56.5% Cu to 0.5% Sn with 67% Cu.—A. R. P.

Bronzes; Composition of ancient Eastern —. M. Chikashige. Chem. Soc. Trans., 1920, 117, 917—922.

ANCIENT Chinese and Korean bronze mirrors are of four types according to their surface colours, viz., white, pale yellow, deep yellow, and red. The percentages of tin become less and less in the above order and correspond with the chronological period of the mirrors. The white bronzes contain on the

average 67% Cu, 27% Sn, and 6% Pb. The alloy corresponds with the ϵ phase in the Cu—Sn diagram, and the lead lies between the granules of the solid solution of these two metals and acts as a cement. Ancient bronze weapons excavated in China and Japan contain 10–20% Sn, together with 4–8% Sb and sometimes appreciable quantities of iron and nickel, and in some cases even lead up to 8 or 9% was found. Ancient Chinese coins generally contain less tin with much lead, so that some of them can scarcely be termed bronze. For instance, coins current during the eighth to the sixth centuries B.C. gave on analysis the following figures:—Bell money, 67.5% Cu, 16.5% Sn, 10.8% Pb, 1.8% Sb, 2.3% As, and 0.9% Ni, and knife money 46.2% Cu, 9.2% Sn, 43.5% Pb, 0.7% Fe, and 0.3% Ag.—G. F. M.

Corrosion [of bronzes and brasses]; Tests for relative —. B. Feuer. Chem. and Met. Eng., 1920, 22, 1197–1198.

THE action of dilute solutions of salts commonly found in water on brass and bronze was examined by plotting the potential fall between the surface of the metal and the solution in which it is immersed as measured by the aid of an auxiliary calomel electrode. The results obtained with ten different alloys in a $N/10$ solution of potassium nitrate are shown in a graph in which current values are plotted against the logarithms of the potential values. The results check those obtained by means of the "ferroxyl" indicator (Walker and others, J., 1907, 1051).—A. R. P.

Zinc alloys; Pyrophoric — and their utilisation. J. Czochralski and E. Lohrke. Z. Metallk., 1920, 12, 145–149. Chem. Zentr., 1920, 91, IV., 252. (Cf. Guertler, J., 1920, 67 A.)

DIFFERENT refining methods for the removal of aluminium from these alloys (Zn with 3% Al and 6% Cu) are discussed. Melting with sodium nitrate and a flux (sodium chloride and potassium carbonate) seems practicable, the aluminium being reduced to traces. In experiments made with perchlorate instead of nitrate, with addition of potassium carbonate and sodium chloride, even more satisfactory results were obtained. By mixing with zinc oxide and chloride and calcium chloride the aluminium can be removed fairly satisfactorily, but a dense fume of aluminium chloride is formed which tends to stop up the flues.—T. H. Bu.

Bearing metals containing calcium. Hart. Metall. Techn., 1920, 46, 73. Chem.-Zeit., 1920, 44, Rep., 220.

BEARING metal containing calcium instead of tin has a yield point (2000 kg. per sq. cm.) $2\frac{1}{2}$ times as high as that of the tin alloy, a m.p. 120° – 200° C. higher (370° C.), and a Brinell hardness number of 35–45. The calcium forms a compound with the lead which is soluble in molten lead, and which crystallises out on cooling in a similar crystalline form to lead. The solid alloy consists of hard crystals of the calcium-lead compound embedded in a softer ground mass of mixed crystals of lead and other metals. Clean iron crucibles are most satisfactory for melting the alloy, and the charge should be poured at about 450° – 480° C. If it is to be used in layers less than 18 mm. thick the metal to which the coating is to be applied should be first tinned, then heated to such a temperature that on pouring the alloy the tin acts as a solder between the metal base and the alloy.—A. R. P.

Aluminium; Electromotive behaviour of —. I. A. Smits. Proc. K. Akad. Wetensch. Amsterdam, 1920, 22, 876–881.

THE valve action of aluminium in the conversion

of alternating current to direct current is explained as follows. On anodic polarisation a small quantity of oxygen is absorbed; this maintains the surface layer of the aluminium in a state of internal strain in which the metal layer behaves as a non-metal and has a high resistance; a film of oxide may form under certain circumstances and also has a high resistance. On reversing the current the oxygen is removed by the liberated hydrogen and the surface of the metal returns to its internal equilibrium and becomes thereby a good conductor. There is no oxide film on ordinary clean aluminium. (Cf. J.C.S., Oct.)—J. F. S.

Alloys of iron-carbon (steels), copper-tin (tin bronzes), copper-zinc (brasses), and copper-aluminium (aluminium bronzes); Similarity in micrographic appearance existing at different states between —. A. Portevin. Comptes rend., 1920, 171, 350–353.

THE author has obtained photomicrographs for copper-zinc alloys showing first the eutectoid resolved during cooling and, secondly, the martensitic state. Thus the austenitic, martensitic, and troostitic states and the two aspects of the eutectoid have now been observed for steels, brasses, tin bronzes, copper, and aluminium bronzes.—W. G.

Thermoelectric power of pure metals. H. Pélabon. Ann. Phys., 1920, 13, 169–187.

THE thermoelectric power of pure metals varies continuously with the temperature, there being no discontinuity at the m.p. For the metals in the liquid state the thermoelectric power is a linear function of the temperature in the case of lead, tin, and bismuth. With antimony the results are not so regular.—W. G.

Molybdate: its occurrence, detection, and determination in partly oxidised molybdenite ores. J. P. Bonardi. Chem. and Met. Eng., 1920, 23, 205–206.

MOLYBDITE ($\text{Fe}_2\text{O}_3, 3\text{MoO}_3, 7\frac{1}{2}\text{H}_2\text{O}$), which commonly accompanies molybdenite, is not amenable to concentration by flotation; hence serious losses of molybdenum may occur when using this method of concentration with ores containing molybdate. The presence of this mineral may be detected by leaching the ore with a boiling 10% solution of ammonia, sodium carbonate, or hydrochloric acid. The solution is filtered, after having been made ammoniacal if acid was used, the filtrate acidified with hydrochloric acid, and thiocyanate added, followed by zinc. A bright cherry-red colour, which is extracted when shaken with ether, shows the presence of molybdate. To determine the molybdenum present in the oxidised form 5–10 g. of finely ground ore is extracted as above, the solution, if alkaline, acidified with hydrochloric acid, treated with a little hydrogen peroxide, then with an excess of ammonia, boiled, and filtered. The filtrate is acidified with hydrochloric acid, 5–10 g. of ammonium acetate added, and the liquid titrated with a standard solution of lead acetate. A slight excess of the acetate is eventually added and the precipitate filtered off, ignited, and weighed as lead molybdate.—A. R. P.

PATENTS.

Iron or steel; Production of siliconised —. J. Dechesne. G.P. 305,106, 6.10.17.

SILICATES and basic compounds in a more or less finely divided condition, together with carbon, are introduced into the metal by means of a jet of fluid iron. A powerful desulphurising effect is also produced.—T. H. Bu.

Iron articles; Preparation of — for coating by the metal spraying process. Frankonia A.-G. Abt. für Metallisierungsanlagen vorm. Metallatom G.m.b.H. G.P. 320,686, 18.10.18.

THE articles are covered with rust and heated to a temperature of at least 100° C. to remove from the surface occluded or chemically combined gases. By coating with rust mechanically scraping to roughen the surface is rendered unnecessary.—T. H. Bu.

Bronze powder. M. Baer. U.S.P. 1,317,927-8, 27.7.20. Appl., 20.1.19 and 9.2.20.

FLUID metal is cooled in the presence of water to render it porous, then subjected to a continued hammering in the presence of a liquid fat or oil until a fluffy or flocculent powder results.

—J. W. D.

Manganese ores; Treatment of —. G. D. Van Arsdale and C. G. Maier. U.S.P. 1,318,068, 27.7.20. Appl., 9.5.18.

MANGANESE ores containing small quantities of other metalliferous substances, the sulphur salts of which are soluble in water and capable of acting as sintering agents, are suspended in water and subjected to the action of an oxide of sulphur capable of converting the manganese and other metalliferous substances into water-soluble salts.

—J. W. D.

Tungsten ores; Process of reducing —. D. J. Giles. U.S.P. 1,348,356, 3.8.20. Appl., 3.5.19.

GROUND impure tungsten ores are treated with carbon at a temperature below the fusing point to reduce the metallic constituents, then the reduced mass is treated with acid to dissolve the metallic impurities, the insoluble tungsten compound is separated, and the impurities are recovered from the solution.—J. W. D.

Deoxidising (a) copper and its alloys and (b) aluminium and its alloys. A. Strasser. U.S.P. (a) 1,348,457 and (b) 1,348,458, 3.8.20. Appl., 5.2.20.

A COPPER or aluminium cartridge containing a phosphorus compound is added to the copper or aluminium respectively, heated above their melting point, to convert the impurities into phosphorus compounds, and when the metal has reached a higher temperature a second cartridge is added containing an alkaline-earth metal. The phosphorus compounds burn at the expense of the latter, and the oxides, being insoluble, float on the top of the metal.—D. W.

Copper; Process of and furnace for refining —. F. L. Antisell. U.S.P. 1,348,470, 3.8.20. Appl., 6.6.13.

COPPER "forms" are fed continuously into the melting zone of a furnace, where they are melted separately and individually, after having been pre-heated and roasted during feeding by a current of products of combustion passing from the furnace.

—J. W. D.

Copper; Extraction of — from slags and residues. Ostermann & Flüs. G.P. 309,160, 12.6.17.

THE slag etc. containing copper is added as a flux to the charge of a coke-gas producer from which fluid slag is withdrawn. The last traces of copper compounds are reduced and the copper obtained in fluid form.—T. H. Bu.

Copper-coated iron and other metals; Process for separating copper from —. Hirsch, Kupfer- und Messingwerke A.-G., and L. von Grothaus. G.P. 321,525, 15.11.18.

THE material is packed in layers in calcium sulphide contained in iron boxes and heated at a high tem-

perature. The layer of cuprous sulphide formed is easily detached from the metal.—T. H. Bu.

Smelting-furnace. R. G. Ward, Assr. to Independent Mines Smelting Co. U.S.P. 1,318,525, 3.8.20. Appl., 19.6.18.

THE furnace consists of an upper charging chamber and a lower combustion chamber with a horizontal hearth interposed between. This hearth consists of an annular, transversely-curved, charge-supporting metal surface forming a hearth wall with a narrow inner zone tangential to a line which coincides with the normal angle of repose of the material on the hearth.—J. W. D.

Metal alloy of great hardness for tools, especially drawing dies. Ges. für Wolframindustrie m.b.H. G.P. 320,996, 3.4.18. Addn. to 310,011 (J., 1920, 30 A).

A SMALL proportion of chromium (as ferrochrome) is introduced into the alloy of the principal patent. An alloy for borers contains W, Fe, Cr, Ti, Cu, and Co.—T. H. Bu.

Steel; Manufacture of —. R. A. Hadfield. U.S.P. 1,317,711, 27.7.20. Appl., 10.8.17. Renewed 27.5.20.

SEE E.P. 127,302 of 1917; J., 1919, 503 A.

Furnace for heating metal articles and the like. W. H. Southorne. U.S.P. 1,317,719, 27.7.20. Appl., 27.12.18.

SEE E.P. 119,787 of 1918; J., 1918, 739 A.

Blast-furnace. C. Stein, Assr. to C. M. Stein et Cie. U.S.P. 1,317,796, 27.7.20. Appl., 18.5.17.

SEE E.P. 114,333 of 1917; J., 1918, 272 A.

Furnace for tinning and like operations. A. Harper, Sons, and Bean, Ltd., and T. M. Conroy. E.P. 148,727, 16.3.20.

Furnaces; [Water-cooled inlets of] ore reducing and refining —. H. L. Charles. U.S.P. 1,348,151, 3.8.20. Appl., 6.11.17.

Electroplating apparatus. W. Dietzel. U.S.P. 1,349,549, 10.8.20. Appl., 18.2.19.

Pickle liquor. U.S.P. 1,318,462. See VII.

XI.—ELECTRO-CHEMISTRY.

Sulphate of manganese peroxide. Nakao. See VII.

Electromotive behaviour of aluminium. Smits. See X.

Thermo-electric power. Pélabon. See X.

PATENTS.

[Electric] furnace-regulator system. E. M. Bouton, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,319,361, 10.8.20. Appl., 9.4.19.

Electric furnaces; Control system for —. O. A. Colby, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,319,363, 10.8.20. Appl., 15.2.19.

Electric furnaces; Control system for —. E. A. Hester, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,319,379, 10.8.20. Appl., 30.10.18.

Electrical purification. G.P. 318,896. See I.

Electrical purification of gases. G.P. 322,188. See I.

Separation of gases. G.P. 321,769. See I.

Insulating coverings. G.P. 315,700. See XIII.

XII.—FATS; OILS; WAXES.

Stearine industry; Practical methods used in the —. Knor. *Seifensied.-Zeit.*, 1920, 47, 105, 164, 192, 261. *Chem.-Zeit.*, 1920, 44, Rep., 215.

Oils and fats for use in the stearine industry are stored in well-ventilated buildings with blue glass windows. They are purified by washing with sulphuric acid (sp. gr. 1.16) and deodorised by blowing with compressed air after heating *in vacuo* at 115° C. The fat is thoroughly washed, heated at 105° C. to remove water, and then hydrolysed in an autoclave by means of steam at 11–12 atm., in the presence of 3% of lime, 1–1.5% magnesia, 1% of zinc oxide, or 0.5% of zinc dust, preferably a mixture of 0.5 ZnO and 0.5 Zn. For "black" fats magnesia is the best and zinc dust for fats which are not to be distilled. Fish fats and vegetable tallows require the addition of more of the hydrolytic agents and a higher pressure. After completion of the hydrolysis (8–10 hrs.) the steam valve is opened and the mixture allowed to stand for 2 hrs. for the glycerin water to separate, and the two layers are then expelled from the autoclave. The soap present is decomposed by running the fatty acids into boiling sulphuric acid (sp. gr. 1.16). The glycerin water has normally a sp. gr. up to 1.0325.—C. A. M.

Tristearin; Anomalies in the solidification point of —. B. H. Nicolet. *J. Ind. Eng. Chem.*, 1920, 12, 741–743.

TRISTEARIN apparently exists in two modifications melting respectively at about 71.6°–72.2° C. and 55° C. Notwithstanding the rapidity with which the unstable form is converted into the stable form of higher m. pt. when heated, the solidif. pt. usually given for tristearin is about 56° C., i.e., that of the unstable form. By "seeding" liquid tristearin with the stable modification above 60° C. solidification takes place without the formation of the unstable modification, but the latter always appears to be formed when the "seeding" with the stable modification is effected below 56° C. A necessary condition for the phenomenon of double solidif. pt. is that the solidification of the unstable form shall raise the temperature sufficiently to reach the temperature of transition to the stable form. Analogous results were obtained with completely hydrogenated soya bean oil, but not with hydrogenated linseed oil and cottonseed oil. (Cf. *J.C.S.*, Oct.) —C. A. M.

Sulphonated oils; Determination of sulphate in —. E. J. Kern. *J. Ind. Eng. Chem.*, 1920, 12, 785.

UNCOMBINED sulphate in sulphonated oils may be determined by shaking 20–40 g. of the oil with 100 c.c. of 10% monosodium phosphate solution, filtering the aqueous extract, acidifying 50 c.c. of the filtrate, and precipitating the sulphuric acid with barium chloride. The extraction is as complete as after 3 extractions with brine and ether. The method may also be used for the separation of the total sulphate after decomposing the sulphonated oil by boiling with hydrochloric acid. A preferable method is to evaporate the oil with sodium carbonate, ignite and fuse the residue, boil the solution of the fused mass with sodium peroxide, and precipitate the sulphuric acid from the acidified liquid.—C. A. M.

Polenske and Reichert-Meissl values; Effect of pressure on the —. V. H. Kirkham. *Analyst*, 1920, 45, 293–297.

THE Polenske value of a fat is a function of the pressure, and unless values are corrected to normal pressure they may be untrustworthy. For instance, a Polenske value of 2.68 at 760 mm. falls to 2.06 when the determination is made at a pressure

of 627 mm. For butter fat the relationship between pressure and the Polenske value is shown by the formula, $V = r(P - k)/(p - K)$ where P = pressure at which the Polenske value is V ; p = pressure at which the Polenske value is r , and K = a constant, or pressure at which the Polenske value is zero, in this case 45 mm. Therefore the value corrected to normal pressure is

$$\frac{\text{observed Polenske} \times (760 - 45)}{(\text{barometric pressure} - 45)}.$$

The Reichert-Meissl value is a logarithmic function of the pressure, and the errors introduced by ordinary variations in the atmospheric pressure are quite small.—W. P. S.

PATENTS.

Oil presses; Press-plate for cotton-seed and other —. N. B. Henry. Assr. to The Murray Co. U.S.P. 1,348,004, 27.7.20. Appl., 9.9.19.

Oil-press. M. B. Green. U.S.P. 1,348,870, 10.8.20. Appl., 6.3.20.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Paints; Instrument for measuring the hiding power of —. R. L. Hallett. Amer. Soc. for Testing Materials, June, 1920. *Chem. and Met. Eng.*, 1920, 22, 1192.

THE instrument consists of a long microscope tube with a plane ground glass objective and a small hole in place of the eye-piece. If a card having a contrasting stripe painted on it is placed under the objective and gradually withdrawn vertically, the distance at which the field, by diffusion, appears uniformly illuminated is proportional to the contrast existing on the card. The objective is prepared by polishing back an overground piece of plane glass until a white card with a black stripe becomes merged under view at a distance of about 15 cm. The instrument is calibrated by viewing various greys from pure white to dark black, each shade having previously been analysed for white and black by a Howland colour photometer. A logarithmic curve is then constructed with objective distances as ordinates and per cent. hiding power as abscissæ.—A. R. P.

Shellac, shellac varnish, and lacquer; Rapid examination of —. T. L. Crossley. *J. Ind. Eng. Chem.*, 1920, 12, 778–781.

THREE samples of shellac of different grades contained 28.7–31.8% (av. 30.1%) of constituents soluble in ether, and 70.0–70.2% insoluble, the discrepancy being attributed to oxidation. A sample of stick-lac contained:—Woody matter, fibre, etc., 8.5; gums insol. in ether, 70.7; and gums sol. in ether, 25.0%. Assuming shellac to contain 70% insol. in ether, four samples of shellac rosin varnish, specified to contain 75% shellac, were found to contain from 40 to 77%, and these results were subsequently confirmed. From 5 to 7 g. of varnish is stirred with 50–60 c.c. of ether, the solution decanted through a filter, and the insoluble residue washed with successive portions of ether (20 c.c.) until colourless, dried on the water bath, and finally at 110°–120° C. and weighed. Any deposit forming in the filtrate is separated, washed, dried, and added to the insoluble residue, the clear solution is evaporated and the residue dried and weighed as above. The total gums are obtained by evaporating 5–10 g. of the varnish. In testing shellac gum the sample is ground to pass a 30-mesh sieve, and 1 g. moistened with 5 c.c. of alcohol before the treatment with ether. In the case of varnishes containing linseed oil care must be taken to prevent oxidation of the oil during the evaporation of the alcohol.—C. A. M.

PATENTS.

Titanium; Compound of — and process for the manufacture of same. V. M. Goldschmidt, Assr. to Titan Co. A/S. U.S.P. 1,318,129, 27.7.20. Appl., 12.10.17.

A COMPLEX pigment contains a cryptocrystalline titanium oxide, prepared by heating the oxide until the particles exhibit a crystalline interior structure and a smooth exterior surface, mixed with a pigment-forming compound of another element having a different refractive index.—L. A. C.

Paint; Manufacture of a — containing zinc oxide, linseed oil, and zinc oxychloride. E. Wütherich. G.P. 313,031, 21.5.17.

Zinc oxide (100 pts.) is ground with 70 pts. of linseed oil, and 50 pts. of chemically pure 20% hydrochloric acid is added to the mixture. The paint dries in a few hours giving a hard and durable surface which does not peel.—L. A. C.

Impregnating material composed of oil or varnish for the fibrous insulating covering of electrical conductors; Production of —. F. Bruggemann. G.P. 315,700, 27.10.16.

Drying of the impregnating material due to oxidation of the oil or varnish is prevented by the addition of a reducing agent such as zinc dust at ordinary temperature.—J. S. G. T.

Acid resins from the manufacture of benzene; Preparation of pale, readily-drying oils from —. J. Schümmer. G.P. 320,255, 7.5.18.

Acid resins from the manufacture of benzene are distilled *in vacuo*, the sulphurous acid fumes being recovered and converted into sulphuric acid.—C. A. M.

Resinous material; Production of a — from wood tar. J. D. Riedel A.-G. G.P. 320,620, 11.7.18.

Wood tar is heated and agitated in the presence of a liquid, gaseous, or nascent halogen, with or without the addition of a catalyst, until a cooled sample solidifies to a brittle resinous mass. The product is as brittle as shellac, and dissolves in alcohol, giving a rapidly drying varnish.—L. A. C.

Varnishes and paint media; Preparation of anti-septic and preservative —. Chem. Fabr. Flörsheim H. Noerdlinger. G.P. 320,656, 16.4.16.

Tar from coniferous woods is freed from constituents boiling below 200° C. by distillation, which may be carried out in a current of air or steam under reduced pressure, and the residue is hardened or converted into driers by any suitable method and dissolved in a varnish solvent.—C. A. M.

Linoleum; Manufacture of floor-covering as substitute for —. J. H. P. Ligterink. U.S.P. 1,348,571, 3.8.20. Appl., 23.11.18.

SEE E.P. 133,481 of 1918; J., 1919, 916A.

Bronze powder. U.S.P. 1,347,927-S. See X.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber micro-fungus. J. Scott. Indianrubber J., 1920, 60, 410—412.

VULCANISED rubbers, particularly the purer more elastic grades, when exposed to moist air are liable to the development of growths of the micro-fungus, *Stemphylium microsporoides*; to the naked eye the growth often resembles dust, but the fungus threads penetrate the surface rubber and may cause disintegration; rubber injured in this way wrinkles in a curious manner and may become very weak.—D. F. T.

XV.—LEATHER; BONE; HORN; GLUE.

Chrome tanning; Single bath —. E. Griliches. Z. Elektrochem., 1920, 26, 312—354.

THE experiments were performed by rotating pieces of calf skin in the requisite solution during 24 hrs. at the ordinary temperature. With chromium sulphate solutions of differing acidity, that component which is in excess in the solution is more readily absorbed at the commencement of tanning; in concentrated violet solutions the more basic, in more dilute solutions the more acidic component is absorbed, whilst in green solutions a more acidic substance is absorbed than in the violet solutions. From feebly basic solutions a more acidic portion is initially absorbed, whilst strongly basic solutions yield a basic component. As soon as the skin is saturated with one component it commences the more strongly to absorb the other. Apparently the skin does not in general possess the ability of absorbing both components equally; only in strongly basic solutions are the variations in acidity comparatively slight. If during the first hours the absorbed salt is more acidic than that contained in the original solution, the total salt absorbed is also more acidic than the solution and conversely. When washed, the leather appears to retain the chromium component much more firmly than the acidic portion. Tanning can be effected by either green or violet chromium solutions, but although the rate of absorption of chromium oxide and the amount of oxide absorbed are approximately the same from either solution, a much greater amount of acid is absorbed from the green solution corresponding thus to the higher degree of hydrolysis.—H. W.

PATENTS.

Bating hides; Processes based on the action of enzymes for — and baths, mixtures, and the like for use therein. F. Rampichini & Co. E.P. 124,718, 24.2.19. Conv., 23.3.18.

ANIMAL glands containing one or more enzymes suitable for emulsifying fats or peptonising albuminoid substances are mixed with rock salt, the mixture triturated, dried at about 40° C. and then added to a solution of salicylic acid (0.5—1 per 1000), phenol, benzonaphthol, arsenious anhydride, or other antiseptic substance which does not damage the tissue of the hide, or hinder the action of the enzymes but prevents fermentative or foreign biochemical action. A substance adapted to eliminate the insoluble calcium salts in the hide is also added to the solution, which can be used for bating.—D. W.

Waste liquors of the hide-treating art; Recovery of proteids from —. C. L. Peck, Assr. to The Dorr Co. U.S.P. (a) 1,347,822 and (b) 1,347,823, 27.7.20. Appl., 20.7.18 and 6.1.20.

THE liquors are separated into lime sludge and an effluent, the proteids in which are precipitated by mixing them with the effluent from waste tanning liquors. The proteid sludge is heated with (a) sodium hydroxide or (b) lime sludge, dried and used as a fertiliser.—D. W.

Leather; Preparation of a substitute for sole —. Von Heynitz. G.P. 320,629, 7.11.17.

BARK of birch is saturated with a solution which fills the pores and renders it waterproof without affecting its flexibility. Suitable solutions are glue which has been treated with potassium chromate or gallotannic acid, or viscose or acetylcellulose solution.—C. A. M.

Cascien solution; Stable — and process of making same. H. V. Danham. U.S.P. 1,317,845, 27.7.20. Appl., 25.3.19.

Boric acid is dissolved in hydrofluoric acid solu-

tion, alkali added to form the salt, then casein added, and the whole agitated and heated.—D. W.

Skin-like [algin] compounds; Method of forming shaped articles from precipitated —. C. E. Swett, Assr. to A. D. Little, Inc. U.S.P. 1,318,459, 3.8.20. Appl., 27.3.19.

A porous form is wetted with an aqueous solution which will precipitate an algin compound from an alkaline solution of an alginate, then a solution of alkaline alginate is applied to the wetted form, and subsequently the algin compound is removed from the form.—D. W.

Tanning. C. V. Greenwood. U.S.P. 1,349,150, 10.8.20. Appl., 28.7.15.

SEE E.P. 7635 of 1915; J., 1916, 748.

Hides and skins; Machine for treating —. D. Mercier. U.S.P. 1,347,866, 27.7.20. Appl., 30.12.19.

Basic sulphate of chrome. E.P. 148,615. See VII.

XVI.—SOILS; FERTILISERS.

Reaction of the soil; Examination of the factors which determine the —. I. Determination of the acid or basic properties of a soil. II. Reaction of liquids which are saturated with calcium carbonate. N. Bjerrum and J. K. Gjaldback. Kong. Vet.- og Landbohøjskole Aarskrift, 1919, 48–91. Chem. Zentr., 1920, 91, IV., 125.

The reaction of a soil is represented best by the hydrogen ion concentration, determined in the soil solution or in a soil extract prepared in a definite manner. It is best to determine the amount of acids or bases which must be added to the soil to maintain a definite reaction. A series of such results can be obtained by means of electrometric titration, and can be plotted as a titration curve. From the law of mass action it follows that the hydrogen ion concentration in a liquid saturated with calcium carbonate is expressed by the relation between the calcium ion concentration and the carbon dioxide pressure, thus:

$$C_H = K \cdot \sqrt{C_{Ca} \cdot P_{CO_2}}$$

By electrometric measurements the value of $\log K$ at 18° C. was determined to be -5.2 . Although a soil may contain calcium carbonate, it may still react acid. The more carbon dioxide there is in the soil-air and the more lime there is in the soil solution, the more acid will be the reaction of the soil.—J. H. J.

Chemosynthesis at denitrification with sulphur as a source of energy. M. W. Beijerinck. Proc. K. Akad. Wetensch. Amsterdam, 1920, 22, 890–908.

SOME of the common denitrifying bacteria such as *B. denitrificans* and *B. Stutzeri* may occur in two physiologically different modifications, which are hereditarily constant when their feeding conditions remain constant. The autotrophic form is adapted to an inorganic medium (sulphur or thiosulphate-chalk-nitrate) and shows chemosynthesis; the heterotrophic form requires organic food. They may be compared with the oligotrophic and the polytrophic conditions of the nitrifying organisms. Intermediate forms are known, and autotrophy may be gradually lost, but the heterotrophic form preserves the power of denitrification with organic food. The organisms which produce nitrites from ammonium salts are also related to hereditary modifications with the character of saprophytes, living on organic food and unable to oxidise ammonium salts.—J. F. S.

Plants; Absorption and assimilation of food materials by —. E. Reinau. Z. Elektrochem., 1920, 26, 329–342.

CORRESPONDING with the recent theory of the assimilation of carbon dioxide by plants, a similar hypothesis is proposed for other elementary food materials, and conclusions are drawn therefrom for the complete metabolism of plants. The individual character of each metabolic process and the interlocking of the various processes and their effect on manuring are considered. (Cf. J.C.S., Sept.)—H. W.

Copper spray liquids. Villedieu and Villedieu. See XIXb.

PATENTS.

Fertiliser from city and like waste; Method of making —. C. W. Rice, Assr. to Chemo-Mechanical Water Improvement Co. U.S.P. 1,348,320, 3.8.20. Appl., 30.9.15.

THE method consists in adding "muck," the product of the process, to raw sewage preparatory to its treatment.—W. P. S.

Phosphatic fertiliser and art of manufacturing the same. C. C. James. U.S.P. 1,348,495, 3.8.20. Appl., 28.5.17.

A POWDERED phosphatic fertiliser in which the phosphoric acid is present chiefly as dicalcium phosphate.—W. P. S.

Fungicide, insecticide, and soil steriliser; Method of manufacturing a preparation for application to the soil or plant life for use as a —. A. E. Hawker, and Hawker and Botwood, Ltd. E.P. 146,678, 16.5.19.

LIMESTONE, chalk, or gypsum is dried, powdered, and mixed with about 12% of its weight of liquid tar; other substances, such as salt, calcium carbide, naphthalene, borax, camphor, formaldehyde, or carbolic acid may also be added in quantity up to 20%.—W. P. S.

Fungicides for treating seeds. Chem. Fabr. L. Meyer. G.P. 320,919, 23.4.18.

AD MIXTURE of volatile liquids or siccatives with the usual fungicides increases the activity of the same without injuring the germinating power of the seeds. Suitable mixtures are tar, anthracene oil, pyridine bases, aniline, or pyrrole with benzene hydrocarbons or with alcohol; tar, or a mixture of tar oil, tar, and oil of turpentine with siccatives; oleic or resinic acid salts with benzene or alcohol; formaldehyde, copper sulphate, mercuric chloride or potassium hydroxyquinolate with alcohol.

—L. A. C.

Nitrifier, fertiliser, and insecticide; Product serving as —. G. Truffaut. U.S.P. 1,347,798, 27.7.20. Appl., 7.2.18.

SEE E.P. 120,288 of 1917; J., 1919, 21a.

Proteids from waste liquors. U.S.P. 1,347,822–3. See XV.

XVII.—SUGARS; STARCHES; GUMS.

Sucrose; Double polarisation method for the estimation of —; and the evaluation of the Clerget divisor. R. F. Jackson and C. L. Gillis. Sci. Paper, No. 375. Bureau of Standards, 126–194.

THE rate of decomposition of invert sugar in the presence of 5 c.c. of 38.8% hydrochloric acid at different temperatures is indicated by the following decrease of rotation (in degrees on sugar scale) per min.: 80° C., 0.167; 70° 0.056; 60° 0.0132; and

50°, 0.0034. Under the conditions of the Herzfeld procedure (*cf.* J., 1920, 379A), the maximum *levo*-rotation is reached in a total time of 5½ min., and during the remaining period of heating specified (2 to 4½ min.) decomposition of the levulose occurs to such an extent as to render reproducibility by different operators difficult if not impossible. Constant results are obtainable when hydrolysis is carried out at 60° C. during a total period of 9 min., using the same amount of hydrochloric acid, preferably diluted with an equal volume of water previous to addition. On determining the divisor *d* required for the calculation of the sucrose α in the formula: $100 P = P'/d$, in which *P* and *P'* are the direct and invert polarisations respectively, the value 133.25 for a concentration of 13 g. of inverted sucrose at 20° C. was established. That this value is correct, and that Herzfeld's factor 132.66 is inaccurate was confirmed by inverting with invertase and adding the usual amount of hydrochloric acid before making the solution up to 100 c.c. To avoid error due to the presence of invert sugar, the rotation of which in the strongly acid solution used for inversion is different to that of the approximately neutral solution used for the direct reading, it is proposed in the case of cane products having a high content of reducing sugars to add to the liquid used for the direct reading 2.315 g. of sodium chloride, which produces the same effect upon invert sugar as 10 c.c. of 6.34 *N* hydrochloric acid (*i.e.*, 5 c.c. of 38.8% acid diluted with an equal volume of water). Another source of error is the presence of amino compounds, the rotation of which is strongly influenced by the presence of hydrochloric acid. This is avoided by neutralising the inverted solution with sodium hydroxide or ammonia before completing to volume, preferably with ammonia, as a slight excess is without influence on the levulose. The rotation of sucrose instead of being 100 becomes 100—0.265*m* in presence of sodium chloride, and 100—0.169*m* in that of ammonium chloride; while the effect on the invert polarisation is shown by the values:—32.00—0.540*m* for sodium chloride, and —32.00—0.563*m* for ammonium chloride, in which *m* is the grms. of salt present in 100 c.c., and —32.00 is the rotation at 20° C. produced by the hydrolysis of 13 g. of sucrose by invertase in 100 c.c. without the addition of either hydrochloric acid or salt. In carrying out the method recommended as applicable to all products, a solution of suitable normality is clarified with dry basic lead acetate (Horne's formula, J., 1901, 340), mixed, and filtered; 50 c.c. portions of the filtrate are pipetted into two 100 c.c. flasks. For the direct polarisation, 3.392 g. of ammonium chloride is added, the volume completed at the temperature at which the observations are to be made, and the liquid filtered and polarised. For the invert polarisation, 10 c.c. of hydrochloric acid (sp. gr. 1.1029=6.31 *N*) and 20 c.c. of water are added, and the flask immersed in a water-bath at 60° C. for 9 mins. After cooling rapidly, the volume of ammonia required for exact neutralisation is added from a burette, the temperature is adjusted, the volume completed, the liquid filtered if necessary, and the reading made at a carefully controlled temperature in a double-jacketed tube. In the case of beet products, the inverted solution is neutralised with ammonia as before, but the direct reading is taken in the absence of ammonium chloride; while in that of cane products, 2.315 g. of sodium chloride is added to the solution used for the direct polarisation, but the inverted solution is not neutralised. Tables showing the appropriate divisors for different concentrations of sucrose and different temperatures of observation are given, together with some results of analyses of mixtures showing a very close agreement between the amount of sucrose taken and that found, the average error being about $\pm 0.05\%$.—J. P. O.

Syrup; Cost of manufacture of cane and sorghum table — by the "Norit" process. J. Sauer. La. Planter, 1920, 66, 221—222. Int. Sugar J., 1920, 22, 470—471.

A superior though more costly table syrup is obtained by the following procedure as compared with that generally followed in Louisiana in which the juice coming from the mill is sulphited, heated, and allowed to settle previous to evaporation: The strained juice is mixed with kieselguhr at the rate of about 10 lb. per ton of cane ground, sufficient lime being added only partly to neutralise the natural acidity. After boiling, the juice is filtered, evaporated to sp. gr. 1.25, mixed with the "Norit" decolorising carbon (about 5% on the total solids), heated to 82° C., filtered, and concentrated to about sp. gr. 1.366. The cost of treatment is about 1.57 cents per gallon of finished syrup (kieselguhr being 0.63 cent, "Norit," 0.10, and regeneration, 0.84).—J. P. O.

Decolorising carbon. J. C. Bock. J. Amer. Chem. Soc., 1920, 42, 1564—1569.

"Norit," a vegetable carbon containing 5.13% of ash, when shaken with urine, extracts the whole of the uric acid, a large amount of the urea, creatinine, total nitrogen, and phosphates, and a small amount of the ammonia-nitrogen, chlorides, and dextrose. Bouillon cultures after a few seconds shaking with "norit" show a marked decrease in the bacteria content. The adsorption of a pure substance is always much greater than from a mixture. Lactose is readily adsorbed from diluted milk. In all cases the adsorption is better with washed "norit" (ash 2.18%) than with the commercial preparation.—J. F. S.

Dextrose; Action of hydrogen cyanide on —. Kiliani's reaction. J. Bougault and J. Perrier. J. Pharm. Chim., 1920, 22, 129—138.

THE transformation of hexoses into acids containing one carbon atom more in the molecule, by the action of cyanides, does not take place in acid solutions, a concentration of *N*/100 hydrochloric acid being sufficient to prevent it; it appears to be catalysed by minute quantities of alkalis. The reaction between dextrose and potassium cyanide is bimolecular. In a solution containing 1% of dextrose and 3 or more molecular equivalents of potassium cyanide the sugar is completely transformed in 2 days at 20° C. or in 3 days at 15° C.; and by employing a known quantity of cyanide and determining the excess the reaction might be used for estimating dextrose, galactose, etc. The transformation products from dextrose have $[\alpha]_D^{20} = +1.87^\circ$ so that the change of rotation might be used to determine dextrose provided a sufficient excess of cyanide is used and other reactive sugars are absent. Sucrose is affected by potassium cyanide to only a very slight extent. In a solution containing about 2% of potassium cyanide and about 40% of reducing sugar the cyanide is completely decomposed within 10 hrs.—J. H. L.

Polysaccharides; Hydrolysis of —. E. Hildt. Bull. Soc. Chim., 1920, 27, 690—695.

A STUDY of the hydrolysis of sucrose, raffinose, and inulin by a mixture of 2 mols. of sodium benzenesulphonate and 1 mol. of sulphuric acid. This catalyst does not affect the optical or reducing properties of dextrose or galactose even after 8—10 hrs. at 95°—98° C. The rotatory power but not the reducing power of levulose diminishes under such conditions. The non-levulose sugars are not hydrolysed at the ordinary temperature, whereas in the case of sucrose, raffinose, or inulin the formation of levulose is completed at 13°—15° C. after sufficient time.—W. G.

Crystallisable sugar and free acids in plants. H. Colin. *Comptes rend.*, 1920, 171, 316—318.

THE existence of sucrose in a number of fruits in the presence of free acid at a concentration more than sufficient to cause its inversion is shown to be due to the presence of alkali salts of the organic acids in the juices, which lessen the hydrolysing action of the free acids.—W. G.

Potassium salts from molasses. Von Stietz. *See* VII.

Fermentation tube. Fuller. *See* XVIII.

PATENTS.

Rice starch; Process for the manufacture of —. Reckitt and Sons, Ltd., and C. H. Hardy. E.P. 147,255, 7.5.19.

RICE grains are steeped in water for a few hours and then ground until 98% passes a 130×130-mesh silk sieve, water being added during the grinding so that the resulting paste shall contain 60—62% of water. The paste is further diluted to 18°—25° Tw. (sp. gr. 1.09—1.125) and the starch separated in a centrifuge, or the paste is diluted to 1.5°—2° Tw. (sp. gr. 1.007—1.01) and the separation made by decantation. The non-starchy substances may be collected and used as fodder.—W. P. S.

Sugar; Process of extracting —. L. Naudet. U.S.P. 1,343,737, 15.6.20. Appl., 24.5.16.

See F.P. 466,590 of 1913; J., 1914, 878.

XVIII.—FERMENTATION INDUSTRIES.

Capillary active substances; Titrations with — as indicators. IV. *Buffer systems in wort and beer.* W. Windisch and W. Dietrich. *Woch. Brau.*, 1920, 37, 255—257, 261—264. (Cf. J., 1920, 128 A, 461 A, 524 A, 607 A, 613 A.)

THE surface tension of wort or beer is in general slightly lowered by addition of N/10 alkali or N/10 acid. It is lowered also both by sodium undecylate and by eucupine dihydrochloride. The changes of surface tension in the course of titration with N/10 alkali and N/10 acid, in presence of the indicators mentioned, indicate in wort an equilibrium between organic acids and their salts and primary and secondary phosphates. During fermentation the system becomes more acid; bicarbonates appear and secondary phosphates disappear.—J. H. L.

Yeast-like fungi; Action of ultra-violet light on —. B. Feuer and F. W. Tanner. *J. Ind. Eng. Chem.*, 1920, 12, 740—741.

OLD cultivations of 30 different kinds of yeast-like fungi were distributed in water in Petri dishes and exposed to ultra-violet light from a R.U.V. quartz mercury vapour lamp (operating on 110 volts) at a distance of 25 cm. Twenty-three of the organisms were killed within 1 min., only 2 survived 7 mins., and one (*Torula monosa*) 10 mins.—C. A. M.

Saccharase [invertase]; Preparation of highly active —. III. *Purification by dialysis.* H. von Euler and O. Svanberg. *Z. physiol. Chem.*, 1920, 119, 175—190.

SEVERAL of the authors' invertase F-preparations (cf. J., 1910, 1324) were submitted to fractional dialysis through a collodion membrane. It was found that the carbohydrate content of the enzyme rose as the dialysis went on until a point was reached when it became stationary. The authors conclude that invertase must consist chiefly of carbohydrates. The nitrogen content of the dialysed invertase, on the other hand, diminished, and

reached a limit rather higher than the one obtained by one of the authors in a previous investigation. These experiments show that invertase does not require a co-enzyme for its function.—S. S. Z.

Saccharase [invertase] preparations; Diffusion experiments with highly active —. H. von Euler, A. Hedelius, and O. Svanberg. *Z. physiol. Chem.*, 1920, 110, 190—217.

DIFFUSION experiments with different invertase preparations have shown velocities of diffusion of varying magnitudes, and consequently the molecular weights calculated from these figures also showed significant variations. From the results obtained, which are given in detail, the authors conclude that the substrate and the products of the enzymic action do not diminish the size of the colloidal particles of the invertase. Neither is the velocity of diffusion influenced by the optimum acidity which is required for the action of the enzyme as compared with the reaction of pure water.—S. S. Z.

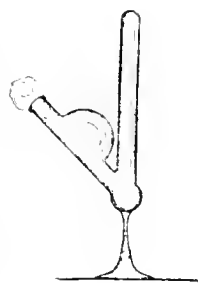
Enzymes of B. coli communis which are concerned in the decomposition of glucose and mannitol. IV. *Fermentation of glucose in the presence of formic acid.* E. C. Grey. *Proc. Roy. Soc.*, 1920, B, 91, 294—305.

THE products of the breaking down of dextrose by *B. coli communis* may be divided into three groups, namely: lactic acid; acetic acid, alcohol, and succinic acid; and formic acid, carbon dioxide, and hydrogen, the second and third groups being closely related. The presence of formic acid as calcium formate does not inhibit the formation of formic acid, and the consequent production of carbon dioxide and hydrogen in the fermentation of dextrose by *B. coli communis*. (Cf. J.C.S., Oct.) —W. G.

Enzyme action in Echinodontium tinctorium, Ellis and Everhart [a wood-destroying fungus]. H. Schmitz. *J. Gen. Physiol.*, 1920, 2, 613—616.

THE presence of esterase, maltase, lactase, sucrose, raffinase, diastase, inulase, cellulase, hemicellulase, urase, reumet, and catalase has been detected in *Echinodontium tinctorium*, Ellis and Everhart, a wood-destroying fungus widely distributed in Western America.—H. W.

Fermentation tube [for sugars etc.]; Modified form of the Smith —. A. V. Fuller. *J. Ind. Eng. Chem.*, 1920, 12, 595.



THE bulb of the fermentation tube, instead of being symmetrical, is trough-shaped on its under side in order to facilitate the falling of the growth to the lowermost portion of the gas tube proper; the gas, as it is liberated, rises vertically and is trapped without appreciable loss in the bulb. A small quantity of coarse, clean sand placed in the depression aids materially in the disengagement of the gas as it is formed.

—W. P. S.

Fusel oil; Composition of a sample of — and origin of acids in alcoholic fermentation. E. Luce. *J. Pharm. Chim.*, 1920, 22, 136—137.

A SAMPLE of fusel oil, after extraction of phenolic constituents and saponification of esters, was fractionally distilled *in vacuo*, and yielded 3% of pelargonic acid and 9.5% of capric acid, besides a residue consisting probably of normal undecylic and lauric acids. These acids were probably formed

by degradation of higher acids derived from the fatty matters of the yeast.—J. H. L.

Denitrification. Beijerinck. See XVI.

Nitrates and nitrites. Luce. See XXIII.

XIXa.—FOODS.

Milk; Micro-organisms persisting in — after pasteurisation: their rôle in the decomposition of hydrogen peroxide. M. Fouassier. *Comptes rend.*, 1920, 171, 327—328.

The organisms, *B. subtilis* and *Tyrophthrix tenuis*, which are frequently found in pasteurised milk, are capable of decomposing hydrogen peroxide in milk, and, if lactic acid-producing organisms are present, abundance of lactic acid is produced. If the latter organisms alone are present they possess very feeble catalysing action on the hydrogen peroxide, but their acid-production is completely inhibited.—W. G.

Gerber's butyrometer; Sulphuric acid unsuitable for use in —. F. Reiss. *Chem.-Zeit.*, 1920, 44, 577.

A stock of sulphuric acid caused trouble when used in Gerber's method for the determination of fat in milk by bursting the butyrometer tubes or blowing out the rubber stoppers. This was due to the presence of a large amount of nitrous acid in the sulphuric acid, oxides of nitrogen being liberated when the acid mixed with the milk in the tube.—W. P. S.

Proteins and methods of determining their composition. A. C. Andersen. *Kong. Vet.-og. Land. Aarskrift*, Copenhagen, 1917, 30—331. *Chem. Zentr.*, 1920, 91, IV., 113.

The Van Slyke method for the analysis of proteins (J., 1911, 1135) is modified, the fifth fraction (amino-nitrogen) being divided into two sub-groups. Glycine, alanine, serine, phenylalanine, tyrosine, valine, and the three isomeric leucines as monoaminomonocarboxylic acids form one sub-group, whilst asparaginic acid and glutamic acid as monoaminodicarboxylic acids form the other. If a mixture of all these amino-acids in solution is neutralised with sodium hydroxide in the way usual for formalin titrations, the monocarboxylic acids, together with proline and oxyproline, remain in the free state whilst the dicarboxylic acids form sodium salts. When the solution is evaporated to dryness and the residue incinerated, the amount of sodium carbonate in the ash is equivalent to the quantity of dicarboxylic acids present.—W. P. S.

Proteins; Relation of the free amino groups to the lysine content in —. K. Felix. *Z. physiol. Chem.*, 1920, 110, 217—229.

The author has estimated the nitrogen titratable with formalin in hydrolysed and non-hydrolysed arachin (from *Arachis hypogaea*), glycinin (from *Sofa hispida*), and gelatin. From the results obtained it is concluded that the fraction of the hydrolysed protein which is precipitated by phosphotungstic acid after the removal of the other diamino-acids contains another nitrogenous substance besides lysine. Van Slyke's assertion that the free amino groups of unhydrolysed proteins contain exactly one half of the nitrogen of the lysine in the protein molecule is not justified.—S. S. Z.

Proteins; Influence of the substrate concentration on the rate of hydrolysis of — by pepsin. J. H. Northrop. *J. Gen. Physiol.*, 1920, 2, 595—611.

The rate of digestion and the conductivity of egg

albumin solutions of different concentrations are approximately proportional at the same pH; this agrees with the hypothesis first expressed by Pauli that the ionised protein is largely or entirely the form which is attacked by the enzyme. (*Cf.* J.C.S., Sept.).—H. W.

Polenske and Retchert-Meissl values. Kirkham. See XII.

PATENTS.

Organic substances; Methods for drying, baking, roasting, and cooking —. G. E. F. Tribes. E.P. 138,101, 21.1.20. Conv., 21.1.19.

DIVIDED organic material, e.g., grains, fruit, coffee, meat, etc., is mixed with a powdered refractory substance so that the surface of the organic material is covered with an air-excluding coating; the mixture is heated to the desired temperature while it is stirred and is then cooled by the further addition of refractory substance, during which addition the stirring is continued.—W. P. S.

Margarine or the like; Manufacture of —. J. R. Sorensen. E.P. 146,567, 18.3.19.

The ingredients are emulsified in an open tank provided with stirrers and a jacket, and are then cooled by circulating cold brine through the jacket.—W. P. S.

Butter substitutes, edible fats, and the like; Manufacture of —. W. Clayton and G. Nodder. E.P. 147,257, 7.4.19.

An emulsion of the required ingredients is passed continuously into one end of a casing containing a hollow rotating worm through which a cooling liquid is circulated. Scraping wheels engage the worm so that the whole of the emulsion is brought in contact with the cooling surface. At the exit end of the casing the material is forced through a fine gauze sieve supported between perforated plates, and then passes through a converging chamber where it is compressed into a compact mass, which is discharged through a nozzle provided with drainage channels for the removal of small amounts of water from the surface of the material. In an alternative apparatus the emulsion is pumped successively through a cooling chamber, a chamber of gradually increasing diameter, a sieve, a converging chamber, and a nozzle fitted with drainage channels. The material may be acted on by beaters or stirrers before and after passing the sieve.—W. P. S.

Bread; Manufacture of leavened —. H. A. Kohman and R. Irvin. U.S.P. 1,345,680, 6.7.20, Appl., 13.12.19.

LAXOLIN is mixed with the dough as a shortening agent.—W. P. S.

Coconut; Process of preparing or treating — and product obtained thereby. F. Baker, jun. U.S.P. (a) 1,318,687, (b) 1,318,688, and (c) 1,348,689, 3.8.20. Appl., (a) 31.10.17, (b, c) 14.10.18.

(A) FINELY-DIVIDED coconut kernel is heated at 212°—225° F. (100°—107° C.) in open receptacles, and the latter, while hot, are transferred to a chamber from which the air is then exhausted. The receptacles are closed while *in vacuo*, heated to a temperature sufficient to sterilise the contents, and then cooled suddenly. (b) The coconut kernel is mixed with a quantity of heated sugar solution, or (c) with heated coconut milk, before it is subjected to the above process.—W. P. S.

Baking powder. F. Rösel. G.P. 322,021, 4.9.18.

The powder consists of a mixture of carbonates with dried and powdered leaven. A fresh leaven, containing about 1.0 acid gives, after air drying at

5°–25° C., a powder containing about 1·8% of effective acid, and also acid-forming enzymes and living yeast cells.—A. G.

Cheese; Process of making Emmenthal or Swiss —. C. F. Doane. E.P. 148,724, 21.1.20.

SEE U.S.P. 1,334,693 of 1920; J., 1920, 346 A.

Edible oil preparations in dry form. H. V. Dunham. E.P. 148,734, 30.1.19.

SEE U.S.P. 1,302,486 of 1919; J., 1919, 511 A.

Food beverage and process of making same. J. L. Kellogg and B. Kazmann, Assrs. to Kellogg Toasted Corn Flake Co. U.S.P. 1,348,999 and 1,349,000, 10.8.20. Appl., 31.3.17. Renewed 27.6.19.

SEE E.P. 120,121 and 120,279 of 1917; J., 1919, 26 A.

XIXB.—WATER PURIFICATION; SANITATION.

Copper spray liquids; Action of rain water on the deposits from —. Villedieu and G. Villedieu. Comptes rend., 1920, 171, 360–363.

ORDINARY country rain water does not dissolve even traces of copper from the precipitates occurring in either Bordeaux or Burgundy mixtures.—W. G.

Potash etc. from kelp. Spencer. See IIB.

Denitrification. Beijerinck. See XVI.

Nitrates and nitrites. Luce. See XXIII.

PATENTS.

Water; Removal of atmospheric oxygen from — by means of iron. L. and C. Steinmüller. G.P. 320,893, 30.5.17.

THE water is passed first through filters with surfaces (plates, tubes, rings, etc.) which are smooth, easily cleansed, and of low activity, and then through filters having very rough surfaces of high activity. In this manner the active surfaces can be maintained clean for a greater length of time.

—J. S. G. T.

Rendering foul waste gases odourless; Process and apparatus for —. H. Neil. G.P. 321,511, 10.12.18.

THE waste gases are led direct to the coal bunkers of the plant, which are provided with the necessary fittings enabling them to be used as deodorising filters.—J. S. G. T.

[*Water*] *purifying material; Manufacture of* —. C. Mas-atsch, Assr. to The Permutit Co. U.S.P. 1,343,927, 22.6.20. Appl., 18.8.14.

SEE E.P. 20,144 of 1914; J., 1915, 886.

Fungicide. E.P. 146,678 and G.P. 320,919. See XVI.

Fertiliser. U.S.P. 1,348,320. See XVI.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Pelletierine and methylpelletierine. G. Tanret. Bull. Soc. Chim., 1920, 27, 612–624.

THE author reaffirms previous results (Comptes rend., 1880, 90, 697) as to the properties of pelletierine and methylpelletierine, found in the bark of the pomegranate tree, and refutes the statements of Hess and Eichel (J., 1918, 559 A) as to the non-existence of optically active forms of these alkaloids. A number of salts and derivatives of the

alkaloids have been prepared. (Cf. J.C.S., 1920, i., 499.)—W. G.

Phenyl derivatives of arsenious acid; Iodometric estimation of —. P. Fleury. Bull. Soc. Chim., 1920, 27, 699–704.

FOR the estimation of each component in a mixture of arsenious, phenylarsine, and diphenylarsine chlorides and triphenylarsine a known amount of the mixture is dissolved in benzene, and the solution is extracted with aqueous sodium hydroxide. The extract, which contains the arsenious acid and phenylarsine oxide, is acidified with hydrochloric acid until the acidity is approximately N/10; an equal volume of alcohol is added, and the mixture is titrated with standard iodine. This titration gives the amount of phenylarsine chloride present in the original mixture. An excess of sodium bicarbonate is added, and the titration continued till the second end-point is reached. The additional iodine required is equivalent to the arsenious chloride originally present. The benzene solution left after the extraction is titrated with standard iodine, the amount required being equivalent to the diphenylarsine chloride and triphenylarsine originally present. To the liquid resulting from the titration excess of aqueous sodium bicarbonate is added, and the liquid is extracted with chloroform. The extract contains the triphenylarsine oxide, and if the aqueous solution is acidified and again extracted with chloroform the second extract will contain the diphenylarsenic acid, which may be obtained in a crystalline form on evaporation of the solvent.

—W. G.

Diphenylarsenious chloride and cyanide. (Diphenylchloroarsine and diphenylcyanoarsine.) G. T. Morgan and D. C. Vining. Chem. Soc. Trans., 1920, 117, 777–783.

TRIPHENYLARSINE was prepared on an experimental technical scale by the interaction of arsenious chloride, chlorobenzene, and sodium in an inert medium, e.g., xylene, using a steel reaction vessel provided with heating and water cooling arrangements and fitted with a stirrer, reflux condenser, a glass reservoir for holding the liquid reagents, and a sodium dropping apparatus. The chief feature of the method is that the sodium is melted under a layer of xylene in this apparatus and allowed to run gradually through a silica sight glass into the other reagents mixed in the reaction vessel, the temperature being maintained at 70° C. The product is isolated without any addition of water by filtering whilst warm from the precipitate of sodium chloride and distilling off the solvent, when triphenylarsine remains as a pale yellow crystalline mass. This was converted into diphenylchloroarsine by heating with arsenious chloride for 3 hrs. in a rotating autoclave and fractionally separating the product by distillation in a current of carbon dioxide at reduced pressure. Diphenylarsenious cyanide was most conveniently obtained by heating diphenylchloroarsine with a 10% excess of silver cyanide in a glass-lined rotating autoclave for 3 hrs. at 150°–160° C. The yield of cyanide of m.p. 28° C. (90% pure) was about 92% of the theory. Other methods in preparing this substance from diphenylarsenious oxide, diphenylcacodyl, and diphenylarsenious sulphide are also described.—G. F. M.

Quinol [; *Reactions of* —]. J. Messner. Pharm. Zentralh., 1920, 61, 454–455.

QUINOL reduces silver nitrate in the cold and changes the colour of Fehling's solution to green, but actual reduction of the copper, with precipitation of cuprous oxide, takes place only after long contact or when the mixture is heated. Statements in literature that Fehling's solution is reduced in the cold and silver nitrate only when heated with

quinol are incorrect. The reactions are of little, if any, use for differentiating between quinol and catechol.—W. P. S.

Catalytic oxidation by unsaturated compounds (oils, hydrocarbons, etc.). J. Bougault and P. Robin. *Comptes rend.*, 1920, 171, 353–355.

DICHLOROETHYL sulphide in solution in turpentine oil when exposed to the air is oxidised to dichloroethyl sulphoxide, $\text{SO}(\text{C}_2\text{H}_4\text{Cl})_2$, m.p. 112°C . The turpentine may be replaced as solvent by various unsaturated oils or hydrocarbons. If pure dichloroethyl sulphide or its solution in a saturated oil is exposed to the air it does not undergo oxidation.

—W. G.

$\beta\beta$ -Dichloroethyl sulphide; Volumetric estimation of —. W. F. Holley. *Chem. Soc. Trans.*, 1920, 117, 898–902.

THE formation of a crystalline double salt with cuprous chloride, of the constitution $[(\text{C}_2\text{H}_4\text{Cl})_2\text{S}]\text{Cu}_2\text{Cl}_2$, is utilised for the volumetric estimation of $\beta\beta$ -dichloroethyl sulphide in mustard gas. Higher chlorinated compounds do not thus react with cuprous chloride. About 1 g. of the sample is weighed into a 100 c.c. stoppered flask, 10 c.c. of a standardised, freshly prepared solution of cuprous chloride in absolute alcoholic hydrogen chloride is added from a burette, the contents of the flask are allowed to remain for 10 mins. in the cold, with occasional agitation, and are then diluted by the addition of 50 c.c. of 5% sodium chloride solution. After mixing well, the contents of the flask, which now measure 59.5 c.c., are filtered through glass wool, and the excess of copper is estimated in an aliquot portion of the filtrate by means of potassium iodide and thiosulphate in the usual way, after oxidising with hydrogen peroxide and boiling three times nearly to dryness to remove the excess of volatile oxidising agents present. The total copper is determined by a blank experiment, and the difference represents the $\beta\beta$ -dichloroethyl sulphide in terms of N/10 thiosulphate, 1 c.c. of which is equivalent to 0.0159 g.—G. F. M.

Tetranitromethane; Effect of reducing agents on — and a rapid method of estimation [and estimation of hydrazine]. A. Baillie, A. K. Macbeth, and N. I. Maxwell. *Chem. Soc. Trans.*, 1920, 117, 880–884.

TETRANITROMETHANE is rapidly converted into nitroform when shaken with aqueous alkaline solutions of reducing agents, such as sulphites, arsenites, sodium potassium tartrate, or hydrazine. If potassium hydroxide is used to keep the solution alkaline, the nitroform separates out as its sparingly soluble potassium salt. The reaction proceeds with sulphites, for example, according to the equation $\text{C}(\text{NO}_2)_4 + \text{K}_2\text{SO}_3 + 2\text{KOH} = (\text{NO})_2\text{CK} + \text{K}_2\text{SO}_4 + \text{KNO}_2 + \text{H}_2\text{O}$. With aqueous solutions of hydrazine the reduction is quantitative, and measurement in a nitrometer of the volume of nitrogen evolved, according to the equation, $\text{NH}_2\text{NH}_2 + 2\text{C}(\text{NO}_2)_4 + 4\text{KOH} = 2(\text{NO})_2\text{CK} + 2\text{KNO}_2 + \text{N}_2 + 4\text{H}_2\text{O}$ affords a convenient method for estimating tetranitromethane, even in the presence of nitric or nitrous acid. Conversely tetranitromethane may be employed for the estimation of hydrazine in solutions of its salts.—G. F. M.

Nitric esters; Decomposition of —. R. C. Farmer. *Chem. Soc. Trans.*, 1920, 117, 896–898.

A DIRECT decomposition of nitric esters under the influence of hydrolytic agents into aldehydes, ketonic acids, and formic and oxalic acids as postulated by Lowry and others (*J.*, 1920, 558A) does not take place, but such oxidation as may occur is always preceded by hydrolysis into nitric acid and the alcohol. When the conditions are

such that the alcohol is protected from oxidation, as, for example, in the case of glycerol by continuous removal as glycerophosphoric acid or by the presence of further nitro-groups, or when the oxidising action of the nitric acid is suppressed by the presence of reducing agents or of sulphuric acid, the alcohol is readily regenerated as such on hydrolysis, thus proving that the first stage in the decomposition is an ester hydrolysis of the normal type. The hydrolysis is, moreover, shown to resemble that of other esters in the relationship between velocity and concentration of hydrogen or hydroxyl ions, and the bearing of this relationship on the stabilisation of nitric esters by means of weak alkalis is shown. The ordinary hydrolytic decomposition passes therefore through the phases: incipient ester hydrolysis; internal oxidation, possibly to an alcohol peroxide; formation of an aldehyde; and decomposition to hydroxy acids. The non-hydrolytic decomposition of nitric esters in the absence of water, by nitric peroxide, is slow, but autocatalytically accelerated, and the action of feeble alkalis in combating acid hydrolysis is shown by the stabilising influence of weak bases such as diphenylamine; stronger bases, on the other hand, such as benzylamine or aniline, cause an even more rapid decomposition (aminolysis).—G. F. M.

Acetaldehyde and acetic acid; Conversion of acetylene into —. B. Neumann and H. Schneider. *Z. angew. Chem.*, 1920, 33, 189–192.

FOR the conversion of acetylene into acetaldehyde on the laboratory scale the best results were obtained by leading the gas with vigorous mechanical stirring into 96% acetic acid containing 3% of mercuric sulphate in solution, the temperature being maintained at about 30°C . Under these conditions nearly 90% of the theoretical yield was obtained. At 40° – 50°C . the rate absorption was actually greater, but the percentage yield of acetaldehyde was reduced to 70–80%. Using a dilute sulphuric acid medium instead of acetic acid the reaction was more erratic, and under favourable circumstances only 70–75% yields were obtained. The best results in the direct conversion of acetylene to acetic acid were obtained by using the above mercuric sulphate-acetic acid catalyst with the addition of vanadium pentoxide, acetylene and oxygen being led in alternately. Yields of acetic acid up to 83% were obtained.—G. F. M.

Sodium formate; Factors in the conversion of — to oxalate. E. H. Leslie and C. D. Carpenter. *Chem. and Met. Eng.*, 1920, 22, 1195–1197.

IN the conversion of sodium formate to oxalate by heating it with caustic soda, the best results are obtained by using approximately 1% of caustic soda and a pressure of 14 in. of mercury at 350°C . The process should be a continuous one in which small quantities of formate only are subjected to these conditions at any one time. A theoretical discussion of the principles underlying the process is given.—A. R. P.

Thymol; Synthesis of — from p-cymene. M. Phillips and H. D. Gibbs. *J. Ind. Eng. Chem.*, 1920, 12, 733–734.

p-CYMESE was isolated from a crude oil derived from a sulphite spruce pulp mill and purified by treatment with sulphuric acid, washing, drying and redistillation. When nitrated it yielded nitrocymene, which was reduced to cymidine by means of iron powder and hydrochloric acid. This was converted into a mixture of *o*- and *p*-cymidinesulphonic acids, and these, in turn, into the corresponding diazo-cymenesulphonic acids, both of which yielded cymene-3-sulphonic acid when treated with ethyl alcohol and copper powder, and by fusing the sodium salt of this acid with sodium hydroxide thymol was formed.—C. A. M.

Volatile oil of Mosla grosseserrata, Maxim. Y. Murayama. J. Pharm. Soc. Japan, 1920, No. 459, 389—408. (Cf. Furukawa and Tomizawa, J., 1919, 877 A.)

THREE samples of oil from wild plants had the following characters:—Sp. gr. 0.9985 at 15° C., 0.9543 at 15° C., 1.0488 at 19° C., saponif. value, 33, 23, —; saponif. value after acetylation, 66.6, 54.5, —; α_D (1 dm.) = -3.75°, -3.2°, -11.75°; methoxy content, 7.07%, —, 11.38%; phenol content, 20%, —, —. Thymol, methyleugenol, *p*-cymol (*p*-hydroxyisopropylbenzoic acid, m.p. 153° C.), a terpene, and a sesquiterpene, b.p. 136° C. at 12 mm. were identified in the first sample, thymoquinol and thymol in the second, and myristicin in the third sample. A sample of oil from the cultivated plant had a dark brown colour, sp. gr. at 15° C., 0.9006; acid value, 0; saponif. value, 37.0; saponif. value after acetylation, 49.2; α_D = -4.5°, methoxy content, 31%. Thymoquinol (0.9%), thymol (13%), and a terpene were detected in the oil.—K. K.

Nitroglycerol; Determination of —. H. D. Richmond. Analyst, 1920, 45, 260—265.

THE following method is described for the analysis of commercial nitroglycerol solutions (*Liquor trinitrini*, *L. glonoini*):—5 c.c. of the sample is mixed with 50 c.c. of water and titrated with *N*/10 barium hydroxide solution. After deducting 0.1 from the number of c.c. of alkali solution used, the result is divided by 1.02 (correction for slight hydrolysis). To determine the nitroglycerol, 5 c.c. of a 10% solution plus 5 c.c. of 90% alcohol, or 10 c.c. of a 5% solution, is treated with 25 c.c. of *N*/2 alcoholic sodium hydroxide solution; 10 c.c. of the latter solution is required for 20 c.c. of a 1% nitroglycerol solution. After 5 mins. phenolphthalein is added, and the excess of alkali titrated with *N*/1 acid. If more than 4 c.c. of acid is used the determination must be repeated, increasing the alcoholic strength, since the final solution must contain at least 81% of alcohol. The volume of alkali solution used is corrected for the acidity as found above; then 1 c.c. of *N*/1 alkali solution is equivalent to 0.05675 g. of nitroglycerol. If 2 c.c. of "100-vol." hydrogen peroxide is added before the addition of alkali, the final alcoholic strength has much less influence on the titration.—W. P. S.

Heat of combustion of carbon compounds. Richards and Davis. See IIA.

Oxidation of toluene. Nakao. See VII.

Decolorising carbon. Bock. See XVII.

Antipyrine in pyrimidone. Luce. See XXIII.

Glycol. Müller. See XXIII.

PATENTS.

Protocatechuic aldehyde; Manufacture of —. Confectionery Ingredients, Ltd., F. E. Matthews, A. T. King, and T. Kane. E.P. 145,871, 1.4.19.

THE acid chloride of any suitable acyl derivative of protocatechuic acid such as the acetate, benzoate, carbonate, or *p*-toluenesulphonate, or of the benzyl ether of protocatechuic acid is reduced to the corresponding derivative of protocatechuic aldehyde by treatment in an inert solvent at boiling temperature with hydrogen in presence of a hydrogenating catalyst, such as palladium or nickel. When the evolution of hydrogen chloride has ceased the aldehyde is recovered from the filtered liquid in the form of its bisulphite compound.—G. F. M.

Esters; Methods of forming —. E. C. R. Marks. From U.S. Industrial Alcohol Co. E.P. 147,337, 23.6.19.

ESTERS of the higher alcohols are produced by

partial esterification in presence of a dehydrating agent, separating the latter, and continuing the esterification at a higher temperature. For instance, a mixture of 3205 g. of fusel oil (sp. gr. 0.8268), 256 g. of sulphuric acid (sp. gr. 1.84), and 2564 g. of glacial acetic acid is kept for 24 hrs. at ordinary temperature, with occasional agitation. The aqueous layer containing all the sulphuric acid is separated, and the oily layer distilled at 100° C. until the separation of water ceases, indicating that esterification is complete. The ester layer in the distillate is from time to time separated from the water and returned to the still. The still contents are finally neutralised with 30% sodium hydroxide solution, the aqueous sodium acetate layer, which separates, being drawn off, and distilled at 100° C. to recover any amyl acetate it may contain.—G. F. M.

Silver salts of aliphatic α -amino acids; Manufacture of complex —. H. R. Napp. E.P. 148,074, 23.9.19.

COMPLEX silver salts of glycine are obtained by treating silver oxide or organic or inorganic salts of silver with a solution containing about 5 equivalents of the amino-acid, and crystallising the filtered solution at temperatures below 0° C. The complex salt obtained is more stable than the normal silver salt. It is soluble in water, to which it imparts an alkaline reaction. Sodium hydroxide causes no immediate precipitation of silver oxide, and no precipitate is produced with albumin. The compound has no irritating effect, and this, together with its other properties, renders it suitable for therapeutic purposes. Complex silver salts of other aliphatic α -amino-acids have similar properties, and are prepared by evaporating solutions of silver oxide or its salts in excess of the respective amino-acids.—G. F. M.

Medicinal oil preparations in dry form. H. V. Dunham. E.P. 148,587, 30.1.19.

SEE U.S. Pat. 1,302,487 of 1919; J., 1919, 511 A.

Soluble compounds from coal. G.P. 306,471. See IIA.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Printing-out papers; Recording the characteristic curve of —. F. Formstecher. Phot. Korrt., 1920, 57, 191—197.

A "FIRST" print on the paper to be examined is made by exposing through a paper-scale of known opacities. After rendering the base of this print transparent by soaking in paraffin oil or wax, a second print is made on any suitable paper through the first print and the exposure scale placed at right angles to one another. Lines of equal density, "Iso-opaken," on the second print represent the required characteristic curve.—B. V. S.

Print-out papers; Colour [of the image] in — and factors affecting it. F. Formstecher. Deuts. Opt. Wochenschr., 1920, 33—34. Chem. Zentr., 1920, 91, IV., 156.

THE colour of the printing image depends on the size of the precipitated silver particles, which is affected by the initial condition of the sensitive silver salt and also by the strength of the printing light and by moisture. Emulsions with a low proportion of citrate give blue images, the particles becoming finer grained and the colour redder with increasing quantities of citrate. The redder images have also a softer gradation.—B. V. S.

XXII.—EXPLOSIVES ; MATCHES.

Nitrocellulose; Viscosity of solutions of — in mixtures of acetone and water. I. Masson and R. McCall, Chem. Soc. Trans., 1920, 117, 819—823. (Cf. J., 1920, 172 r).

THE viscosity of sols of nitrocellulose in anhydrous or aqueous acetone rises rapidly within a few minutes of mixing to a maximum value and then gradually falls, and the same is true with ether-alcohol sols of minimum viscosity, but the more widely the composition of the solvents deviates from this "optimum" value the longer is the time taken in attaining the highest viscosity. The viscosities of nitrocellulose in anhydrous acetone are relatively high, but traces of water have a marked effect in lowering the viscosity down to a certain minimum value, after which it again rises with further addition of water, until when upwards of 12% of water is present the liquid no longer permits of the dispersion of the nitrocellulose. The optimum solvent composition at which viscosity is at a minimum is not the same for all kinds of nitrocellulose, nor is it quite independent of concentration; in fact, the gel absorbs selectively varying quantities of the fluid, and hence the composition of the fluid phase at equilibrium is not necessarily the same as that of the solvent before admixture with the nitrocellulose. None of the equations which have been hitherto suggested for correlating the viscosity of sols with the concentration of the disperse phase is applicable to the sols under discussion, except over small ranges of concentration.—G. F. M.

Blasting explosive; Modified TNT (mixture of Trojan grenade powder and trinitrotoluene) as a —. C. E. Munroe and S. P. Howell. U.S. Bureau of Mines. Reports of Investigations, June, 1920.

THE composition of Trojan grenade powder is: Moisture, 0.71%; nitrostarch, 21.37%; oils, 1.13%; sodium nitrate, 37.62%; ammonium nitrate, 33.34%; ammonium chloride, 0.39%; carbon and ash, 2.44%. It gave the following results: Velocity of detonation, 3741 m. per sec.; Trauzl block, 216 c.c.; small block, 15.9 mm.; pressure, 8836 kg. per sq. cm.; unit defective charge, 256 g.; calorimeter, 1037.1 cal. per kg.; volume of gas, 131.7 l. Comparative tests with 40% ammonia-dynamite gave: 3157 m. per sec.; 202 c.c.; 12.8 mm.; 8921 kg. per sq. cm.; 243 g.; 1122 cal. per kg.; 140 l. Large surplus stocks of Trojan grenade powder deteriorated on storage on account of deliquescence and when submitted to the influence test with a No. 8 detonator were found to be too insensitive for use. A mixture of equal parts of the powder and trinitrotoluene, however, proved to be a satisfactory explosive and was styled "modified TNT." The craters produced on explosion were larger than those obtained with trinitrotoluene alone. The mixture is free from dust and is consequently more readily packed, with less risk to the health of the workers. It may be packed in paraffined paper cases, a cartridge measuring 14" x 8" having apparent sp. gr. 1.15, or it may be used in loose form in containers. A detonator of the same grade as required for trinitrotoluene must be used.

—W. J. W.

Explosives; Apparatus for determining the explosion temperature of —. A. Langhaus. Z. ges. Schiess- und Sprengstoffw., 1920, 45, 161—163.

THE apparatus comprises a rectangular or cylindrical lead block surrounded on all sides except the top by an iron or copper jacket, and having drilled sockets in which are placed test tubes containing the samples of explosive, an additional one being provided for a thermometer. To reduce the

weight of the apparatus it may be constructed of iron, copper, or brass, and drilled to admit of the insertion of small leaden containers, preferably of conical shape to facilitate their removal for re-melting. To avoid adherence of the lead to the block, an iron or copper sheath may be used. The explosive may be kept under observation during the test by a suitable arrangement of lateral and vertical holes and inclined mirrors.—W. J. W.

Nitrometer; Explosions during operations with the —. Witt. Z. ges. Schiess- und Sprengstoffw., 1920, 45, 145.

NITROGLYCERIN and trinitrotoluene residues, obtained by extraction with ether in a Soxhlet apparatus, and then dried in a vacuum desiccator and treated with sulphuric acid for the nitrogen estimation, were found to ignite when transferred to the nitrometer. In some cases an explosion resulted during shaking in the apparatus. Impure ether is the origin of the trouble, to avoid which the purest quality must be used; the extract is then completely dried in a vacuum desiccator and preferably heated to 50° C., only a very slight loss of nitroglycerin resulting at this temperature.

—W. J. W.

Nitric esters. Farmer. See XX.

PATENTS.

Nitrocellulose; Stabilisation of — and apparatus therefor. B. van der Laan. E.P. 147,378, 2.10.19.

FOR stabilising and simultaneously sub-dividing nitrocellulose use is made of a rotary boiler of wood or aluminium provided with paddles and having two opposite manholes. Steam is introduced by means of a pipe through the axis of the vessel, exhaust steam escaping through a pipe which is separated from the interior of the boiler by a sieve bottom. The contents may be discharged into a hopper, whence they can be passed to the pulping machine by pumps. The process ensures energetic and rapid stabilisation of the material, economises steam consumption, and simplifies the discharge of the stabilised nitrocellulose.—W. J. W.

Explosives; Gelatinisation of cellulose nitric esters in the manufacture of —. W. Rintoul and T. J. Nolan, Assrs. to Nobel's Explosives Co., Ltd. U.S.P. 1,318,711, 3.8.20. Appl. 19.5.19.

SEE E.P. 131,389 of 1918; J., 1919, 795 A.

Explosive substances; Preventing danger of explosion in the preparation and handling of dangerous —. G. Darier and C. Gondet, Assrs. to Soc. d'Etudes Chim. pour l'Ind. U.S.P. 1,349,111, 10.8.20. Appl., 14.9.18.

SEE E.P. 130,166 of 1918; J., 1919, 712 A.

XXIII.—ANALYSIS.

Potassium hydrogen phthalate; Use of — as standard in alkalimetry. F. D. Dodge. J. Amer. Chem. Soc., 1920, 42, 1655—1656.

POTASSIUM hydrogen phthalate for use as a standard in alkalimetry should be crystallised above 20° C., whereby the formation of a more acid salt, $2\text{KHC}_8\text{H}_4\text{O}_4 \cdot \text{C}_8\text{H}_4\text{O}_4$, is effectually prevented. (Cf. J., 1920, 530 A.)—J. F. S.

Magnesia mixture. O. Kulmt. Chem.-Zeit., 1920, 44, 5*6.

MAGNESIA mixture for the precipitation of phosphoric acid if prepared without the addition of ammonia may be kept indefinitely in glass bottles without the glass being attacked. The requisite quantity of ammonia is added after the phosphate solution has been treated with a slight excess of the reagent.—W. P. S.

Metals; Use of organic solvents in the quantitative separation of —. III. Separation of magnesium from sodium and potassium chlorides. S. Palkin. J. Amer. Chem. Soc., 1920, 42, 1618—1621.

THE chlorides of magnesium, sodium, and potassium are dissolved in the minimum quantity of water (about 1.5 c.c. for 0.5 g.) in a 100—150 c.c. beaker flask, 1 drop of concentrated hydrochloric acid is added, and gradually 25 c.c. of absolute alcohol is dropped into the middle of the beaker while rotating; the sodium and potassium chlorides should precipitate in a uniform granular condition. In a similar manner, while rotating the beaker 25 c.c. of ether (U.S.P.) is added. When the precipitate has agglomerated and the supernatant liquid is nearly clear, the mixture is filtered through a weighed Gooch crucible into a 150 c.c. conical flask, using only mild suction to avoid drying the precipitate too soon. The beaker is washed with a mixture of 1 pt. of alcohol and 4 pts. of ether and the washings passed through the filter. The filtrate is evaporated to dryness and the residue treated with 10 c.c. of alcohol. One drop of concentrated hydrochloric acid is added and the mixture warmed until practically all has dissolved. The beaker is then rotated, 50 c.c. of ether slowly added, and the mixture kept for 20 min. The precipitate is collected in the same Gooch crucible, washed as before, then dried, ignited, and weighed. The filtrate is evaporated to dryness, the residue treated with 5—10 c.c. of hydrochloric acid and a little water, and the magnesium estimated in the usual manner.—J. F. S.

Halogens in organic compounds; Liquid ammonia-sodium method for the estimation of —. F. B. Dains and R. Q. Brewster. J. Amer. Chem. Soc., 1920, 42, 1573—1579.

CHLOROFORM, bromoform, chloral hydrate, bromal hydrate, methyl cyanide, benzyl cyanide, cyanoacetic ester, ethylidene chloride, tetrachloroethylene, and acetylene tetrachloride all form considerable amounts of sodium cyanide during the estimation of halogens by the liquid ammonia-sodium method (J., 1918, 448A). The halogen in these substances may be estimated by Clifford's modification of the process (J., 1919, 663A). (Cf. J.C.S., Oct.).—J. F. S.

Nitrotes and nitrites, antipyrine (in pyrimidine), phenol, and α - and β -naphthols; Colour tests for —. A. Escaich. J. Pharm. Chim., 1920, 22, 138—141.

IN applying the test for nitrates, previously described (J., 1918, 484A), to waters rich in chlorides, it is advisable, after reduction with amalgamated aluminium and filtration, to add sufficient silver nitrate solution to precipitate the chlorides, and then to filter before applying the colour test for nitrites. Magnesium may be activated similarly to aluminium, by immersion in mercuric cyanide solution, and is preferable to the latter metal for the detection of nitrates in diluted wines. For the detection of antipyrine in pyrimidine 1 g. dissolved in 10 or 20 c.c. of water is treated with 1 or 2 drops of a 5% solution of alkali nitrite and 5 drops of sulphuric acid. When the transient blue colour which appears has vanished, addition of 0.05—0.1 g. of lead peroxide will produce, if antipyrine was present, a violet colour changing to permanent cherry-red. The following modification of the quinone-imide test for phenol (or aniline after its conversion into phenol by diazotising and boiling with water) is very sensitive:—10—15 c.c. of the solution to be tested is treated with 5—10 drops of ammonia, 0.05—0.1 g. of sodium persulphate, and 4 or 5 drops of N/10 silver nitrate solution. A green coloration is produced which rapidly inten-

sifies and changes to blue for concentrations of phenol exceeding 0.05 g. per litre, and which becomes yellow for lower concentrations. α -Naphthol in aqueous alcoholic solution, treated with sodium nitrite and a few drops of acid mercuric sulphate solution, produces a fine red coloration and then a precipitate resembling mercuric iodide. β -Naphthol yields a yellowish precipitate under similar conditions.—J. H. L.

Glycol; Determination of —. B. Müller. Chem.-Zeit., 1920, 44, 513—515.

THE bichromate and acetin methods used in glycerol analysis are trustworthy for the determination of ethylene-glycol, but the acetin method can be applied only when the glycol is in a concentrated form. In the bichromate method the mixture must be heated for 4 hrs. to ensure complete oxidation; the excess of bichromate used may be determined iodometrically. Oxidation of the glycol with chromic acid and sulphuric acid and gravimetric determination of the carbon dioxide formed also yields accurate results. Benedikt and Zsigmondy's permanganate method cannot be used for the determination of ethylene-glycol, since the latter is not oxidised completely to oxalic acid.—W. P. S.

Spectrophotometry; Combination of fractionation with — in proximate organic analysis. W. E. Mathewson. J. Amer. Chem. Soc., 1920, 42, 1277—1279.

THE spectrophotometric method of estimating dye-stuffs may be applied to colourless organic compounds by first converting them into coloured compounds, which are separated from other coloured substances by extraction with suitable solvents. Procedures are described for the estimation of sulphanic acid, acetone, and β -naphthol depending on their condensation respectively with picryl chloride, 2,4-dinitrophenylhydrazine, and diazotised sulphanic acid.—J. K.

See also pages (A) 620, *Coal* (Strache); 621, *Gasoline in natural gas* (Anderson and Hinckley), *Extracting bitumens* (Walczak and Rice); 622, *Free carbon in tar etc.* (Hodurek); 625, *Hydrosulphides, sulphides, etc.* (Wöber), *Ferric oxide precipitates* (Toporescu); 626, *Phosphates* (Seeligmann); 630, *Corrosion* (Feurer), *Molybdate* (Bonardi); 632, *Sulphonated oils* (Kern), *Polenske and Reichert-Meißl values* (Kirkham), *Paints* (Hallett), *Shellac, varnish, etc.* (Crossley); 634, *Sucrose* (Jackson and Gillis); 635, *Dextrose and hydrogen cyanide* (Bougault and Perrier); 637, *Sulphuric acid unsuitable for use in Gerber's butyrometer* (Reiss), *Proteins* (Andersen); 638, *Phenyl derivatives of arsenious acid* (Fleury), *Quinol* (Messner); 639, *$\beta\beta'$ -Dichloroethyl sulphide* (Hollely), *Tetranitromethane and hydrazine* (Baillie and others); 641, *Explosives* (Langhans), *Explosions during operations with the nitrometer* (Witt).

PATENTS.

Coal; Apparatus for analysing or estimating volatile constituents of —. A. Naito. E.P. 147,254, 7.4.19.

A WEIGHEN sample of coal is distilled in a closed crucible, and the gas is led into kerosene in a closed flask fitted with a reflux condenser. Water accumulates at the bottom of the flask, and tar is dissolved in the kerosene, while the gas passes on to a measuring bell immersed in a tank of water, the volume of gas being measured by the amount of water drawn off from the tank. When distillation is complete, the water is separated from the kerosene and measured, while the increase in the weight of the kerosene indicates the amount of tar dissolved.—W. F. F.

Hardness of materials; Appliances for determining the [ball] —. A. Herbert and P. V. Vernon. E.P. 118,056, 22.8.19.

Hardness of metals; Means of testing the —. B. P. Haigh. E.P. 141,076, 27.5.19.

Hygrometers actuated by animal or vegetable fibre. F. L. Halliwell. E.P. 111,141, 18.8.19.

Gas-analysis apparatus. O. Rohde, Assr. to Svenska Aktiebolaget Mono. U.S.P. 1,340,838, 18.5.20. Appl., 23.11.18.

SEE F.P. 480,661 of 1916; J., 1917, 166.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Curran, Johns, Lowe, and Trescott. Retorts. 25,481. Sept. 3.

Facks. Apparatus for grading and separating dry powdered material. 25,511. Sept. 4.

Garrett. Filling for rectifying columns of liquor stills or gas-scrubbing towers. 26,019. Sept. 10.

Griffiths. Continuous-tunnel kilns. 25,714. Sept. 7.

Hurrell. Filters and filter-presses. 25,937. Sept. 9.

Johns. Distillation of material carrying volatile matter. 25,391. Sept. 2.

Johns. Vapour-condensing apparatus. 25,392. Sept. 2.

Jorgensen. Manufacture of heat-exchanging bodies. 25,386. Sept. 2.

Kreutzberg. Pulverising apparatus. 26,001. Sept. 9.

Low and others. 25,997. See IX.

Morgan, and Thermal Industrial and Chemical Research Co. Method of immersing subdivided solids or liquids in liquids. 25,801. Sept. 7.

Pfannenschmidt. 25,585. See VII.

Robinson, and Robinson and Son. Apparatus for separating solid particles from air. 25,299. Sept. 2.

Sanford. Grinding-machine. 25,223. Sept. 1.

Skinner. Conducting furnace operations. 25,531. Sept. 4.

Soc. Franco-Belge de Fours à Coke. Apparatus for causing air or gas to bubble into a liquid. 25,880. Sept. 8. (Belg., 11.3.20.)

Zack. Raising liquids easily evaporating at low temperature and standing under low pressure. 25,121. Sept. 3. (Switz., 10.10.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

9301 (1918). Elektrochem. Werke. Furnace for carrying out endothermic gas reactions. (116,503.) Sept. 15.

3046 (1919). Dick. Evaporating apparatus. (150,007.) Sept. 8.

4740 (1919). Lunden. See XIX.

8111 (1919). La Bour. Effecting intimate contact between liquids and gases for evaporating, cooling, concentrating, washing, etc. (125,380.) Sept. 15.

10,035 (1919). Stokes. Refrigerating apparatus. (150,017.) Sept. 8.

13,339 (1919). Boberg, and Techno-Chemical Laboratories, Ltd. Drying processes and apparatus. (150,068.) Sept. 8.

13,553 (1919). Smith & Co. Grinding-machines. (127,589.) Sept. 8.

15,812 (1919). Smallwood. Furnaces. (150,467.) Sept. 15.

15,928 (1919). Ochterbeck. Crushing-machines. (128,936.) Sept. 8.

16,571 (1919). Whitfield. Tunnel ovens or kilns. (150,482.) Sept. 15.

17,128 (1919). Soc. Anon. l'Oxyhydrique Franç. Apparatus for intensifying the mutual action of gases with liquids or solids during mixing. (129,978.) Sept. 15.

18,299 (1919). Somerville. Kilns. (150,157.) Sept. 8.

3896 (1920). Metallbank u. Metallurg. Ges. Evaporating liquors. (140,059.) Sept. 15.

7385 (1920). Crida. Control of drying processes. (140,101.) Sept. 8.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Banyard. Carbonisation of coal. 25,020. Aug. 30.

Carmichael and Guillaume. 25,072. See XII.

Curran and others. 25,181. See I.

Deutsche Koks-Gas Ges. Water-gas producers. 25,570. Sept. 4.

Duckham, and Woodall, Duckham, and Jones. Gasification of coal. 25,255. Sept. 1.

Dulac, Lajoux, and Lemoine. Transforming liquid fuel into solid fuel. 25,908. Sept. 8.

Elias. Illuminant by incandescence. 25,334. Sept. 2.

Farbw. vorm. Meister, Lucius, u. Brüning. Desulphurising gases. 25,151. Aug. 11. (Ger., 2.7.20.)

Johns. 25,391. See I.

Lewis. Method of carbonising coal etc. 25,151. Aug. 21.

Schott. Manufacture of briquettes. 25,111. Aug. 31.

Willemse. Production of water-gas. 25,750. Sept. 7.

COMPLETE SPECIFICATIONS ACCEPTED.

8413 (1919). Smith. Treatment of coal, especially in the manufacture of fuel briquettes. (125,379.) Sept. 15.

14,086 (1919). Ronseau. Production of hydrocarbon liquids from carbonaceous materials. (132,190.) Sept. 15.

15,126 (1919). U.S. Industrial Alcohol Co. Aeroplane motor fuel. (125,917.) Sept. 8.

15,916 (1919). Galusha. Gas-producers. (129,971.) Sept. 8.

31,799 (1919). Woodall, Duckham, and Jones, and Duckham. Introduction of steam into vertical retorts or ovens. (150,589.) Sept. 15.

2789 (1920). Muhlfeld. Burning pulverulent or liquid fuel in furnaces. (150,230.) Sept. 8.

III.—TAR AND TAR PRODUCTS.

COMPLETE SPECIFICATION ACCEPTED.

13,619 (1919). Tcherniac. Reduction of aromatic nitro-compounds. (150,412.) Sept. 15.

IV.—COLOURING MATTERS AND DYES.

APPLICATION.

Cassella und Co. Manufacture of a colour of the anthraquinone series. 25,263. Sept. 1. (Ger., 1.9.19.)

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Hollaender. Production of a plastic substance. 25,079. Aug. 31.

Hollaender. Production of artificial fibres, films, etc. 25,080. Aug. 31.

Jenkins. Apparatus for drying, cleansing, or carbonising wool etc. 25,417. Sept. 3.

Jenner. Apparatus for drying varnished paper etc. 25,061. Aug. 31.

Kirschbraun. Waterproof paper and process of making same. 25,753. Sept. 7.

Marx. Paper-making etc. machines. 25,092. Aug. 31.

COMPLETE SPECIFICATION ACCEPTED.

29,474 (1919). Gratz. De-acidification of raw wood pulp liquor. (150,171.) Sept. 15.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Boucherie. Impregnating animal and vegetable fibres. 25,160. Aug. 31. (Fr., 15.6.20.)

Hindle. Printing cotton and woven fabrics. 25,493. Sept. 4.

Leach, Pollitt, and Willows. Production of pattern effects in cotton etc. fabrics and silk. 25,294. Sept. 2.

Surpass Chemical Co. Process of dyeing. 26,079. Sept. 10. (U.S., 30.1.20.)

COMPLETE SPECIFICATION ACCEPTED.

29,410 (1919). Watremez. Scouring fibres of vegetable origin to facilitate and accelerate subsequent bleaching. (139,457.) Sept. 8.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Goldschmidt A.-G. Production of alkali sulphate and hydrochloric acid. 26,001. Sept. 9. (Ger., 9.9.19.)

Hansford. Manufacture of neutral sulphate of ammonia. 25,505. Sept. 4.

Nitrogen Corporation. Production of sodium bicarbonate and hydrogen. 25,359. Sept. 2. (U.S., 7.2.20.)

Pattison (Mathieson Alkali Works). Recovery of ammonia. 25,402. Sept. 3.

Pfannenschmidt. Acid chambers, acid towers, acid mains, etc. 25,585. Sept. 6. (Ger., 8.9.19.)

Robertson, and Robertson & Sons. Recovery of high-boiling acid. 25,206. Sept. 1.

Soc. l'Air Liquide. Direct synthesis of ammonia. 25,766. Sept. 11. (Fr., 12.9.19.)

Terrell. Preparation of thoria and thorium salts. 25,435. Sept. 3.

United Alkali Co. (Barker). Electrolytic cells for producing alkali chlorates. 26,104. Sept. 10.

Zack. Separating oxygen and nitrogen. 25,423. Sept. 3. (Switz., 15.10.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

15,296 (1919). Carborundum Co. (Hutchins). Purifying aluminous materials. (150,116.) Sept. 8.

15,765 (1919). Marks (Nitrogen Products Co.). Apparatus for fixation of nitrogen. (150,127.) Sept. 8.

31,909 (1919). Pestalozza. Electrolytic apparatus for direct production of hypochlorites. (150,214.) Sept. 8.

32,672 (1919). Fabr. de Prod. Chim. de Thann et de Mulhouse. Manufacture of potassium sulphate. (137,519.) Sept. 15.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

British Thomson-Houston Co. (General Electric Co.). Finishing glass. 25,982. Sept. 9.

Clarke and Gaudin. Drying china clay. 25,075. Aug. 31.

Marino. Metallising articles of porcelain, china, etc. 26,175. Sept. 11.

COMPLETE SPECIFICATIONS ACCEPTED.

674 (1920). British Thomson-Houston Co. (General Electric Co.). Substances for coating glass surfaces. (150,598.) Sept. 15.

15,973 (1920). Ueda. Process for treating glass surfaces. (144,737.) Sept. 15.

IX.—BUILDING MATERIALS.

APPLICATIONS.

John. Roofing, walling, etc. material. 25,137. Aug. 31.

Lloyd and Maclean. Manufacture of cement. 26,017. Sept. 10.

Low, Smith, and Tullock. Composition for heat insulation. 25,997. Sept. 9.

Ziekgraf. Production of artificial limestone. 26,048. Sept. 10.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Baines, Clarke, and De Lavand. Heat treatment of metallic etc. subjects. 25,145. Aug. 31.

British Thomson-Houston Co. (General Electric Co.). Arc-welding compositions. 25,888. Sept. 8.

Cyclops Steel Co. Alloys. 25,788. Sept. 7. (U.S., 3,10,19.)

De Bats. Melting and casting metals. 25,018. Aug. 30.

Erikson. Process and furnace for reducing metallic oxides. 25,652. Sept. 6.

Festa. Furnaces for fusion of steel etc. 25,262. Sept. 1. (Fr., 5,12,19.)

Schiff and Vivian. Conversion and separation of complex ores and minerals. 25,605. Sept. 6.

Wichmann. Manufacture of alloys. 25,218. Sept. 1.

COMPLETE SPECIFICATIONS ACCEPTED.

7186 (1914). Chiapponi, Hesse, and Rauschenplat. Removal of iron from oxide or roasted sulphide tin ores. Sept. 15.

10,338 (1919). Speer. Chrome nickel steel. (150,020.) Sept. 8.

12,796 (1919). Whyte. Case-hardening, tempering, etc. (150,035.) Sept. 8.

12,908 (1919). Cohen and Catterall. Furnaces for melting and refining metals. (150,012.) Sept. 8.

13,247 (1919). Coles. Continuous electrolytic production of copper wire. (150,063.) Sept. 8.

14,151 (1919). British Thomson-Houston Co. (General Electric Co.). Annealing metal sheets. (150,137.) Sept. 15.

14,517 (1919). Gaunt, Walters, Marle, and Gibbons Bros. Furnaces for heat treating metals. (150,099.) Sept. 8.

15,109 (1919). Martin. Water concentration of ores. (150,113.) Sept. 8.

16,411 (1919). Wurstenberger and Frei. Manufacture of fluid for preventing selective corrosion of copper tubes etc. (130,323.) Sept. 8.

16,833 (1919). Mellor. Reduction of metals to powder. (150,490.) Sept. 15.

29,504 (1919). Electrolytic Zinc Co. Recovery of zinc by electrolysis. (136,151.) Sept. 15.

1013 (1920). International Nickel Co. Manufacture of nickel and copper. (138,600.) Sept. 15.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Davey, and Electro Metals, Ltd. Operating electrodes of electric furnaces. 25,965. Sept. 9.

Davis. Storage batteries. 25,035. Aug. 30.

Gonin and Roesel. Alkaline storage batteries. 25,999. Sept. 9. (Fr., 10,9,19.)

Powell. Electric batteries. 25,191. Sept. 1.

Stevens. Electric accumulators. 25,825. Sept. 8.

United Alkali Co. (Barker). 26,101. *See VII.*

COMPLETE SPECIFICATIONS ACCEPTED.

13,247 (1919). Coles. *See X.*

20,920 (1919). Gardiner and Duggan. Storage batteries. (150,171.) Sept. 8.

21,582 (1919). De Luca. Electric furnaces. (150,532.) Sept. 15.

29,504 (1919). Electrolytic Zinc Co. *See X.*

31,909 (1919). Pestalozza. *See VII.*

363 (1920). Jackson (Allen Electrolytic Cell Corp.). Electrolytic cells. (150,595.) Sept. 15.

651 (1920). Coles. Zinc battery elements. (150,597.) Sept. 15.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Carmichael and Guillaume. Apparatus for extracting, distilling, etc. oils, greases, sewage, etc. 25,072. Aug. 31.

Dayton Metal Products Co. Hydrogenation apparatus. 25,141. Aug. 31. (U.S., 5,10,18.)

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Barrett Co. Manufacture of resins. 25,811. Sept. 7. (U.S., 8,3,20.)

Hollaender. 25,079. *See V.*

Mitchell. Manufacture of lithopone. 25,282. Sept. 1.

COMPLETE SPECIFICATIONS ACCEPTED.

24,606 (1919). Isaacs. Coating compositions or paints. (150,551.) Sept. 15.

7488 (1920). Pooley and Strevens. Extraction of gum from grass trees. (150,638.) Sept. 15.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Crouch and Speedy. Rubber-mixing. 25,095. Aug. 31.

Feldenheimer, Plowman, and Schidrowitz. Manufacture of rubber. 25,813. Sept. 7.

Goodyear Tire and Rubber Co. Manufacture of vulcanised caoutchouc. 25,810. Sept. 7. (U.S., 10,11,19.)

Kenny and Oetberg. Rubber material. 25,474. Sept. 3.

COMPLETE SPECIFICATION ACCEPTED.

12,928 (1919). Dunlop Rubber Co., Worthington, and Hyde. Treatment of raw rubber. (150,043.) Sept. 8.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

British United Shoe Machinery Co., and Reid. Finishing etc. leather goods etc. 25,050. Aug. 30.

Turner Tanning Machinery Co. Machine for treating hides, skins, and leather. 24,969. Aug. 30. (U.S., 30,8,19.)

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Johnson (Badische Anilin und Soda-Fabrik) Manufacture of fertilisers. 25,892. Sept. 8.

Soc. d'Etudes Chim. pour l'Industrie. 25,336. *See XX.*

Soc. d'Etudes Chim. pour l'Industrie. Manufacture of manures. 25,337-8, 25,340-1, 25,344-5. Sept. 2. (Switz., 26,9 and 15,11,19, 2,3,20.)

COMPLETE SPECIFICATION ACCEPTED.

10,592 (1919). Young and Watson. Utilising sewage sludge to produce marketable manure and other products. (150,375.) Sept. 15.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

White (Perkins Glue Co.) Modifying or converting starch. 26,074. Sept. 10.

COMPLETE SPECIFICATION ACCEPTED.

31,741 (1919). Ballochmyle Creamery Co., and McCrone. Process of sugar-boiling. (150,588.) Sept. 15.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Herriott (Deerr). Recovery of alcohol and concentrated slop from fermented materials. 26,159. Sept. 11.

Jensen. Manufacture of yeast. 26,152. Sept. 11. (Denmark, 12.9.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

7552 (1917). Weizmann. *See* XX.

11,302 (1917). Diamalt A.-G. *See* XX.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Atmosteral, Ltd., and Dunsford. Antiseptics, disinfectants, etc. 26,086. Sept. 10.

Bate and Dexter. Utilising chlorpicrin. 25,773. Sept. 7.

Carmichael and Guillaume. 25,072. *See* XII.

Clayton. Preservatives for margarine, butter, cream, etc. 25,034. Aug. 30.

Cocks and Wayman. Beverage. 25,225. Sept. 1.

Hardcastle. Food for animals etc. 25,195-6. Sept. 1.

Springhorn. Treatment of peat moss for purification of sewage effluents etc. 25,485. Sept. 3.

COMPLETE SPECIFICATIONS ACCEPTED.

4740 (1919). Linden. Drying sewage sludge or other wet material. (150,368.) Sept. 15.

10,592 (1919). Young and Watson. *See* XVI.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Dayton Metal Products Co. 25,144. *See* XII.

Röhm und Haas. Manufacture of alkylene-cyanhydrins. 25,261. Sept. 1. (Ger., 3.9.19.)

Sander. Production of synthetic alcohol. 25,738. Sept. 7.

Soc. d'Etudes Chimiques pour l'Industrie. Conversion of cyanamide into urea. 25,336. Sept. 2. (Switz., 26.9.19.)

White (Spirocid Corp.). Producing medicinal compounds. 25,471. Sept. 3.

COMPLETE SPECIFICATIONS ACCEPTED.

7552 (1917). Weizmann. Fermentation processes for producing acetone and butyl alcohol. (150,360.) Sept. 15.

11,302 (1917). Diamalt A.-G. Production of saccharic acid and tartaric acid from carbohydrates. (108,494.) Sept. 15.

13,502 (1919). Imray (Soc. Chem. Ind. in Basle). Manufacture of allyl ester of 2-phenylquinoline-4-carboxylic acid. (150,401.) Sept. 15.

13,619 (1919). Tcherniac. *See* III.

22,482 (1919). Soc. des Acieries et Forges de Firminy. Catalytic preparation of acetic acid from acetylene. (132,529.) Sept. 8.

12,753 (1920). Bregeat. Recovery of camphor from gaseous mixtures. (150,654.) Sept. 15.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Middleton. Transfer-processes. 25,619. Sept. 6.

Middleton. Production of three-colour heliochromes. 25,620. Sept. 6.

Mimesa A.-G. Toning baths. 25,762. Sept. 7.

XXII.—EXPLOSIVES; MATCHES.

APPLICATIONS.

Claessen. Manufacture of gelatinous explosives. 25,802. Sept. 7. (Ger., 22.4.20.)

Jones and Kelly. Explosives. 24,984. Aug. 30.

Lundsgaard. Explosives. 25,486. Sept. 3.

I.—GENERAL; PLANT; MACHINERY.

Evaporator design; Studies in —. W. L. Badger and P. W. Shepard. Amer. Inst. Chem. Eng., 27.6.20. Chem. and Met. Eng., 1920, 23, 237-241, 281-281.

This is the first contribution from the Evaporator Experiment Station of the University of Michigan, and deals with the influence of temperature drop and temperature level on heat transmission in a vertical tube evaporator. The following conclusions from the experimental results are given, with the reservation that they must be applied with caution to other types of apparatus. The apparent coefficient of heat transmission in vertical tube evaporators decreases with decrease in temperature drop and approaches zero for zero temperature drop. It increases with boiling point for a given temperature drop (which also means increasing steam temperature). Correcting the apparent heat transmission coefficients for hydrostatic head gives figures which change but little with temperature drop. This remaining change is probably due to increased circulation at increased rates of boiling. The corrected heat transmission coefficients used show considerable differences when the boiling point is increased and the temperature drop held constant. It is impossible to represent heat transmission coefficients for different boiling points, different temperature drops, and different steam temperatures by any expression which does not consider at least two of these three factors.—W. H. C.

Evaporating apparatus; Study of condensation in —. E. Depasse. Bull. Assoc. Chim. Sucr., 1920, 37, 434-444.

In evaporating a liquid particularly liable to incrustation, it was found that incrustation was most rapid where the temperature difference and the amount of evaporation per unit area of heating surface were greatest; in these cases the coefficient of evaporation, K (as defined by Horsin-Déon), was increased and incrustation diminished by reducing the temperature difference. From an investigation of a number of apparatus of different types it is concluded that K varies according to the construction of the apparatus and the nature and concentration of the liquid evaporated. Curves are given showing for the evaporation of pure water in a single unit the relation between the temperature of the heating steam, the temperature difference, and the rate of evaporation per unit area.—J. H. L.

Heat transfer in flues. L. H. Fry. Engineering, 1920, 110, 265-268.

The influence of length and dimensions of flue, the rate of flow of gas, and temperature drop on heat transference in flues has been studied by the aid of a double logarithmic (olog) formula. The results are shown in a number of tables of experimental results and of calculated data, and the formulæ have been applied to the published results of other experimenters to bring them into harmony.—W. H. C.

PATENTS.

Temperature-exchanging apparatus. E. Harter. E.P. 127,565, 6.5.19. Conv., 25.5.18.

A gas which is to gain or lose heat is passed through a centrifugal fan the rotor of which is surrounded by concentric rings of gilled tubes; the tubes carry hot or cold water or other liquid, and the gills are directly in the path of, but are so shaped as to avoid shock to, the issuing gas.—B. M. V.

Tunnel ovens and like structures; Cooling chamber for —. Dressler Tunnel Ovens, Ltd., and C. Dressler. E.P. 149,085, 11.5.19.

The cooling chamber of a tunnel oven is provided with a nest of horizontal or slightly inclined tubes connected with a water supply at the cooler end and with a steam drum at the end nearest the furnace. To prevent the heat of the furnace heating the water in the tubes directly, there may be interposed a passage, built of refractory material, which is only just large enough to permit the goods on their truck to pass.—B. M. V.

Tunnel kiln. P. A. Meehan, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. (A) 1,348,510 and (B) 1,348,511, 3.8.20. Appl., 5.12.19.

(A) A HORIZONTAL kiln for the heat treatment of articles has a port at one end for the entrance and exit of the carrier containing the articles. A high temperature zone is maintained at a distance from the port, and the carrier is reciprocated between the port and the high temperature zone. (B) The combustion chamber for the kiln described in (A) is divided into two longitudinal passages which communicate at one end, forming a continuous passage. Air and fuel enter at the free end of one of the passages, and burnt gas is drawn off at the free end of the other passages.—W. F. F.

Tunnel oven. F. K. Meiser. G.P. 320,800, 4.3.19. Addn. to 319,440 (J., 1920, 475 A).

Gas leads with detachable flexible connexions are arranged in parallel underneath the trolleys bearing the crucibles, each of which thereby obtains a fresh supply of gas.—G. F. M.

Kiln. C. E. Carpenter. U.S.P. 1,349,185, 10.8.20. Appl., 20.11.19.

The floor of a kiln contains a pair of crossed flues communicating at their point of intersection and an annular flue intersecting and communicating with the crossed flues. One of the crossed flues and the annular flue are open at the top to the interior of the kiln and one end of the other crossed flue is open to the stack.—W. F. F.

Pot furnace. E. L. Knoedler, Assr. to Welsbach Co. U.S.P. 1,349,159, 10.8.20. Appl., 1.5.18.

A POT is mounted on horizontal trunnions, so that it can be tipped, and a rotary stirrer is provided, mounted on an inclined axis so that the pot may be tipped without interfering with the stirrer.—W. F. F.

Kiln; Rotary —. A. E. Truesdell and L. F. Phillips. U.S.P. 1,350,750, 21.8.20. Appl., 20.10.16.

AN inclined rotary kiln is heated by combustible gas and primary air, which are supplied at its lower end. The portion of the kiln forming the burning chamber is surrounded by a jacket through which secondary air is passed to preheat it. The secondary air is delivered to the burning chamber at a point intermediate between its ends.—W. F. F.

Decantation apparatus. J. Denöel. E.P. 149,551, 14.10.19.

The liquid containing suspended matter flows in at the top of an inverted funnel, from under the lower edge of which the clear liquid flows up through the outer space to an overflow, while the settled mud flows continuously downwards to a bottom outlet, the apparatus is so proportioned that the liquid falls with decreasing velocity until it reaches the bottom edge of the funnel, and the velocity is not increased on the upward journey of

the clear liquid. To avoid shock and permit the escape of froth, the feed may be from an auxiliary tank to an inlet slightly below the surface of the liquid, and any air rising up the walls of the funnel is permitted to escape through an annular space around the inlet.—B. M. V.

Filter. W. D. Mount. U.S.P. 1,348,036, 27.7.20. Appl., 11.12.19.

A ROTARY drum filter is built up of two end flanges and a series of interchangeable grids forming the cylindrical surface. Each grid covers only a part of the surface, and the abutting edges of all the grids are secured by a single clamping device.

—W. F. F.

Filter. P. C. Forrester. U.S.P. 1,349,056, 10.8.20. Appl., 22.3.19.

A small container open at the top is arranged centrally within a reservoir near the top. The container accommodates two filtering bags, each of which is hinged at one side to the side of the container, the two hinges being on opposite sides. Each bag may be swung in a vertical plane, so that it is suspended over the reservoir to filter the liquid which is supplied to the reservoir, and the pivots of the two bags are geared together by racks and pinions, so that when one is in use over the reservoir the other is withdrawn into its container to dry the residue formed on it.—W. F. F.

Filter. E. J. Sweetland, Assr. to United Filters Corp. U.S.P. 1,349,238, 10.8.20. Appl., 30.3.15.

A FILTER plate has the central web portion reduced in thickness to provide drainage spaces when covered with filtering fabric on both sides. The fabric is supported on a sheet of reticulated material underneath, and means, such as a clamping ring, are provided for clamping the edges of the fabric to the plate and sealing the joint. The same ring also seals the joint between two adjacent filter plates.—W. F. F.

Filtering materials. J. H. Berrigan, Assr. to H. R. Worthington. U.S.P. 1,350,292, 24.8.20. Appl., 8.12.15.

LIQUID to be filtered is supplied to the interior of a casing and the filtered liquid is discharged from the interior of a hollow element having walls of filtering cloth supported in the casing. A hot fluid is continuously supplied to the interior of the filtering element during the filtering operation.

—W. F. F.

Filter. F. K. and E. F. Atkins. U.S.P. 1,350,433, 24.8.20. Appl., 20.9.16.

SOLID material is deposited on the outer surface of a filter cloth supported on a flat plate. To remove the cake, liquid is forced through the cloth from the inner side so as to raise it from the surface of the plate into contact with a scraper which extends over the active surface of the cloth, the scraper and plate being movable relatively to each other. The scraper is normally out of contact with the cloth.—W. F. F.

Dryer. G. L. Spencer. U.S.P. 1,348,757, 3.8.20. Appl., 24.7.19.

A VERTICAL cylindrical casing is provided with a concentric tube, open at both ends and extending from the bottom of the casing nearly to the top. The casing is divided into two parts by a horizontal partition having openings, which are covered by receptacles containing the material to be dried. The bottom of each receptacle is perforated and an electric heater is contained in a chamber below the casing communicating with the lower end of the vertical tube. When suction is applied to the lower compartment hot air is drawn upward through the tube and then downward through the material.—W. F. F.

Dehydrating plant. L. E. Post and D. A. Riedy. U.S.P. 1,349,908, 17.8.20. Appl., 9.6.19.

THE plant comprises a series of chambers each containing a number of superposed trays, together with means for leading hot air independently to each of the chambers, and for guiding the air within the chambers. Each chamber has an air outlet near the bottom leading into a central chamber, and the central chamber has an outlet to the atmosphere.

Drying solutions; Process of—. C. L. Dean. U.S.P. 1,350,072, 17.8.20. Appl., 27.4.18.

THE liquid is sprayed into a chamber and subjected to streams of hot, dry gases which are alternately convergent and parallel.—B. M. V.

Precipitating system; Electrical —. L. W. Chubb, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,349,362, 10.8.20. Appl., 6.1.17.

A DISCHARGE electrode and a co-operating rotary collecting electrode are placed in a treating chamber in positions such that permanently active and inactive zones of the electric field extending between them are formed. Suspended particles are precipitated electrically from fluid streams flowing between the electrodes, and the particles are collected on a stream boundary.—J. W. D.

[Fractionating apparatus.] Aerial condenser. L. P. Litzinger, P. F. Conerty, and W. Snodgrass. U.S.P. 1,349,681, 17.8.20. Appl., 19.6.19.

VAPOUR passes upwards from a still to both ends of a horizontal pipe forming an expansion chamber. Other horizontal pipes of decreasing capacity are superposed one above the other, both ends of each being connected with the pipe below. All the pipes are air-cooled. The lowest condensing pipe and the highest are connected by separate valved pipes with a liquid-cooled condenser.—W. F. F.

Separating solids from liquids; Apparatus for —. H. N. Herrick. U.S.P. 1,349,881, 17.8.20. Appl., 19.2.20.

A MIXTURE of solid and liquid is rotated by a centrifugal pump having means for retaining the solid matter and discharging it separately. The solid matter is received in a casing having an outlet normally closed but capable of being opened by means within the casing.—W. F. F.

Centrifugal clarifier. A. M. del Valle. U.S.P. 1,350,009, 17.8.20. Appl., 16.8.19.

A NUMBER of rotating concentric cylindrical pans communicate with one another, and liquid is supplied to the innermost pan. Scum is removed from the surface of the liquid in each pan by scoops attached to discharge pipes, and the clarified liquid is collected from the outermost pan.—W. F. F.

Liquid and gas contact apparatus. H. J. Zimmermann, Assr. to The G. A. Buhl Co. U.S.P. 1,350,202, 17.8.20. Appl., 5.7.18.

A LIQUID spray is delivered into the centre of a gas conduit, a portion of which is obstructed by a shield surrounding the spray nozzle, and the annular portion surrounding the shield is provided with helical baffles, so that the gas has a rotary motion on reaching the spray.—B. M. V.

Conveying and treating [powdered] materials; Process and apparatus for —. G. I. Rhodes and H. V. Coes. U.S.P. 1,350,337, 24.8.20. Appl., 13.11.17.

SOLID materials which are likely to be burned or oxidised in air are conveyed by a current of gas which is air-free and inert with respect to the

material; the latter is removed from the gas after arrival at its destination.—B. M. V.

Liquefaction of permanent gases; Counter-current apparatus with internal expansion engine for —. R. Mewes. G.P. 317,757, 29.8.15.

In order to avoid a rise in temperature of the wall of the liquefaction chamber near to the expansion engine, the driving shaft of the latter is carried through the chamber, emerging at the warmer end, and is then coupled outside to the engine.

—W. J. W.

Distilling and rectifying apparatus. R. Hübner. G.P. 318,477, 20.8.18.

The still is divided into two compartments, and the running off of the spent wash from the lower compartment and the passing of steam from the lower to the upper compartment are controlled by two valves on the same vertical rod in such manner that the opening of the valve in the steam supply pipe is accompanied by closing of the valve in the run-off pipe and *vice-versa*. The wash is fed into the upper compartment, in which part of the alcohol is distilled off, and is then passed through a valved pipe into the lower compartment, whilst the heating steam is introduced into the lower compartment and then passes into the upper compartment.—J. S. G. T.

Mixing gases and liquids; Process for —. K. Morawe. G.P. 318,766, 5.1.17.

FINELY-perforated sieves are arranged one above the other to form an enclosed chamber. The gas is forced under pressure through the lower sieve-plate, and the liquid is introduced through the upper one.—W. J. W.

Separating solids or liquids from gases; Filling material for apparatus for —. R. Forster. G.P. 318,889, 29.10.18.

PIECES of metal, earthenware, porcelain, etc., shaped like screw propellers are thrown haphazard into the separating chamber, and thus form an irregular, many-sided packing material which facilitates separation of solid or liquid particles from the gases. Choking of the channels and formation of inactive spaces are avoided.

—W. J. W.

Gases; Process for purification of very hot —. W. Freytag. G.P. 319,796, 1.4.19.

The gases are passed through molten slag of low viscosity which retains the dust. A portion of the gases may be used to heat the slag. A slag furnace having one or more chambers, and with pipes dipping into the slag-bath, may be used, and the slag-bath may be provided with a siphon which allows the slag to run off without escape of the gases.

—W. J. W.

Crystal masses; Method of obtaining large — with well-formed crystals from hot concentrated liquors. E. Waskowsky. G.P. 320,315, 22.8.17. Addn. to 306,173 (J., 1918, 724 A).

To lengthen the period of cooling of the liquor, it is first sprayed upwards either vertically or at an angle, and then allowed to drop into the crystallising vessel.—W. J. W.

Concentrating liquids; Process and apparatus for —. A. Herrlich. G.P. 320,662, 24.1.19. Addn. to 315,035 (J., 1920, 356 A).

The heating plates described in the original patent are grooved radially so as to give an externally-pointed surface to the hollow heating arrangements, and thus prevent accumulation of thick liquids, which are enabled to flow outwards and pass

through openings in the plate on to the next heater. The level of the liquid on the plates is maintained by overflow apertures.—W. J. W.

Evaporation of liquids which are liable to burn, such as sulphite cellulose liquors; Apparatus for the —. C. Tittel. G.P. 322,462, 17.11.17.

TROUGHS, divided into small compartments by parallel cross partitions with openings alternately at opposite ends, are arranged side by side in step formation. Each is provided with an overflow and independently operated heating coils, so that any desired number of successive troughs can be put into operation as a single unit, and, if need be, the liquid in different stages of concentration in the individual troughs can also be handled separately.—G. F. M.

Filtration and/or percolation of liquids. C. Yeoman. U.S.P. 1,349,798, 17.8.20. Appl., 2.10.16. SEE E.P. 11,287 of 1915; J., 1916, 1251.

Furnace. M. Mathy. U.S.P. 1,349,899, 17.8.20. Appl., 26.6.19.

SEE E.P. 131,169 of 1919; J., 1920, 1 A.

Separating solid particles from air employed for carrying off said particles from machinery or processes in which they are produced or given off; Apparatus for —. T. Robinson and Son, Ltd., C. J. Robinson, and T. J. Stevenson. E.P. 149,089, 16.5.19.

Moulds for casting chemical and other sticks. T. Tucker. E.P. 149,477, 31.5.19.

Conveying or transporting goods; Apparatus for — particularly as a device for charging furnaces. A. Smallwood. E.P. 149,495, 24.6.19.

Furnace [; Boiler — for burning powdered fuel]. J. U. McDonald. U.S.P. 1,349,971, 17.8.20. Appl., 15.9.19.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Lignite from the Rhine district; Gasification of —. J. Weiss and H. Becker. Stahl u. Eisen, 1920, 46, 1067—1073.

TESTS were made in a small producer of 1 m. diam. The air blast was preheated to 70° C. and contained 0.81% CO₂ due to contamination with flue gases. The average composition of the lignite through the trial was 57.1% moisture, 27.1% C, 2.3% H, 11.0% O, N and S, and 2.5% ash. The average yield of gas was 1.6 cb. m. per kg., with 8.6% CO₂, 0.21% O₂, 23.7% CO, 1.60% CH₄, and 12.5% H₂; calorific value 1085 cals. per cub. m. With briquettes containing 15% of moisture the gas contained 3.7% CO₂, 0.3% O₂, 30.0% CO, 2.0% CH₄, and 10.1% H₂. If the gas from raw lignite is dried, it is quite as good as gas from better coals. It can be dried by cooling in spray, Theisen- or disintegrator-washers, and the tar can be recovered from the liquor. Raw lignite can also be gasified in rotary-grate producers, no steam being admitted and the fuel level being kept 25" higher than with briquettes.—A. G.

Coke-oven engineering; Recent developments in by-product —. J. Becker and F. W. Sperr. Blast Furnace and Coke Assoc., Chicago. Gas World, 1920, 73, Coking Sect., 82—85.

THE new Koppers oven is a combination, cross-regenerative oven with triangular flues, and can be operated either with producer gas or with

coke-oven gas with waste gas return, or with straight coke-oven gas. The gas mains and stack flues are all on one side of the battery; this is of importance from the point of view of cost and economy of operation. There are two coal-gas distributing flues, the upper communicating with the nozzles on the half nearest to the gas mains, while the lower flue communicates with the nozzles on the far half of the wall. There are two lower gas supply mains. One of these is for producer gas and the other for returned waste gas. The waste gas outlets at the bottom of the regenerators are so placed that the passages from the regenerators on the far side of the battery pass along the bottom of the regenerators on the near side and communicate with the stack flue. Alternate regenerators serve for air and for producer gas, or, as the case may be, for air and for returned waste gas. Thus, all of the fuel gas and returned waste gas inlets as well as all of the waste gas outlets are brought together on the same side of the battery—an arrangement which greatly favours convenience of operation. The oven is designed to have a capacity of 7.6 net tons of coal, and is of such width as to work with 12 hrs. coking time with a daily capacity of 15.2 net tons of coal, producing 150,000 cub. ft. of gas. A plant having ample gas storage capacity can be operated with two 8-hr. shifts, only the heaters and producer operators being required on the third shift.—A. G.

Gas-producer process; Addition of carbon dioxide in the —. O. Essich. *Feuerungstech.*, 1920, 8, 184—185.

IN producers operating without steam, the temperature of the exit gases can be reduced by the addition of carbon dioxide to the air-blast. By the addition of 1 cub. m. of carbon dioxide to every 5 cub. m. of oxygen the temperature can be lowered from 1000° C. to 500° C. If the carbon dioxide is added in the form of waste gases the blast has a composition of 3.5% CO₂, 17.5% O₂, and 79% N₂. With hot waste gases the carbon dioxide can be increased by 0.5% for every 100° C. increase in temperature. The economy in coal amounts to CO₂ × 100 / (CO₂ + O₂)%. The amount of carbon dioxide added must be carefully regulated in proportion to the cross-section of the chamber.—A. G.

Gas producers. K. Bunte. *J. Gasbeleucht.*, 1920, 63, 541—545.

THE advantages of heating gas retorts by means of gas from a large central gas-producer plant are discussed, and recent improvements in the construction and operation of gas producers are described.

Mineral oils; Calculation of the viscosity of a mixture of two — from the viscosities of its components. H. Schwedhelm. *Chem.-Zeit.*, 1920, 44, 638.

THE viscosity, z , of the mixture may be calculated from the formula: $z = (n-d)[(m-d)/(n-d)]^p + d$, or $(z-d) = (n-d)^{1-p} \times (m-d)^p$, where m = the viscosity of the more viscous oil, n = that of the less viscous oil, and d = a constant; $p = r/(r+q)$, r being the volume of the more viscous oil and q that of the less viscous oil.—W. P. S.

Paraffin wax; Refractometric determination of — as a control test in the manufacturing process. M. Freund and G. Palik. *Petroleum*, 1920, 15, 757—758. *Chem. Zentr.*, 1920, 91, II., 816.

THE proportion of paraffin wax, x , may be calculated from the refractive index (n_3) of a mixture of paraffin wax and a filtrate oil, if the respective coefficients (n_2) and (n_1) are known: $x = (100n_3 - 100n_2) / (n_1 - n_2)$ and, conversely, the refractive index of the mixture may be calculated when the % of

paraffin wax is known: $n_3 = [xn_2 - (100-x)n_1] / 100$. The formulæ are only applicable when the sp. grs. of the constituents are practically the same. The results are lower than those obtained by the methods of Holde or Neustadt.—C. A. M.

Bituminous acid-proof coatings. See XIII.

PATENTS.

Peat; Treatment of —. M. C. Olsson. E.P. 147,286, 16.4.19.

RAW peat is partly dehydrated at ordinary temperature, and is then subjected to violent ebullition of the contained water by means of rapid lowering of pressure whilst the peat is being heated. The water can then be extracted by pressing or by other means.—A. E. D.

Furnace retorts. C. H. Smith. E.P. 123,739, 17.2.19. *Conv.*, 18.2.18.

A HORIZONTAL retort for carbonising coal is heated by longitudinal flues in the walls which are built up of hollow tiles. The coal is moved continuously through the retort by two conveyors mounted on parallel horizontal shafts and carrying blades which overlap. The retort is made inverted heart shape in cross section to accommodate the conveyors. The retort walls are constructed of carborundum or a material containing it, and are surrounded in succession by a layer of fire-brick, a layer of a diatomaceous earth brick known as "sigur" brick, and finally by a metal casing. A layer of asbestos is placed between the bricks and the metal casing in the upper part to allow for expansion. The conveyor shafts are journaled in removable covers which are sufficiently large to allow the removal of the conveyors. The conveyor shafts are hollow and are made in sections secured by spigot and socket joints which are locked by the conveyor blades passing through both parts.—W. F. F.

Coal gas; Manufacture of — and recovery of tar. H. Geyer. G.P. 319,523, 6.5.19.

NITROGEN-FREE heating gases, obtained by introducing a mixture of oxygen and steam into red-hot coke, are mixed with cold tar-free coal gas in a vessel directly connected with the rotary distillation retort. By this means the heating gases are cooled to the necessary temperature for the distillation retort, without the necessity of adding so much steam as to cause deterioration of the coal gas produced in the retort.—C. A. M.

Coal; Process for distillation and gasification of —. T. Limberg. G.P. 322,646, 28.10.17. *Addn.* to 303,954 (J., 1920, 441 A).

THE process described in the original patent may be carried out without the use of reduced pressure and with a single receiver for the distillation products. The devices for heating and for drawing off the products pass through the wall of the retort and are provided with openings for cleaning purposes. The removal of the tar vapours without decomposition is rapidly effected by the steam which is present.—W. J. W.

Hydrocarbon gas; Method of making —. D. and R. F. Macdonald. E.P. 146,560, 8.3.19.

COMBUSTIBLE gas (consisting largely of acetylene) is produced by creating an arc between carbon electrodes immersed in water.—A. E. D.

Gases; Apparatus for purifying —. H. F. Smith, Assr. to The Smith Gas Engineering Co. U.S.P. 1,348,966, 10.8.20. *Appl.*, 5.7.18.

A HOUSING is provided with a hollow rotating pad, so placed that the gas inlet and outlet openings of the housing are adapted to communicate, one

with the space inside and the other with the space outside the pad.—A. G.

Coal-gas; Process and apparatus for recovering the ammonia content of —. B. Torrey, jun., Assr. to Semet-Solway Co. U.S.P. 1,319,393, 10.8.20. Appl., 3.5.18.

THE water in the hydraulic main is heated by the sensible heat of the crude gas which is subsequently washed and cooled to remove tar and fixed ammonia. The gas is then reheated by washing it with the heated water from the main and is passed through a bath of sulphuric acid to recover the ammonia as sulphate.—A. G.

Motor fuel for aeroplanes and other motors. U.S. Industrial Alcohol Co., Assces. to F. W. T. Schreiber. E.P. 128,915, 16.6.19. Conv., 25.6.18.

AN alcohol, an aromatic hydrocarbon, and at least 4% of an ether are blended, e.g., 30 pts. by vol. of benzol, 30 of gasoline or naphtha, and 5 of ether. —A. E. D.

Hydrocarbons; Treatment of —. G. F. Forwood and J. G. Taplay. E.P. 145,198, 16.4. and 7.7.19.

PETROLEUM or coal tar distillates are passed together with steam over carbon heated to a specified temperature, e.g. for kerosene to 600°—630° C. and for heavy fuel oil to 850°—950° C., in order to obtain lower-boiling products.—A. E. D.

Petroleum oils; Purification of —. J. J. Hood. E.P. 145,818, 24.2.19.

THE vaporised oil is brought in contact with heated alumina, bauxite, or magnesite, so that the sulphur compounds present in the oil are dissociated and hydrogen sulphide is formed therefrom. The hydrogen sulphide is subsequently absorbed. —A. E. D.

[Mineral] oil; Refining — by distillation. C. B. Forward. E.P. 145,894, 5.4.19.

PREHEATED oil is conveyed through a series of sets of heaters, supplied with superheated steam, which is used repeatedly in the successive heaters after periodic reheating. The steam discharged from the final heater of each set is used for atomising the heated oil in a separator, the volatile products from which are condensed.—A. E. D.

Liquid fuel from petroleum; Manufacture of —. H. Wade. From Standard Oil Co. E.P. 147,353, 21.7.19.

CRUDE petroleum is distilled by direct fire and superheated steam (or inert gas) until high melting pitch is left (e.g., of m.p. 192° F.), and the pitch is then fluxed with low viscosity oil.—A. E. D.

Petroleum hydrocarbons; Process for treating —. T. T. Gray. U.S.P. 1,340,889, 25.5.20. Appl., 18.5.14.

VAPORISED oil is refined by passing it through heated fuller's earth.—A. E. D.

"Gas-oil"; Making —. C. Ellis, Assr. to Ellis-Foster Co. U.S.P. 1,311,975, 1.6.20. Appl., 29.5.13.

GASOLINE-LIKE material is made by mixing petroleum vapour and gaseous petroleum decomposition products and subjecting the mixture to several atmospheres pressure and a decomposition temperature. The yields of heavier and lighter products are thereby increased.—A. E. D.

Petroleum oils; Distillation of —. R. E. Humphreys and W. M. Burton, Assrs. to Standard Oil Co. U.S.P. 1,343,674, 15.6.20. Appl., 28.11.16.

OIL is distilled under 75 lb. pressure, the vapours being cooled under the same pressure, in such a way

that all insufficiently cracked material is returned to the still.—A. E. D.

Hydrocarbons; Process of treating —. L. W. McOmber, Assr. to C. B. Belknap. U.S.P. 1,345,452, 6.7.20. Appl., 24.2.16.

OIL is heated to 60 lb. pressure and then subjected to condensing action at a still higher pressure. —A. E. D.

Light hydrocarbons; Apparatus for producing —. J. A. Yunc. U.S.P. 1,345,656, 6.7.20. Appl., 15.10.18.

IS the cracking of crude oil or residues electric heating is made use of both for the pressure still and the vapour space.—A. E. D.

Petroleum; Process of distilling —. P. M. Bid-dison and H. T. Boyd. U.S.P. 1,345,740, 6.7.20. Appl., 15.2.18.

HIGH-BOILING oil is circulated to and from a bulk supply, and heat and pressure are supplied. The vapours are passed into an auxiliary condenser which returns uncracked material. The uncondensed vapours are cooled and the gas stripped. The heating is provided by a current of highly-heated gas.—A. E. D.

Hydrocarbon oils; Apparatus and process for the conversion [cracking] of —. V. L. Emerson. U.S.P. (a) 1,346,797, and (b) 1,346,798, 13.7.20. Appl., 6.5.20.

(A) GASEOUS products of distillation from a pressure still are submitted to impact, and the products are returned to the oil in the still. (b) Kinetic energy is imparted to oil which is "subjected to impact," as are the resulting products, which ultimately are intimately mixed with fresh oil and the gaseous products withdrawn.—A. E. D.

Petroleum; Apparatus for cracking —. Method of treating hydrocarbons. H. W. Jones, F. E. Jones, Administrator, Assr. to Kansas City Gasoline Co. U.S.P. (a) 1,347,543 and (b) 1,347,544, 27.7.20. Appl., 13.2.18.

(A) THE plant consists of a number of small, separately fired cracking stills, compressors, tanks, and condensing system. (b) The oil is compressed and forced into separately heated cracking zones. The cracked products are discharged separately, collected together, and condensed.—A. E. D.

[Mineral] oil-refining apparatus. F. E. Wellman, Assr. to Kansas City Gasoline Co. U.S.P. 1,347,567, 27.7.20. Appl., 1.8.16.

OIL under pressure is caused to expand into a cracking coil of larger bore, which is connected with a condenser having a control valve.—A. E. D.

Cracking hydrocarbons; Method of —. F. E. Wellman, Assr. to Kansas City Gasoline Co. U.S.P. 1,347,568, 27.7.20. Appl., 12.2.19.

A HYDROCARBON oil is heated at a high temperature and pressure in a still; the oil occupies 10% of the still volume, the remainder being filled with gas under pressure, which is retained in the still when the treated oil is removed.—A. E. D.

[Mineral] oil; Method of refining —. F. E. Wellman, Assr. to Kansas City Gasoline Co. U.S.P. 1,347,664, 27.7.20. Appl., 30.3.18.

INTERMITTENT and equal charges of oil are heated under pressure, and the vapours are severally condensed under pressure.—A. E. D.

Cracking hydrocarbons; Apparatus for and art of —. J. W. Coast, jun., Assr. to The Process Co. U.S.P. (A to E) 1,348,264-8, 3.8.20. Appl. (A,B) 22.3.17, (C) 27.3.17, (D) 9.4.17, and (E) 16.4.17.

(A) THE usual pressure still has one end protected against direct heat. Reflux condensed liquid is conducted towards the protected end. (B) The two ends of the pressure still are protected against direct heat. The condensed product is conducted by a branched pipe through the still contents and deposited on the cool end portions of the still. (C) A series of pressure stills at different levels communicate with one another so that pressure is equalised and oil flows from one to the other through a coke trap. (D) Refluxing of uncracked oil is effected by maintaining the top of the still in a relatively cool condition and deflecting vapour to the end and along the cool upper surface. (E) Refluxing of uncracked oil is effected by a baffling device located near the top of the still so as to provide a narrow passage-way. The baffle is an inverted trough with its sides dipping into the heated oil.—A. E. D.

[Hydrocarbon] oils; Process and apparatus for continuously cracking —. W. B. Price and E. Dietz. U.S.P. 1,349,294, 10.8.20. Appl., 6.7.16.

A CONTINUOUS stream of a heavy hydrocarbon oil is passed through an apparatus in which successive portions of the oil are subjected simultaneously to progressively higher temperatures and pressures. The light hydrocarbons are removed as they are formed.—L. A. C.

Paraffin wax; Separation of — from the neutral oil and the oxygen-containing constituent of producer or low-temperature tar. Allgem. Ges. für Chem. Ind. m.b.H. G.P. 319,656, 19.3.18.

PARAFFIN wax is separated from the heavy oil by fractional precipitation with pyridine or pyridine bases and water, especially in the cold. The remainder of the paraffin wax may then be obtained by treating the liquid with more pyridine or by diluting it with water, and then by filtration and further dilution the neutral oil (i.e., the portion insoluble in alkali) is obtained.—C. A. M.

Shale and other bituminous minerals; Production of oil from —. Zeller und Gmelin. G.P. 321,870, 23.8.18.

IN the distillation of shale in externally heated rotary retorts, with simultaneous introduction of steam or other gases free from oxygen, the strongly superheated steam or other gases are introduced at a temperature about 50° C. higher than that of the material in the retort.—J. H. L.

Distillation-gases of coal; Purification of —. E. Ciselet and C. Deguide. U.S.P. 1,349,750, 17.8.20. Appl. 6.7.15.

SEE G.P. 297,101 of 1915; J., 1917, 637.

Lubricating oils; Process of manufacturing —. H. Klever, L. C. Lautenschläger, and O. H. Göhring, Assrs. to The Chemical Foundation, Inc. U.S.P. 1,350,814, 24.8.20. Appl., 14.2.16.

SEE G.P. 301,774 of 1915; J., 1920, 327 A.

Benzol recovery. G.P. 298,823. See III.

Hydrogen sulphide from gases. G.P. 302,555 and 303,292. See VII.

Lampblack and hydrogen. G.P. 312,546. See XIII.

Aldehydes and ketones. G.P. 321,567. See XX.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Recovery of alcohol etc. Mariller. See XVIII.

PATENTS.

Coal and other materials capable of being distilled, vaporised, or decomposed by heat; Treatment of —. Dressler Tunnel Ovens, Ltd., and C. Dressler. E.P. 149,086, 14.5.19.

COAL, peat, or the like is fractionally distilled in a horizontal oven, which is heated to varying temperatures along its length by combustion flues at the sides. The coal is passed through the oven in trucks. Transverse walls project downwards from the roof to a height just sufficient to allow clearance for the trucks, thus forming a series of pockets along the roof. The fractional distillates are withdrawn from these pockets.—W. F. F.

Distilling apparatus; Rotary dry —. F. G. Ten Broeck and M. T. Weston, Assrs. to West Virginia Pulp and Paper Co. U.S.P. 1,350,627, 24.8.20. Appl., 6.8.18.

MATERIAL is fed from a hopper into one end of a horizontal conduit containing a helical conveyor, and delivered from the other end into one end of a horizontal rotating retort. The conveyor conduit is mounted in a part of the head of the retort, which can be rotated relatively to the remainder of the retort, and which also contains the vapour-discharge passage. The outlet of the hopper is provided with a controlling gate, which is interconnected with the controlling switch of the electric motor driving the conveyor, so that the speed of the conveyor is automatically reduced as the outlet of the hopper is restricted.—W. F. F.

Incandescence lamp bulbs or the like; Methods for forming conducting seals for —. T. Yanai, Assr. to Nihon Denki Kogyo Kabushiki Kaisha. U.S.P. 1,350,907, 24.8.20. Appl., 30.9.16.

SEE E.P. 103,648 of 1917; J., 1918, 84 A.

III.—TAR AND TAR PRODUCTS.

Phenol; Determination of — in the presence of certain other phenols. R. M. Chapin. J. Ind. Eng. Chem., 1920, 12, 771-775.

A COLORIMETRIC method is described depending on the red coloration yielded by Millon's reagent with phenol but not with other phenols. The reagent is prepared by dissolving 2 c.c. of mercury in 20 c.c. of concentrated nitric acid, diluting the solution to 35 c.c., adding a few drops of nitric acid if basic salt separates out, and then adding 10% sodium hydroxide solution until the precipitate first formed no longer re-dissolves but disperses to a permanent turbidity; a mixture of 0.2 c.c. of concentrated nitric acid and 4.8 c.c. of water is then added and the solution mixed. Four c.c. of a solution containing 0.1% of total phenols is measured into each of two test-tubes and to one is added about one-half of the amount of phenol believed to be already present; the two solutions are diluted to 6 c.c., treated with 5 c.c. of Millon's reagent and heated at 100° C. for 30 mins. In this time the colour due to the phenol is developed fully whilst the red colour at first produced by other phenols is discharged or changed to yellow. The two tubes are then placed in cold water for 10 mins., their contents acidified with a mixture of 0.2 c.c. of concentrated nitric acid and 4.8 c.c. of water, diluted to 25 c.c., filtered after 10 mins., and the colorations of the filtrates compared in a colorimeter. The standard Duboscq colorimeter was used. The quantity of phenol, P, is calculated by the formula

$P = 1/(1 - I)[pR/(S - R) - IT]$ where p is the amount of added phenol, S the reading of the test solution, R that of the stronger solution, I the relative intensity of the colour yielded by other phenols as compared with that of phenol (I has a mean value of 0.007), and T the total quantity of phenols. The red colour given by phenol may be changed to yellow by treatment, under suitable conditions, with formaldehyde without any marked effect on the yellow colour present originally, and this modification of the method is recommended for the determination of small proportions of phenol. Results of experiments with a considerable number of phenols are given; only two substances, salicylic acid and β -naphthol, invalidate the test, the former giving a deep red coloration and the latter a dark brown.—W. P. S.

Bituminous acid-proof coatings. See XIII.

PATENTS.

Benzol hydrocarbons of coke-oven gas; Recovery of — from the washing oil. A. Hartmann. G.P. 298,823, 14.11.13.

BENZENE hydrocarbons are distilled from the washing oil by means of indirect steam at the ordinary pressure with the aid of a vacuum. The presence of the higher benzene hydrocarbons, which are simultaneously condensed, causes a reduction of the vapour pressure below that of the constituent of lowest b.p.—C. A. M.

Hydrocarbons; Production of liquid — from naphthalene. F. Fischer. G.P. 299,131, 22.12.15.

A MIXTURE of naphthalene and aluminium chloride is heated under pressure to above the normal boiling point of naphthalene; e.g., by employing 4% of aluminium chloride and a temperature of 330° C. one-third of the naphthalene is converted to liquid hydrocarbons, mainly di- and tetrahydronaphthalene, and the remainder forms a mixture of pitch and a coal-like product.—L. A. C.

Tar oils; Agents for dissolving, separating and extracting the solid constituents of —. Tetralin G. m. b. H. G.P. 301,651, 23.4.16.

HYDRONAPHTHALENES are used alone or in combination with other substances. In particular, tetrahydronaphthalene and higher hydrogenised products dissolve considerable quantities of naphthalene at 30° C. and unlimited quantities at higher temperatures.—J. H. L.

SEE also pp. (A) 651, *Hydrocarbons* (E.P. 145,198); 652, *Paraffin wax* (G.P. 319,656); 665, *Paint* (G.P. 318,699), *Solvent* (G.P. 320,807), *Varnish* (G.P. 320,808); 675, *Aldehydes and ketones* (G.P. 321,567); 676, *Nitro-compounds* (G.P. 299,015).

IV.—COLOURING MATTERS AND DYES.

PATENTS.

Dye; Alkali proof — and method of producing the same. L. C. Cates, Assr. to Sunbeam Chemical Co. U.S.P. 1,349,265, 10.8.20. Appl., 27.7.18.

AN excess of a saponifiable oil is added to a boiling solution of a non-alkali-proof dye in $N/4$ sodium hydroxide, and the reaction is allowed to continue until the alkali is exhausted. Sufficient alkali is then added to saponify the excess oil, and the product, after it has hardened, is powdered.

—L. A. C.

Solvent for dyestuffs. G.P. 320,807. See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Paper makers' quicklime and hydrated lime; Specifications for —. U.S. Bureau of Standards, Circ. 96, 15.6.20.

THE specifications given have been approved by the National Lime Association and the Technical Association of the Pulp and Paper Industry (U.S.A.). The percentage compositions specified are:—Quicklime: CaO, 95 (min.); MgO, 2 (max.); insol. matter, including SiO_2 , Fe_2O_3 , and Al_2O_3 , 2 (max.); CO_2 , 2 (max.). Hydrated lime: CaO, 72 (min.); MgO, 1.6 (max.); insol. matter, including SiO_2 , Fe_2O_3 , and Al_2O_3 , 1.6 (max.); CO_2 , 1.6 (max.). For analytical methods reference is made to U.S. Geological Survey, Bull. 700 (Hillebrand, Analysis of silicate and carbonate rocks).

PATENTS.

Fibrous vegetable material [Spanish moss]; Process of treating — and the resulting product. G. S. McDaniel, Assr. to E. H. Kimbell. U.S.P. 1,350,031, 17.8.20. Appl., 24.7.19.

GREEN, partly cured, or cured Spanish moss is treated with an alkaline solution of definite strength, whereby the outer coating shrinks upon the inner fibres.—A. J. H.

Fibres; Process for treating —. J. H. Pickup, Assr. to G. A. Wilson. U.S.P. 1,350,621, 24.8.20. Appl., 19.3.19.

VEGETABLE fibre is prepared for spinning by allowing it to ferment in a hot bath for a considerable time, boiling it under pressure in lime water, and then subjecting it alternately to acid and alkaline baths.—W. F. F.

Pyroxylin sheet of low inflammability. J. G. Jarvis. U.S.P. 1,349,156, 10.8.20. Appl., 13.3.19.

THE material is composed of cellulose acetate, acetone, and cinchonine and its salts.—W. F. F.

Parchment paper; Manufacture of —. P. Hoering. G.P. 303,266, 1.9.16.

THE leaves, or stalks, or both, of the reed mace are subjected to the action of caustic alkalis under pressure, and the product after washing is employed as paper pulp without any special beating and without sizing. The material consisting of bast fibres and parenchyma, furnishes a transparent, grease-proof paper of a brownish-yellow colour.—G. F. M.

Cardboard [paper and paper fabrics]; Process for rendering — grease- and waterproof. L. Buchbinder. G.P. 316,527, 19.3.19. Addition to 306,028.

CARDBOARD which has been coated with a glue precipitate containing a filling material, in accordance with the chief patent (J., 1918, 575 A), and dried, is further coated with a mixture consisting of a saturated solution of tannin in spirit and a varnish containing a drier, to which mixture zinc white and formaldehyde have been added.—J. H. L.

Paper, pulps, paper yarns and fabrics; Sizing and waterproofing of —. E. Fues and E. Braumüller. G.P. 318,923, 3.1.17.

THE paper material is treated with colloids such as dialysed solutions of hydroxides of trivalent metals, preferably iron or chromium, with or without the addition of solutions of other, preferably organic, colloids.—C. A. M.

Paper, fabrics and the like; Sizing, waterproofing, dressing, etc. —. W. Schmidt and E. Heuser. G.P. 321,232, 15.3.18. Addn. to 296,124.

THE paper pulp or fabric is treated with a solution

of tar soap prepared by saponification of wood-, lignite-, or coal-tar, previously extracted with water. Injurious acid substances may be removed from the tar by distillation, and a soap prepared by incomplete saponification of the tar may be used.—J. H. L.

Paper; Removal of ink from writing —. H. Gethe. G.P. 321,545, 26.7.19.

THE paper is passed through four consecutive baths, of which the first and third contain oxalic acid, the second potassium permanganate, and the fourth aluminium sulphate; it is afterwards passed between hot rollers.—J. H. L.

Straw; Utilisation of the waste lyes from the alkaline digestion of —. S. Herzberg. G.P. 319,068, 13.11.17.

ALKALINE lyes from the treatment of straw are electrolysed to obtain the caustic soda in a concentration suitable for further use. The degree of evolution of oxygen or of oxidation of organic substances at the anode varies with the strength of the current.—C. A. M.

Straw; Utilisation of waste liquors obtained in the digestion of —. S. Herzberg. G.P. 321,453, 28.3.19. Addn. to 319,068 (preceding).

THE liquor, or at least the portion to be used as anodic bath, is treated in the cold with mineral acid or bisulphate and heated to 70° C.; it is then filtered from precipitated organic substances and electrolysed. The precipitated lignic acid can be utilised, and if common salt solution is used as anodic electrolyte the chlorine produced may be employed in the dissolution of chopped straw previously treated with milk of lime.—J. H. L.

Waste liquors; Purification of alkaline — contaminated and coloured with organic impurities. F. Küttner, E. Profeld, and E. Sidler. G.P. 322,461, 8.7.19.

WASTE liquors from the mercerisation process and viscose manufacture are concentrated and simultaneously oxidised in open vessels, whereby the hemicellulose is converted into insoluble oxycellulose. The oxidation may be carried out by injecting air into the liquors or by the addition of alkali nitrate.—G. F. M.

Paper making. Great Northern Paper Co., Assees. of C. E. Pope. E.P. 136,839, 18.12.19. Conv., 14.3.18.

SEE U.S.P. 1,279,756 of 1918; J., 1918, 764 A.

Alkyl ethers of starches and similar carbohydrates and method of producing the same. L. Lilienfeld. Assr. to The Chemical Foundation, Inc. U.S.P. 1,350,820, 24.8.20. Appl., 16.2.14.

SEE F.P. 468,162 of 1914; J., 1914, 958.

Paper-making machines. A. Wood and G. Russell. E.P. 149,359, 21.12.17.

Paper, cardboard, and the like; Machines for manufacture of —. L. Thiry. E.P. 149,416, 10.5.19.

Evaporation of sulphite-cellulose liquors. G.P. 322,462. See I.

Fat from sulphite-cellulose lyes. G.P. 305,091. See XII.

Paint. G.P. 320,011. See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Vegetable fibres; Process for treating —. Gillet et Fils, Assees. of C. Schwartz. E.P. 136,569, 12.12.19. Conv., 13.12.18. (Cf. E.P. 136,568 and 144,204; J., 1920, 513 A, 542 A.)

WOOL-LIKE characteristics can be imparted to vegetable fibres by simple immersion in nitric acid (65–75%) for not more than half an hour, followed by washing with water. The fibres may be treated in any stage of their preparation and the temperature should be less than 20° C. If nitric acid stronger than 75% be used, the treatment must be carried out more quickly. Print effects may be produced directly or by means of reserves. The fibres so treated increase in weight.—A. J. H.

[Azo] dyestuffs containing the sulphonamide group; Application of — to dyeing and printing. J. W. McMyen. E.P. 149,428, 13.5.19.

Azo dyes which contain a sulphonamide or substituted sulphonamide group are soluble only in strong alkalis from which they are easily precipitated by weak acids, alum, ammonium chloride, bicarbonates, and carbon dioxide. Cloth is dyed by impregnation with a solution of the dye in caustic soda, squeezing, drying, treatment in a fixing bath containing one of the above precipitants, rinsing, and drying. Cloth is printed with a paste containing the dye dissolved in caustic soda and thickened with starch, then dried, and fixed in an alum bath. The addition of aluminium salts to the printing paste increases the brightness and fastness. Suitable scarlet, red and light red dyes are formed respectively from β -naphthol and diazotised 2-aminotoluene-4-sulphonamide; 2-aminotoluene-4-sulphonanilide; *o*-, *m*-, *p*-aminophenylsulphonamides. They can be used for discharges or resists with Aniline Black. 2-Aminotoluene-4-sulphonamide coupled with diazotised *m*-phenylenediamine gives an orange dye.—A. J. H.

Dyeing-machine. W. A. Ainsworth. U.S.P. 1,318,974, 10.8.20. Appl., 19.1.20.

A ROTATING roller covered with an absorbent material is fitted in a reservoir for colouring matter, and projects above the top of the reservoir. Thread, yarn, or the like passes over the reservoir and is intermittently brought in contact with the absorbent material on the roller.—L. A. C.

Dyeing-machine. E. Schnurrenberger. U.S.P. 1,350,740, 24.8.20. Appl., 9.1.20.

A MACHINE for the treatment of skeins with liquids consists of a liquor container, and means for suspending a skein in, and causing it to trail through the liquor, including a skein-trailing member movable in one plane.—A. J. H.

Dyeing [Sulphur Black]. C. S. Althouse. U.S.P. 1,349,867, 17.8.20. Appl., 24.6.20.

IN dyeing goods with Sulphur Black, magnesium sulphate is used in the softening bath.—A. J. H.

Leuco-compounds of vat-dyes; Preparation of — suitable for dyeing wool. L. Cassella und Co. G.m.b.H. G.P. 321,119, 16.11.15.

THE leuco-compound is prepared in the presence of a higher alcohol which can be salted out of aqueous solution, and it is thereby separated simultaneously with the alcohol. An example is given of the reduction of Thioindigo-Red B with sodium hydroxide and hydrosulphite, with the addition of isobutyl alcohol.—G. F. M.

Aniline Black; Production of — upon vegetable textile fibres. E. A. Fourneauux. U.S.P. 1,350,600, 21.8.20. Appl. 18.1.18.

SEE E.P. 117,987 of 1917; J., 1918, 576 A.

Waste liquors. G.P. 322,461. See V.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric acid manufacture; Use of sodium nitrate solution in the lead chambers in —. H. Lemaître. Monit. Scient., 1920, 10, 145–157.

THE continuous introduction of sodium nitrate in solution through the Glover tower produces saturation of the acid with sodium bisulphate and consequent choking of pipes. Dilution of the Glover acid with chamber acid to prevent saturation is rendered impracticable by the limited concentration capacity of the Glover. Alternate introduction of nitric acid and sodium nitrate may be suitable provided that the supply of nitrate is arrested before saturation is reached, this stage being determined by calculation or by control analysis of the bisulphate content of the acid. To avoid saturation entirely, it is preferable to introduce the nitre as a mixture of nitric acid (36° B., sp. gr. 1.33) and sodium nitrate solution. Loss of nitric acid resulting from imperfect working of the Glover seldom exceeds 5–10%; in the Gay-Lussac tower it amounts to 50–75% of the total, but these losses may be considerably reduced, and the economy achieved may equal or exceed that attained by use of sodium nitrate. The reactions which take place in the Gay-Lussac towers are indicated by the author, and the control of the chamber process by the use of graphs is advised.—W. J. W.

Nitric acid; Production of concentrated — from nitrous vapours. M. Kaltenbach. Chim. et Ind., 1920, 4, 576–590.

AFTER a preliminary survey of the reactions involved and the conditions necessary for the conversion into nitric acid of nitrogen oxides derived from the ammonia oxidation process, a plant is described for the production of 10 tons of nitric acid per 24 hrs. The hot gases are first passed through a pre-concentration tower fed with weak nitric acid (45% HNO₃) in which the gases are cooled and at the same time a concentration of the weak acid is effected. The removal of the water vapour from the gases takes place in externally cooled towers, which the gases leave at 30° C., then passing into a tower in which oxidation and polymerisation of nitrogen peroxide are brought about. After traversing a series of absorbing and regenerating towers, an outflowing acid of 60% HNO₃ is obtained. Concentration of this weak acid is carried out in denitrating towers with the aid of sulphuric acid, an important economy of the latter being effected by a preliminary heating of the nitric acid and its introduction in the form of vapour, by which means the necessity for steam injection is avoided.—W. J. W.

Ammonia; Synthesis of — at very high pressures. G. Claude. Chim. et Ind., 1920, 4, 5–16.

THE author recapitulates the advantages claimed for the employment of pressures up to 1000 atm.: diminution in size of contact chamber and reduction of number of times of passage of the reacting gases, increased yields of ammonia per 0.5 g. of catalyst, easy condensation of the ammonia and consequent elimination of absorption by water, and realisation of the auto-reaction even in small plants by the enormous heat developed. (Cf. J., 1919, 885 A; 1920, 61 A, 187 A.) Tightness of the joints

presents no greater difficulty at 1000 than at 100 atm. and may be more easily secured on account of their smaller size. Cupped leather packings have been employed successfully in the compressors. With high pressures loss of heat is reduced and the cost of plant erection and maintenance is smaller. Coke-oven gas appears to be a promising source of hydrogen. A plant dealing with 300 tons of coke per day yields 100,000 cub. m. of gas containing 45% hydrogen, of which 30,000 cub. m. can be extracted and utilised to fix 10 tons of nitrogen per 24 hrs.—W. J. W.

Phosphotungstic and phosphomolybdic acids. H. Wu. J. Biol. Chem., 1920, 43, 189–220.

THESE compounds belong to two distinct series, represented by P₂O₅.18XO₃ and P₂O₅.21XO₃. The properties and preparation of these acids and other members of these complex groups are described fully. (Cf. J.C.S., Oct.)—J. C. D.

Potash alum; Analysis of —. A. Harvey. J. Soc. Leather Trades' Chem., 1920, 4, 219–220.

SAMPLES of pure potash alum mixed with known amounts of iron sulphide and analysed by the method proposed by Codwise (J., 1920, 416 A) did not give theoretical results. The iron impurities are included in the titration.—D. W.

Acid clay [fuller's earth]; Acidity of Japanese —. K. Kobayashi. J. Chem. Ind. Tokyo, 1920, 23, 543–549.

JAPANESE acid clay (fuller's earth) is regarded as being a mixture of colloidal hydrous aluminium silicates, Al₂O₃.2SiO₂.xH₂O, and an amorphous compound of orthosilicic acid, SiO₂.xH₂O, and may be expressed by the formula: Al₂O₃.6SiO₂.xH₂O, where x is more than 6. The clay itself is not a true acid substance, and the acidity is to be ascribed to its adsorptive property. On adding the clay to a solution of a basic dye (R.Cl) hydroxyl ions are adsorbed from the solution and hydrogen ions are liberated and unite with the chlorine ions of the basic dye to form hydrochloric acid. Reaction between an acid clay and neutral potassium chloride solution similarly produces hydrochloric acid, which dissolves alumina from the clay. The aluminium chloride thus formed will be decomposed by hydrolysis into aluminium hydroxide and hydrochloric acid.—K. K.

Potassium plumbite; Oxidation of — to potassium plumbate by alternating current. F. Jirza. Z. physik. Chem., 1920, 94, 1–5.

POTASSIUM plumbite is the only product of the electrolysis of N/1 potassium hydroxide solution between lead electrodes by an alternating current of 49 periods. The solution of plumbite is oxidised to plumbate by a similar current if electrodes of copper, silver, gold, cadmium, magnesium, mercury, nickel, iron, platinum, or palladium are used, but there is no oxidation if the electrodes are of lead, bismuth, cobalt, thallium, or carbon. A number of other factors also play a part in the plumbate formation.—J. F. S.

Arsenic; Electrometric titration of —. C. S. Robinson and O. B. Winter. J. Ind. Eng. Chem., 1920, 12, 775–778.

IN certain coloured solutions the electrometric titration of arsenious acid is to be preferred to the ordinary method. The titration is made with iodine solution in the presence of excess of sodium bicarbonate, a mechanical stirrer is used, and the end-point is indicated by the change in direction of the swing of a beam of light from a reflecting galvanometer. Arsenic acid is titrated at 90° C. in 50% sulphuric acid solution with sodium iodide solution.—W. P. S.

Copper hydroxide; Formation and behaviour of —. V. Kohlschütter and J. L. Tüscher. Z. anorg. Chem., 1920, 111, 193–236.

THE authors describe experiments on the formation of copper hydroxide by precipitation, the pseudomorphic transformation of solid copper salts into hydroxide by the action of sodium hydroxide solution, the processes occurring at copper anodes in sodium hydroxide solution, and the formation of colloidal copper hydroxide or oxide by "discharge electrolysis" (J., 1919, 911A). It is concluded that the dehydration of copper hydroxide is due to an internal neutralisation and is to be represented by the equations $\text{Cu}(\text{OH})_2 \rightleftharpoons \text{Cu}'' + 2\text{OH}'$, $\text{CuH}_2\text{O}_2 \rightleftharpoons \text{CuO}''_2 + 2\text{H}'$, $2\text{H}' + 2\text{OH}' \rightleftharpoons 2\text{H}_2\text{O}$ and $\text{CuO}''_2 + \text{Cu}'' = 2\text{CuO}$. It is, however, not simple $\text{Cu}(\text{OH})_2$ molecules that are involved in the reaction, but molecular complexes of colloidal dimensions, the degree of dispersity, and hence the reactivity of which vary with the conditions of formation.—J. F. S.

Hydrogen peroxide; Rate of decomposition of — in the presence of iron ions. J. von Bertalan. Z. physik. Chem., 1920, 95, 328–348.

THE decomposition of hydrogen peroxide in the presence of ferrous sulphate or ferric sulphate at 40° C. is in accordance with a unimolecular catalytic equation. The state of oxidation of the iron makes no difference to the reaction, which depends on the total quantity of iron present.—J. F. S.

Colloidal sulphur. P. Bary. Comptes rend., 1920, 171, 433–435.

USING Odén's results (Nova Acta Upsala, 1913, 3 [4]) on the viscosity of suspensions of sulphur at different concentrations and temperatures, the author has calculated the mean coefficients of swelling of the granules, the results indicating that such suspensions are hydrophilic colloids. When the swelling, due to the absorption of water, becomes small the granules lose their transparency, and the liquid becomes milky and loses its stability. This absorption is favoured by the presence in solution of small amounts of salts of univalent metals and is checked by salts of divalent metals.—W. G.

Thermodynamics; Applications of chemical —. G. Urbain. Chim. et Ind., 1920, 4, 31–42.

THE author discusses the rôle played by thermodynamics in various branches of chemical industry. The application of the phase rule to saturated solutions is considered, especially with respect to the extraction of salts from carnallite. Carnot's principle of reversibility is illustrated in the case of the manufacture of hydrogen by the action of iron on water, and the contact process of sulphuric acid manufacture is also discussed from the thermodynamic standpoint. The laws of mass action and of displacement of equilibrium are among others examined in their relation to industrial applications.—W. J. W.

Quicklime and hydrated lime. See V.

Bituminous acid-proof coatings. See XIII.

Cyanogen chloride. Reed. See XIXB.

Phosphoric acid. Kleinmann and Feigl. See XXIII.

PATENTS.

Sulphuric acid; Apparatus with rectifying column for the concentration of —. H. Pauling. G.P. 299,774, 25.8.15.

A VESSEL of cast iron or an alloy thereof is used. The acid to be concentrated is fed into the top of the column. It passes from the column to the heating vessel by way of a separate vessel with overflow device so that acid cannot pass back from the heater to the column; and is at this point strong

enough to act only slightly on the material of the heater.—C. I.

Retorts for decomposition of sodium or potassium nitrate. Farbenfabriken vorm. F. Bayer und Co. G.P. 301,702, 19.4.17.

By connecting a series of retorts by means of pipes to equalise the pressures and charging the retorts at regular intervals, uniformity of the process is ensured.—W. J. W.

Nitrous gases; Process for the absorption of —. J. L. la Cour, Assr. to Norsk Hydro-Elektrisk Kvaelfstøfaktieselskab. U.S.P. 1,348,227, 3.8.20. Appl., 22.10.18.

GASEOUS nitrogen compounds from an electric furnace are partially condensed, the residual undensified gases are reheated to a temperature suitable for absorption, and then absorbed by dry bases at an elevated temperature.—W. J. W.

Nitric acid and nitrogen compounds; Process of manufacturing — from atmospheric air. J. F. Sanders, Assr. to F. H. Churchill and O. P. Coshaw. U.S.P. 1,349,919, 17.8.20. Appl., 23.10.17.

A MIXTURE of nitrogen, oxygen, and hydrogen is passed over platinum heated to incandescence by an electric current.—W. J. W.

Ammonia synthesis; Preparation of a mixture of hydrogen and nitrogen for —. G. P. Gerfin and M. Mauran, Assrs. to The Nitrogen Corp. U.S.P. 1,349,756, 17.8.20. Appl., 15.11.18.

A MIXTURE of hydrogen and air is formed in which the hydrogen is in excess of the amount which would combine with the oxygen in the air. The mixture is passed through a conduit in which it is ignited, the temperature being kept sufficiently high to ensure complete elimination of oxygen, after which the reaction products are cooled, dried, compressed, and purified.—W. J. W.

Hydrogen; Material for use in operations with — under pressure and at high temperatures, specially for catalytic preparation of ammonia. Badische Anilin- u. Soda-Fabr. G.P. (A) 298,199, 14.5.13, and (B) 306,333, 18.6.16. Additions to 291,582 (see E.P. 29,260 of 1912 and 13,258 of 1913; J., 1913, 1010).

(A) STEEL alloys containing chromium, vanadium, tungsten, etc., remain suitable for use even if they contain relatively much carbon. Provided the alloying metals are present in sufficient quantity the strength is maintained even when the carbon is converted into gaseous compounds and liberated. A high proportion of nickel should be avoided. (B) If larger quantities of the alloying metals (chromium, tungsten, etc.) are present, steel containing a relatively high proportion of nickel can be used. A suitable alloy contains tungsten 5%, nickel 5%.—W. J. W.

Ammonia; Preparation of dry — from nitrolim by means of superheated steam. E. Wiedemann. G.P. 321,204, 28.5.14. Addn. to 311,959 (J., 1919, 630 A).

For the process described in the original patent, a furnace with superposed hearths or shelves provided with stirring gear is employed, nitrolim being introduced at the top, and superheated steam passed in at the lower end.—W. J. W.

Ammonium nitrate; Manufacture of —. F. A. Freeth and H. E. Cocksedge. E.P. 149,095, 20.5.19.

AMMONIUM sulphate and sodium nitrate are caused to react in suitable proportions, and the sodium sulphate precipitated is removed. Calcium sulphate is added to the solution to separate sodium sulphate as a double salt. After removal of this salt,

ammonium nitrate may be obtained by cooling the solution without further dilution, the mother liquor being used for a fresh cycle of operations. (Cf. E.P. 126,678; J., 1919, 499 A.)—W. J. W.

Hydrocyanic acid and cyanides; Manufacture of —. M. E. Mueller. U.S.P. 1,317,518, 27.7.20. Appl., 25.8.19.

FERROCYANIDES are treated with metallic copper in presence of an acid capable of reacting with the ferrocyanide, the liberated hydrocyanic acid being distilled off.—W. J. W.

Furnace [for fixing nitrogen]. C. P. Hidden. Assr. to Nitrogen Products Co. U.S.P. 1,318,175, 3.8.20. Appl., 27.1.19.

A VERTICAL cylindrical retort is provided with an inner concentric cylinder, and the nitrogen-absorbing material is fed downwards through the annular column thus formed. This column is heated externally by hot gases, and internally by burning fuel gas and air which are injected downwards into the lower end of the inner cylinder by central vertical supply pipes. Nitrogen passes upwards through the annular column of the material. The material delivered at the bottom is cooled externally and internally, and adhesion of the charge to any part of the retort is prevented. The cooling means include a relatively cool jet of nitrogen directed into contact with the lower end of the column.—W. F. F.

Aluminous materials; Conversion of powdered — into granular form. J. J. Hood. E.P. 149,453, 20.5.19.

POWDERED aluminium hydroxide, bauxite, china clay and the like are converted into granular form by moistening with nitric or hydrochloric acid or a solution of their aluminium salts, heating to form a cake, crushing, and decomposing the salt by heating to dull redness. The granular material obtained is suitable for refining mineral oils (E.P. 16,617 of 1908; J., 1909, 1081) or for decomposing carbon bisulphide in coal gas (cf. E.P. 143,641; J., 1920, 510 A).—C. I.

Zirconium oxide (zirconia); Process of separating and extracting — from ores and minerals. J. Gordon. U.S.P. 1,340,888, 25.5.20. Appl., 16.10.18.

MINERALS containing zirconium are fused with a mixture of a chloride and an oxide as a step in the process of producing zirconia.

Titanic oxide; Method of obtaining —. L. E. Barton. Assr. to The Titanium Alloy Manufacturing Co. U.S.P. 1,318,843, 10.8.20. Appl., 27.2.19.

In the preparation of titanic oxide concentrates by the acid treatment of titaniferous materials containing sulphides of iron, the latter are first converted into oxygenated compounds.—C. I.

Magnesium compounds; Purifying —. C. E. Dolbear. U.S.P. 1,348,933, 10.8.20. Appl., 3.9.19.

MAGNESIUM compounds are treated at an elevated temperature with a solution of an ammonium salt, the gas given off is passed into a comparatively cool solution of the magnesium salt formed in a previous operation, and the precipitated magnesium compound separated.—C. I.

Potassium and other alkali metal compounds; Process of obtaining —. E. H. Westling. U.S.P. 1,349,113, 10.8.20. Appl., 14.10.18.

ANHYDROUS compounds of an alkali metal are heated with the sulphate of a heavy metal to a temperature sufficient to decompose the latter.

—C. I.

Potassium salts and borax; Recovery of —. K. J. Jacobi. Assr. to Pacific Coast Borax Co. U.S.P. 1,319,131, 10.8.20. Appl., 25.8.19.

THE alkalinity of brine containing potassium salts and borax is reduced by addition of acid, and the brine concentrated and cooled.—C. I.

Alkaline deposits; Process of recovering constituents of value from natural —. C. Sundstrom. Assr. to The Solvay Process Co. U.S.P. 1,319,415, 10.8.20. Appl., 6.10.18.

SOLUTIONS containing potassium chloride, along with sodium chloride, sodium sulphate, sodium carbonate, and borax are cooled to precipitate the three last, and the cleared brine evaporated at first by solar heat, then by artificial heat, with removal at each stage of the precipitated salts. The final saturated potassium chloride solution is cooled and crystallised.—C. I.

Borax; Process of recovering — from solutions containing sodium borates. C. Sundstrom. Assr. to The Solvay Process Co. U.S.P. 1,319,416, 10.8.20. Appl., 21.9.19.

CARBON dioxide is generated in a solution containing sodium metaborate whereby sodium tetraborate is precipitated.—C. I.

Sodium bicarbonate; Process of obtaining — from alkaline waters. C. Sundstrom. Assr. to The Solvay Process Co. U.S.P. 1,319,117, 10.8.20. Appl., 3.1.20.

BRINE containing sodium carbonate is cooled below 0° C. and the precipitated sodium carbonate separated. The precipitated salts are warmed so as to melt the sodium carbonate, the unmelted portion separated and the clear liquor carbonated to precipitate sodium bicarbonate.—C. I.

Potassium chloride and borax; Method of separately obtaining — from certain waters. Method of separating borax from potassium chloride. N. Wrinkle and W. A. Kuhnert. U.S.P. (A) 1,350,089 and (B) 1,350,090, 17.8.20. Appl., 14.7. and 21.7.19.

THE borax is converted into more soluble compounds by addition of (A) sodium hydroxide or (B) an acid, and the potassium chloride is crystallised.—C. I.

Barium monoxide; Process of oxidising — to dioxide. H. Fleck. Assr. to The Peroxide Specialty Co. U.S.P. 1,319,417, 10.8.20. Appl., 5.11.17. Renewed 29.10.19.

BARIUM monoxide is subjected simultaneously to attrition and to the action of an oxidising gas at the requisite temperature.—C. I.

Aluminium nitride; Process for producing — from materials containing aluminic oxide. Process of producing aluminium from aluminic oxide. V. Gerber. U.S.P. (A) 1,350,149 and (B) 1,350,150, 17.8.20. Appl., 28.1.20.

(A) ALUMINIUM nitride is produced by melting aluminium oxide with a reducing agent in the presence of a nitrogenous gas in an electric furnace. (B) Alumina is melted in an electric furnace and treated with carbon and hydrogen for the production of metallic aluminium.—A. R. P.

Carbon bisulphide; Method of making —. H. H. Dow and C. J. Strosacker. Assrs. to The Dow Chemical Co. U.S.P. 1,350,558, 21.8.20. Appl., 15.7.18.

SULPHUR dioxide is brought into contact with heated coke after mixing it with sufficient oxygen to maintain the coke at the requisite temperature.

—C. I.

Hydrogen sulphide; Removal of — from gases. Badische Anilin and Soda Fabrik. G.P. (A) 302,555 and (B) 303,292, 2.7.16.

(A) THE gases are washed with aqueous solutions or suspensions of oxides of iron first in neutral medium, and then, for complete desulphurisation, in alkaline medium. The greater portion of the sulphur is removed by the first wash liquor, and this can very easily be revived by blowing air or oxygen through it, either simultaneously with the hydrogen sulphide gases or subsequently. (B) The gases are treated with alkaline wash liquors containing oxides of iron partly in solution and partly in suspension, a quantity of oxalic acid or cream of tartar, for example, being added, which is insufficient for the complete dissolution of the iron. The liquors are revived by air or oxygen as in the first patent.—G. F. M.

Lime; Process for burning — in a rotary furnace. E. Natho. G.P. 319,054, 6.7.17.

By means of a pipe with nozzles, water is introduced into the furnace cooler under pressure, and, in evaporating, serves to slake the lime uniformly and rapidly.—W. J. W.

Arsenic; Manufacture of —. C. Göpner. G.P. 320,086, 21.11.17.

ARSENIC acid is sublimed and passed over charcoal or coke heated to not less than 300° C., the sublimation being assisted by means of a gas heated above 185° C. The arsenic acid may be produced by roasting arsenic ores at a maximum temperature of 400°.—W. J. W.

Cryolite; Manufacture of —. J. L. C. Eckelt. G.P. 320,690, 12.7.17.

HYDROGEN fluoride is drawn from the vessel in which it is formed, by means of a water pump actuated by a solution of sodium aluminate. —W. J. W.

Soluble salts; Conversion of — into other salts, especially nitrates of potassium, sodium, and magnesium. H. Hampel. G.P. 321,039, 11.3.19.

THE acid radicle of the initial salt is precipitated in a slightly soluble form, and then converted into a soluble salt in combination with a less soluble one by the action of ammonia and an acid gas or anhydride. These salts in turn are treated with the desired acid for the end product, in its free state, by which means the initial precipitating salt for the process is recovered. The method is applicable to the production of nitrates of potassium, sodium, and magnesium, the sulphates being first treated with calcium nitrate, the precipitated calcium sulphate then converted into calcium carbonate and ammonium sulphate by means of ammonia and carbon dioxide, and the calcium carbonate finally converted into calcium nitrate by treatment with nitric acid.—W. J. W.

Salt solutions, especially solutions of potassium salts; Clarification of —. F. Krupp A.-G. Grusonwerk. G.P. 321,767, 19.10.17.

THE mud in salt solutions is mechanically removed in a continuous fashion and washed in another vessel, where its soluble portions are dissolved by fractional extraction and adherent liquor is separated.—W. J. W.

Radium; Production of compounds of —. H. O. Hedstrom. E.P. 149,552, 20.10.19. Addn. to 136,768 (J., 1920, 156A).

SEE U.S.P. 1,338,831 of 1920; J., 1920, 449A.

Potassium chloride; Manufacture of — from natural silicates, rocks, minerals, residues, or wastes containing potassium. E. A. Ashcroft. U.S.P. 1,350,091, 17.8.20. Appl., 13.11.17.

SEE E.P. 113,211 of 1917; J., 1918, 180A.

Copper sulphate solutions from ores; Process of producing —. J. S. Ross. U.S.P. 1,350,239, 17.8.20. Appl., 14.7.20

SEE E.P. 143,973 of 1919; J., 1920, 544A.

Silicon tetrachloride; Process of making —. R. W. Moore, Assr. to General Electric Co. U.S.P. 1,350,932, 24.8.20. Appl., 18.2.18.

SEE E.P. 141,908 of 1919; J., 1920, 448A.

Ammonia from coal-gas. U.S.P. 1,349,393. See IIA.

Acid-resisting material. G.P. 321,029. See VIII.

Lampblack and hydrogen. G.P. 312,546. See XIII.

Solvent for sulphur. G.P. 320,807. See XIII.

VIII.—GLASS ; CERAMICS.

Porosity [of ceramic materials]; Quick determination of —. L. Bertrand and C. Boulanger. La Ceramique, 1920, 23, 77. Keram. Rundsch., 1920, 28, 345—346.

THE material is immersed in carbon tetrachloride instead of in water. Owing to the lower surface tension of the former, a paving tile previously dried at 250° C. is saturated with the liquid in one-quarter to two hrs., compared with 24—48 hrs. required if water is used. For accurate work the body after immersion is weighed in a light metal vessel of zinc or brass in order to prevent losses due to rapid evaporation of the carbon tetrachloride. If p' be the difference between the dry weight of the test-piece and its weight when suspended in the immersion liquid, and p'' be the difference between the weight after immersion and the dry weight, then the porosity is $p''/(p'+p'')$.—H. S. H.

PATENTS.

Glass-drawing bait and method of using it. H. K. Hitchcock, Assr. to Pittsburgh Plate Glass Co. U.S.P. 1,349,200, 10.8.20. Appl., 23.10.17.

THE glass-engaging portion of a cylindrical bait is made of metal with a coefficient of expansion substantially the same as that of glass. When drawing glass cylinders the relatively thin lower edge of the metal bait is immersed in molten glass until the glass is welded to it. The bait is then drawn upwards, and the resulting cylinder severed at a point near the bait.—H. S. H.

Quartz; Production of articles impervious to gases from —. Deutsch-Englische Quarzschmelze G.m.b.H. G.P. 319,895, 5.2.18.

A MASS of fused quartz from an electric furnace is subjected to blowing and pressing under heat, and heat is then applied directly to the surface by which means a high surface-glaze is obtained. During the operation a layer of quartz may be sintered on to the under surface, on which a glaze may also be produced by heating. The process ensures absolute imperviousness of the material to gases.—W. J. W.

Clays; Treatment of — and manufacture of articles and materials therefrom. W. Smith. E.P. 149,440, 13.5.19.

ARTICLES and materials uniting the properties of clays and carbon are made by mixing clay with bituminous or oil shale or other material containing

a volatile compound of carbon which, on heating, is decomposed with deposition of carbon. The mixture, or an article made from it, is embedded in powdered coke or oil shale so as to impose a resistance to the escape of the volatile matter, and is then fired at 700°—800° C., at which temperature the product has a maximum porosity and carbon content. If the product is then fired to 1600° C. it becomes very hard and resistant to acids (resembling carborundum), and may be ground and used as an abrasive material. Alternatively, biscuitware may be saturated with a suitable liquid hydrocarbon and fired with exclusion of air so as to produce a similar product.—A. B. S.

Bauxite; Process of manufacturing dense ware from —. A. F. Meyerhofer, Assr. to Chemical Foundation, Inc. U.S.P. 1,350,825, 24.8.20. Appl., 11.10.16.

DENSE, non-porous refractory bauxite wares are obtained by heating the bauxite, finely grinding the product and mixing it with water to form a plastic mass which is then shaped and baked.

—D. F. T.

Acid-resisting material impervious to water; Preparation of —. J. Frenz. G.P. 321,029, 11.10.17.

PRIME or other absorptive material is finely powdered, dried, mixed with cement or hydraulic lime, strongly heated, and then impregnated with paraffin. A tar product is then added, after which the mass is heated to promote intimate admixture of the ingredients, and cooled.—W. J. W.

Flooring material; Manufacture of a permanently rough stoneware —. M. Gotthard. G.P. 321,158, 28.12.13.

GRAINS of clay, rich in iron, are mixed with stoneware raw material, the action of heat during the firing causing these to swell.—W. J. W.

Tunnel kilns. U.S.P. 1,348,510—1. See I.

Corundum. G.P. 319,814. See X.

IX.—BUILDING MATERIALS.

Concrete. Lea. See X.

Bituminous coatings for concrete. See XIII.

PATENTS.

Walls, linings, panels, roof-coverings, coach bodies or the like; Composition for use in the construction of —. J. F. Leger. E.P. 2311, 29.1.14.

A **STRUCTURAL** composition with a metallic trellis work reinforcement consists of a mixture of magnesia 1 pt., Canadian asbestos fibre $\frac{1}{2}$, common asbestos fibre, powder or waste $\frac{1}{2}$, corkwood $\frac{1}{2}$, wood pulp 1, tale $\frac{1}{2}$, barium sulphate $\frac{1}{2}$ pt., and sufficient magnesium chloride solution of 15°—30° B. (sp. gr. 1.08—1.26) to form a paste which, when set, binds the materials together.—A. B. S.

Fibrous composition [for paving]; Apparatus and process for manufacturing —. G. A. Henderson. E.P. 148,611, 30.4.19.

Wood chips varying in size from impalpable dust to particles which will pass a mesh of 4" x 4" are boiled in order to remove sap, etc., after which, if necessary, they are freed from pyroligneous acid by agitation with a neutralising agent. The chips containing approximately 66% of moisture are then introduced by means of a screw conveyor into a drum with central revolving blades, which is surrounded by a tank of bitumen heated by a furnace; the fumes from the tank are drawn through the drum and carry off the dust to a collecting bin.

The chips with 15—20% of residual moisture are cooled and mixed for 1 min. in steam-jacketed pug mixers with sulphuretted bitumen prepared from sulphur and artificial asphalt produced from petroleum and preheated to 300° F. (149° C.), the proportions being chips 40 and bitumen 60%. The mixture flows into a drum of the type described, where it is heated to 250° F. (121° C.) in 5 mins. The impalpable wood dust, which may be mixed with road dust or other mineral dust, is introduced in small quantities at regular intervals, and the mixture is cooled and compressed into the required shape.—W. J. W.

Tiles, flags, sheetings or the like of cement and fibre; Manufacture of —. E. Steiger. E.P. 149,120, 26.6.19.

EFFLORESCENCE on tiles etc. made of asbestos fibre and Portland cement may be prevented if tiles containing more than 2% of water, but not sufficient to saturate them, are exposed to the action of carbon dioxide. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 1, of Patents and Designs Acts, 1907 and 1919, to E.P. 23,559 of 1912; J., 1913, 912.) —A. B. S.

Concrete hardening. C. Ellis and H. S. Riederer. U.S.P. 1,348,099, 27.7.20. Appl., 23.3.15.

PORTLAND cement concrete is made with a superficial layer in which are distributed copperised iron granules of a fineness between 15- and 25-mesh. —W. J. W.

Flooring material. G.P. 321,158. See VIII.

Coating composition. E.P. 149,365. See XIII.

Protective coating. U.S.P. 1,350,343. See XIII.

Slag from refuse. E.P. 119,033. See XIXb.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Steel; Influence of vanadium on the determination of chromium in high-speed tungsten —. P. Slawik. Chem.-Zeit., 1920, 44, 633.

In the titration of chromium in steel containing vanadium and tungsten, a red-brown coloration develops as soon as an excess of ferrous sulphate solution has been added and the colour changes to yellow during the titration with permanganate, thus obscuring the end-point. The red-brown coloration is due to the formation of a complex vanadium phosphotungstate (phosphoric acid is used with sulphuric acid to dissolve the sample), but its presence does not affect the accuracy of the determination of the chromium if, in titrating the excess of added ferrous sulphate, permanganate solution is added until a red coloration is obtained which changes to yellowish-brown and not to pink. —W. P. S.

Steel; Effect of arsenic in —. P. E. McKinney. Chem. and Met. Eng., 1920, 23, 294.

COMPARISON of steel without addition of arsenic with the same steel after the addition of 0.1 and 0.5%, respectively, showed that up to 0.3% As appears to be without detrimental action so far as the static tests are concerned.—W. H. C.

Carbon-free ferronickel in a high-frequency induction furnace. D. Wilson. Chem. and Met. Eng., 1920, 23, 251.

Discs of electrolytic nickel, contained in a quartz crucible, placed within a quartz tube and surrounded with a coil, were melted and thoroughly agitated and mixed by the electro-magnetic forces induced in the nickel when an electric current was

passed through the coil. Iron was added from time to time, and the mixing was so efficient that the opposite ends of a cast rod of alloy differed by less than 0.01% in composition. 4.4 lb. of metal was charged, melted, and poured within 40 mins. with an expenditure of 12 kw. of electric energy.

—W. H. C.

Lead and silver refining slags and residues; Complete analysis of —. W. Stahl. Chem. Zeit., 1920, 44, 649—650.

THE finely-divided material is dissolved in nitric acid, the insoluble matter filtered off and fused with sulphur-sodium carbonate mixture. The insoluble sulphides in the melt are dissolved in nitric acid and added to the original filtrate, which is treated with hydrogen sulphide. The precipitate is extracted with ammonium sulphide, the extract added to the filtered solution of the fusion melt, and the residue used for the determination of copper, lead, and silver, while iron and zinc are determined in the filtrate from the hydrogen sulphide treatment. The solution of the sulpho-acids is acidified with dilute sulphuric acid, the precipitate is dissolved in colourless ammonium sulphide, the solution treated with potassium chlorate and hydrochloric acid, diluted, tartaric acid added, and the arsenic precipitated with magnesia mixture in the slightly ammoniacal solution. Tin and antimony are separated by hydrogen sulphide in boiling oxalic acid solution, the antimony sulphide being weighed as such after ignition at 300° C. in carbon dioxide, and the tin in the filtrate deposited by electrolysis and weighed as metal. Sulphur is determined in a separate portion by fusion with sodium carbonate-nitrate mixture, and eventual precipitation as barium sulphate, the lead being removed by passing carbon dioxide through the alkaline solution of the melt.—A. R. P.

Copper ores; Leaching and concentrating mixed —. A. Crowfoot and K. H. Donaldson. Eng. and Min. J., 1920, 110, 471—474.

THE material used in the tests consisted of old slime tailings from a dump and contained 1.39% Cu, 0.85% Cu being soluble in acid. The slime mixed with sufficient water to give a 1:1 pulp was passed through a tube mill; sulphuric acid was added to the discharge from the mill, which was then pumped into leaching tanks. After standing for some time the pulp was passed over a drag belt classifier; the sands were returned to the mill and the slimes passed directly to a flotation plant. The concentrates from this went to the smelter, while the tailings were run into a Dorr thickener. The overflow from the latter went to the precipitating tanks, and the slimes were filtered and washed in an Oliver filter, the filtrate going to the precipitating tanks, where the copper was precipitated on iron. The process gave an 84% extraction.—A. R. P.

Copper refinery; The power problem in a —. L. Addicks. Chem. and Met. Eng., 1920, 23, 275—278.

SUFFICIENT steam cannot be obtained from the boilers heated by the waste gases from the smelting furnaces to meet the requirements for generating current, for heating the electrolyte, for evaporating and other needs. The methods of supplying the extra quantity are discussed, and several tables and diagrams are given, together with a summary showing the distribution of the steam requirements.

—W. H. C.

Zinc concentrates; Roasting — in suspension. C. H. Fulton and J. B. Read. Eng. and Min. J., 1920, 110, 405—408.

THE dry, finely-divided ore (e.g., flotation concentrate), preheated to 60°—100° C., is fed by means

of a hopper and screw conveyor into a pipe where it meets a stream of moderately preheated high-pressure air (20—60 lb.), by which it is carried through an injector pipe into a long vertical combustion tube lined with refractory material. An excess of air over that necessary to roast the ore is forced by means of a cycloidal blower through two stoves, heated by natural gas, wherein it is heated to the necessary temperature (800° C.) to start the combustion, after which it enters the vertical tube of the furnace below the injector pipe, thus carrying the ore up the tube and at the same time roasting it. When the ore reaches the top of the tube it falls through a larger annular space, in which the roasting is completed, into the ore bin. In this way the heat of combustion of the ore is fully utilised, and the issuing gases, after passing through a Cottrell plant, are sufficiently rich in sulphur dioxide and oxygen to be utilised directly in the manufacture of sulphuric acid. The average sulphur content of the roasted product from several tests was 3.6%, much of which was present as sulphate. The amount of air required was 41—55 cub. ft. per lb. of blende, and the volume of the issuing gases about 50 cub. ft. per lb., containing 8% SO₂, 10% O₂, and 82% N₂.—A. R. P.

Zinc; Analysis of commercial —. E. Olivier. Ann. Chim. Analyt., 1920, 2, 199—207, 226—234.

METHODS, chiefly colorimetric, are described for the determination of impurities in commercial zinc. The methods of separation used are, briefly, as follows:—The sample is dissolved in nitric acid; a clear solution is obtained unless tin is present. The solution (clear or turbid) is treated with 10 c.c. of ferric chloride solution (0.1 g. Fe), and excess of ammonia is added; the ferric hydroxide precipitate formed also contains the tin, lead, arsenic, and antimony, whilst the solution contains the zinc, copper, and cadmium. When the mixture is filtered and the filtrate treated, drop by drop, with sodium sulphide solution, cadmium and copper sulphides are precipitated before the zinc. Addition of cyanide prevents the formation of copper sulphide. All commercial zincs are completely soluble in a mixture of nitric and tartaric acids, and the solution remains clear when an excess of ammonia is added; the addition of sodium sulphide precipitates first the lead, then the copper and cadmium, and finally a portion of the zinc, whilst arsenic, antimony, tin, and the greater portion of the zinc remain in solution. In the colorimetric determinations, iron is estimated as thiocyanate and the other metals as sulphides, the comparison solutions being prepared from a specimen of zinc containing known amounts of the metals.—W. P. S.

Zinc; Allotropy of —. K. E. Bingham. Inst. Metals, Sept., 1920. [Advance copy.] 21 pages.

FROM observations of the electrical resistance, thermal E.M.F., electrolytic potential, specific gravity, hardness, mechanical properties, thermal curves, and microstructure of the pure metal (99.97% Zn), it is concluded that zinc exists in three allotropic forms, the temperatures at which the critical changes occur being between 160° and 210° C. and between 300° and 340° C.—W. E. F. P.

Casting temperature; Measurement of — in the brass foundry. J. Arnott. Engineering, 1920, 110, 277.

MEASUREMENTS of the temperature of molten brass by radiation and optical pyrometers are rendered unreliable by the oxide films on the surface of the metal and the quantities of fume evolved. When using thermocouples with protecting sheaths the time lag is an undesirable feature; it is, therefore, recommended to use a nickel—nickel-chromium (9% Cr) couple insulated with asbestos and en-

cased for the greater part of its length in a steel tube, a few inches above the junction being left bare for immersion. This couple, which has an E.M.F. of 40 milliv. at 1000° C., will indicate the temperature of the molten metal within 1 min. of immersion and, when the junction is burnt through the ends, may be readily welded together by means of the oxy-acetylene flame. Examples are given of the effect of casting temperature on the tensile strength of gun-metal.—A. R. P.

Ferrocium. A. Hirsch. Trans. Amer. Electrochem. Soc., 1920, 711—714. [Advance copy.]

THE preparation and properties of ferrocium are described, and the use of "Misch-metal" (an alloy of metals of the cerium group) as a deoxidiser discussed. Addition of the latter to cast iron renders the castings softer, denser, more easily machinable and free from gas and pinholes and increases the transverse strength. When added to molten copper just before pouring, the resulting metal is free from oxides and gases, and the tensile strength, elongation, and reduction of area are raised.—A. R. P.

Copper-zinc alloys prepared by melting or by electro-chemical processes; Chemical and electro-chemical properties of —. F. Sauerwald. Z. anorg. Chem., 1920, 111, 243—279.

THE properties of copper-zinc alloys obtained by melting are to a large extent independent of the composition; this is due to the protecting action of the copper in the mixed crystals. At 380° C. the protecting action of the copper is no longer operative, owing to the increased mobility of the atoms, and the properties are then dependent on the concentration. In alloys electrolytically deposited at the ordinary temperature the copper has not this protecting action, but if the alloy is formed by electrolysis of fused salts at 300° C. the product is much the same as that formed by melting. (Cf. J.C.S., Oct.)—J. F. S.

Copper-aluminium-zinc alloy; Corrosion of a screw fitting made of —. O. Bauer. Z. Metallkunde, 1920, 12, 129—131. Chem. Zentr., 1920, 91, IV., 256.

A SCREW fitting made of a zinc alloy containing 4.85% Cu, 2.19% Al, 0.92% Pb, 0.15% Sn, and 0.03% Fe became brittle and partially disintegrated after a short time in use. Alloys containing much zinc should never be used on boilers, especially in places where they are likely to become damp in contact with more electropositive metals or iron, bronze, or brass.—G. F. M.

Materials [metals and alloys and concrete]; Effect of temperature on some of the properties of —. F. C. Lea. Sect. G., Brit. Ass., 1920. Engineering, 1920, 110, 293—298.

TENSILE tests from 0° to 900° C. and hardness tests from 0° to 500° C. were carried out in electrically-heated furnaces on metals and alloys. Both tensile strength and hardness were found to decrease as the temperature was raised. In the case of steel used for reinforcing concrete a breaking strength of 7 tons per sq. in. was reached between 615° and 640° C., so that concrete beams cannot be expected to stand if exposed to a fire for sufficiently long to allow the steel to reach approximately 650° C. Further tests were carried out on concrete cylinders and cement briquettes and cubes to ascertain the protective influence of concrete on the steel reinforcement. The results indicated that the usual thickness of concrete was insufficient to prevent the steel from being heated to the dangerous temperature of 650° C. if exposed for several hours to a fierce fire.—W. H. C.

Metals; Metastability of the — as a consequence of allotropy and its significance for chemistry, physics, and technics. H. E. Cohen and H. B. Bruins. Z. physik. Chem., 1920, 94, 443—449.

A REPETITION and confirmation of previously published work (see J., 1914, 597, 1159; 1915, 908) in connection with a change in density of metals after various treatments.—J. F. S.

PATENTS.

Iron; Process of extracting — from its ores. C. C. Jones. U.S.P. 1,348,804, 3.8.20. Appl., 19.6.16.

A MIXTURE of iron ore and petroleum is embedded in solid carbonaceous fuel in containers from which air is excluded, and the containers are then heated to a temperature below fusion point, but sufficiently high to reduce the ore. The reduced iron retains enough carbon from the petroleum in its interstices to form a conglomerate mass.—W. J. W.

Spiegeleisen or high-percentage ferromanganese; Reducing pyrolusite [to manganous oxide] before use in the production of —. J. Hub. G.P. 322,610, 25.12.12.

A COMPACT mixture of pyrolusite and coal or small coke is gradually fed into a furnace and heated to red-heat; the reaction between the coal and the oxygen liberated raises the mass to white-heat, the charge melts, and reduction is completed, a slag of manganous oxide being formed.—L. A. C.

Rusting of iron; Prevention of the —. H. Koelsch. G.P. 319,855, 25.3.14.

THE surface of the iron is treated with a nitrite—for example, a solution of sodium nitrite in glycerin or a mixture of sodium nitrite and vaseline.

—G. F. M.

Metallic oxide ores; Reduction of —. J. W. Moffat. U.S.P. 1,348,889, 10.8.20. Appl., 15.8.19.

A FURNACE for reduction of oxide ores consists of a stationary, horizontal, cylindrical reduction chamber with an air-tight charging gate at the top and an air-tight discharge outlet at the bottom, and is fitted with apparatus working in closed orbits for lifting and re-lifting the charge from the bottom of the furnace and dropping it again from the upper part of the furnace.—J. W. D.

Ores, metal oxides and the like; Reduction of —. Kohle und Erz, G.m.b.H. G.P. 320,065, 30.11.15.

REDUCING gases are heated above the theoretical reduction temperature, by electrical means or otherwise, before being passed through the charge of ore and coal.—C. A. M.

Manganese ores; Treating oxide —. G. H. Clevenger and M. H. Caron, Assrs. to Research Corp. U.S.P. 1,349,322, 10.8.20. Appl., 23.9.18.

THE ores are treated to reduce the manganese to a stage lower than the manganic oxide stage and the manganese is then concentrated by magnetic separation.—J. W. D.

Lead; Removing — from copper scrap. J. L. Jones, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,319,382, 10.8.20. Appl., 16.1.19.

COPPER scale is added to molten copper scrap to oxidise the impurities, and the purified copper is separated from the oxidised impurities.—J. W. D.

Heat regenerator [for metallurgical furnaces]; Process of protecting metal pipes exposed to high temperatures. L. B. Skinner. U.S.P. (A) 1,350,267 and (B) 1,350,269, 17.8.20. Appl., (A) 10.2.19, (B) 11.2.18; (B) renewed 9.7.20.

(A) THE passages for conducting the gases to and

from the furnace are separated by a metallic wall, and the interiors of the passages are coated with an adherent protective coating of condensed metallic fume. (b) The interior surfaces of the conduits of heat regenerators are coated with the compounds of volatile metals present in the fume (e.g., zinc oxide) by passing the gases through the pipes at such a temperature as to deposit a coating of the fume on them without burning the iron. The furnace is then operated in such a manner that the coated pipes are at a temperature above 1000° C. while the air for combustion is passing through them.—A. R. P.

[Nickel] alloy. H. S. Cooper, Assr. to Zircon Tool and Alloy Co. U.S.P. 1,350,359, 24.8.20. Appl., 22.8.18.

AN alloy of nickel with 0.5–2% Zr and at least twice as much aluminium and silicon as zirconium is suitable for making high-speed cutting tools.

—A. R. P.

Copper and zinc; Recovery of — from burnt pyrites. Reisenegger. G.P. 307,085, 7.3.17.

CHLORINE is led into a sludge of burnt pyrites and water; copper and zinc are dissolved as chlorides and are separated by crystallisation.—L. A. C.

Pyrites; Process for extracting zinc from burnt —. Process for separating zinc from burnt pyrites by extraction with dilute acid. Farbenfabr. vorm. F. Bayer und Co. G.P. (a) 312,384 and (b) 320,066, 11.5.18.

(A) BURNED pyrites is leached with hydrochloric acid or sulphuric acid and sodium chloride, while chlorine is simultaneously introduced. (b) By first mixing the burnt pyrites with manganese are the zinc content is reduced below 1%, and the extracted material may then be smelted to obtain the iron.—C. A. M.

Noble metals; Process and apparatus for obtaining — from very dilute solutions (sea-water etc.). E. Zander. G.P. 310,722, 21.3.17.

A MEASURED larger quantity of another substance (e.g., a mercury salt) is added to a very dilute solution of a noble metal, and precipitated simultaneously with the latter, e.g., by means of bases, or hydrogen sulphide, in a form which can be rapidly separated by filtration or otherwise.—C. A. M.

Light metals; Process for recovering — from turnings, residues, and ashes. K. Hess. G.P. 320,220, 30.7.18. Addn. to 318,304 (J., 1920, 520 A).

RESIDUES etc. containing light metals are fused with rock salt and the mass of salt saturated with impurities cooled and left in cold water until the salt has dissolved. The salt may subsequently be recovered from the solution.—C. A. M.

Zinc ore; Preparation of briquettes of — suitable for smelting. Schlesische A.-G. für Bergbau und Zinkhüttenbetrieb. G.P. 319,813, 28.3.14.

PLASTER of Paris is used as a binding agent for zinc ore briquettes, with or without the addition of pitch or "cell-pitch".—C. A. M.

Zinc and corundum; Alumino-thermic process of obtaining —. Kohle und Erz G.m.b.H. G.P. 319,814, 8.5.17.

AN intimate mixture of aluminium with zinc oxide (as pure as possible) and the same quantity of zinc dust is subjected to the thermite process. The speed of the reaction can be regulated, and corundum is obtained as a by-product.—C. A. M.

Zinc distillation process; Utilisation of the residual ash from the — by blowing with air or gas. Schlesische A.-G. für Bergbau und Zinkhüttenbetrieb. G.P. 321,526, 7.11.13.

AFTER each distillation the ash is collected in pockets situated below the muffles, and by means of a current of air or other gas, the heat contained in the ash is utilised for preheating the next charge in the muffles.—J. H. L.

Aluminium; Treatment of — with cadmium. S. Buchalo. G.P. 320,139, 26.1.15.

FROM 0.5 to 5% of cadmium is added to fused aluminium at a temperature not materially exceeding the m.p. of the bath, and the mixture vigorously stirred and rapidly heated to over 770° C., before being poured into moulds. The cadmium volatilises at 770° C., and combines with the oxygen in the air-bubbles in the molten metal, so that the particles of aluminium oxide in the latter become coated with cadmium oxide, which separates readily from molten aluminium. The resulting casting is finely granular, homogeneous, free from bubbles, and has increased strength and elasticity.

—C. A. M.

Blast-furnaces; Process for treating ores in —. L. P. Basset. U.S.P. 1,349,598, 17.8.20. Appl., 12.10.17.

SEE E.P. 112,275 of 1917; J., 1920, 269 A.

Alloys containing nickel; Method for producing metallic —. M. N. Salvati; G. Beer and A. Tedesco, exors., Assrs. to Soc. Anon. Stabilimenti Biak. U.S.P. 1,349,918, 17.8.20. Appl., 19.12.17.

SEE E.P. 111,290 of 1917; J., 1918, 473 A.

Furnace; Regenerative [soaking-pit] —. C. Stein, Assr. to C. M. Stein et Cie. U.S.P. 1,350,624, 24.8.20. Appl., 30.4.19.

SEE E.P. 120,039 of 1918; J., 1919, 779 A.

Lead; Process for the extraction of — from its ores. F. E. Elmore. U.S.P. 1,350,959, 24.8.20. Appl., 11.4.19.

SEE E.P. 129,773 of 1918; J., 1919, 685 A.

Tinplates and other metal-coated plates or sheets; Apparatus to be used in manufacture of —. H. S. Thomas and W. R. Davies. E.P. 149,106, 30.5.19.

Cast ingots; Method and means for producing — [in vacuum moulds]. R. I. Henderson. E.P. 149,479, 2.6.19.

Furnaces; Continuous reheating —. The Wellman Smith Owen Engineering Corp., Ltd., and A. V. Kemp. E.P. 149,509, 18.7.19.

Aluminium. U.S.P. 1,350,150. See VII.

XI.—ELECTRO-CHEMISTRY.

International Weston cell; Metastability of the — and its unsuitability as a standard of [electrical] tension. E. Cohen and A. L. T. Moesveld. Z. physik. Chem., 1920, 95, 285–304.

THE cell recommended by the International Conference on Electrical Standards and Units, containing a 12.5% cadmium amalgam, and the cells supplied by the Weston Co., containing 12–13% cadmium amalgam, are both metastable below 12° C., and suddenly become stable with a change of E.M.F. of 6 millivolts. They are therefore unsuitable as standards of electromotive force. A cell,

built on the same lines, but containing 8% cadmium amalgam is recommended; this is stable from 0° to 40° C., and follows the temperature formula put forward by the International Conference. (*Cf.* J.C.S., Oct.)—J. F. S.

Oxidation of potassium plumbite. Jirza. *See* VII.

Carbon-free ferronickel. Wilson. *See* X.

PATENTS.

Non-metallic resistance element and process of making same. A. L. Feild, Assr. to National Carbon Co., Inc. U.S.P. 1,349,053, 10.8.20. Appl. 16.6.19.

As electrical resistance element having a temperature resistance coefficient substantially zero is composed of a compound of a metal of the titanium-zirconium group and a refractory oxide.—W. F. F.

Electric furnace doors. Booth Electric Furnace Co., Assecs. of J. R. Hall. E.P. 137,510, 8.1.20. Conv., 4.4.18.

Electrical precipitation. U.S.P. 1,319,362. *See* I.

Hydrocarbon gas. E.P. 146,560. *See* IIa.

Waste liquors from digestion of straw. G.P. 319,068 and 321,453. *See* V.

Insulating coating. U.S.P. 1,350,313. *See* XIII.

Ageing spirits. E.P. 141,687. *See* XVIII.

XII.—FATS; OILS; WAXES.

Oxidised fatty acids in olive oils extracted with carbon bisulphide; Determination of —. H. Stadlinger. Z. öffentl. Chem., 1920, 26, 162—161, 169—172.

RESIN-LIKE substances giving the Liebermann-Storch reaction, and apparently related to oxidised fatty acids, were isolated by Twitchell's method from technical olive oils which had been extracted with carbon bisulphide. The following results were obtained with 47 commercial samples of such oils:—Unsaponifiable matter, 0.2—9.6; oxidised fatty acids, 1.3—12.8; and normal fatty acids, 77.2—91.7%. The results obtained in the determination of oxidised fatty acids will vary with the nature of the petroleum spirit used for their separation. Thus, the amounts determined in 7 samples by means of commercial petroleum spirit fractionated at about 70° C. ranged from 1.3 to 6.5%, whilst with "normal benzene" they were 7.1—14.0%. The petroleum spirit used should preferably have sp. gr. 0.695—0.705 at 15° C., b.p. 65°—95° C., and should be as free as possible from unsaturated and benzene hydrocarbons.—C. A. M.

Cottonseed oil products; The Kreis [rancidity] reaction of —. W. B. Smith. J. Ind. Eng. Chem., 1920, 12, 764—766.

REFINED cottonseed oils and their products sometimes contain chromogenic substances, derived from the crude oil, which may cause a non-rancid oil to give a positive reaction with the Kreis test (*cf.* Kerr, J., 1918, 475 A); the latter is, therefore, not entirely trustworthy for the detection of rancidity in cottonseed oil.—W. P. S.

Oils and fats resistant to changes of temperature, and melting and solidification points of fats. A. Eisenstein. Oel u. Fettind., 1919, 1, 499—501, 527—528, 548—549, 573—575. Chem. Zentr., 1920, 94, II., 811—812.

MIXTURES of triglycerides probably behave like mixtures of fatty acids in showing a m.p. lower than

the calculated value. The melting point curve of mixtures of palmitic acid and stearic acid has portions which are nearly straight lines between 70 and 100% of palmitic acid and 60 and 100% of stearic acid. The m.p. curve of mixtures of Borneo tallow and coconut oil lies partly below and partly above the calculated curve. The more mixed glycerides contained in an oil the greater its resistance to cold, and the smaller the proportion of high-melting fat resistant to heat which can be obtained at a given temperature.—C. A. M.

Catalytic actions at solid surfaces. I. Rate of change conditioned by a nickel catalyst and its bearing on the law of mass action. E. F. Armstrong and T. P. Hilditch. Proc. Roy. Soc., 1920, A, 98, 27—40. (*Cf.* J., 1920, 116 A.)

THE hydrogenation of linseed oil yields a curve, the initial portion of which (30% of the whole curve) corresponds to the hydrogenation of the whole of the linolenin and most of the linolein; a very abrupt curvature then sets in and this is followed by an approximately linear curve at a much lower slope. Experiments carried out with oil loads of 200, 300, and 400 g. and 0.45 and 1.5 g. of nickel show that the factor v/t (J., 1920, 120 T) depends on the mass of nickel present; the impurities in the oil gradually affect the catalyst, and with the small quantity of nickel the rates are practically the same for loads of 200 and 300 g., but with 100 g. the linear portion of the curve is shortened owing to increased effect of the impurity.—J. F. S.

Fat from starch. Taylor and Nelson. *See* XVII.

PATENTS.

Cotton-seed meats; Treatment of —. C. O. Phillips. U.S.P. 1,347,870, 27.7.20. Appl., 3.8.18.

THE material is incorporated intimately with dilute caustic soda solution, in quantity insufficient to neutralise more than a small fraction of the total fatty acids present, the mixture is subjected to a cooking operation, and the oil then expressed.

A. de W.

Fat; Process for obtaining — from sulphite-cellulose liquors by biological methods. W. Jeroch, and Kriegsausschuss f. pflanzliche u. tierische Oele u. Fette, G.m.b.H. G.P. 305,091, 25.1.17.

AERATED and neutralised sulphite-cellulose liquors to which a nitrogenous substance, such as beet juice, has been added, are inoculated with a culture of *Endomyces vernalis*.—G. F. M.

Silicate soaps; Production of —. T. E. Blasweiler. G.P. 320,829, 11.4.19.

WATER-GLASS is used in such excess that no separation of silicic acid occurs; *e.g.*, 5 kg. of resin is kneaded with 120 kg. of water-glass of 38° B. (sp. gr. 1.35), or 5 kg. of fatty acids from soya bean oil with 150 kg. of water-glass.—J. H. L.

Emulsions; Method of making —. J. A. de Cew. U.S.P. 1,347,734, 27.7.20. Appl., 30.12.18.

As emulsifiable substance is forced into contact with an aqueous solution, the mixture vigorously agitated at about 180° F. (82° C.), under pressure, and the resulting emulsion or emulsoid discharged into an aqueous solution having a lower temperature.—C. A. M.

Soapy liquors; Treatment of —. C. L. Peck. Assr. to The Dorr Co. U.S.P. 1,319,530, 10.8.20. Appl., 1.7.19.

SOAPY liquors are coagulated, and the coagulated particles removed from the liquid by flotation, *i.e.*, the liquid is subjected to the action of gas bubbles and the entrapped coagulated matter floated off. The organic substances in the coagulum are subsequently recovered.—A. de W.

Roller mills [; Controlling the feed of oil seed or the like to —]. J. D. Lee, B. P. Flockton, and Manlove, Alliott and Co., Ltd. E.P. 149,048, 2.5.19.

Solvent for fats. G.P. 320,807. See XIII.

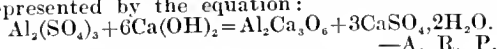
Moulds rich in fat. G.P. 320,560. See XVIII.

Calamus root. G.P. 307,623. See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Satin-white; Preparation and chemistry of —. A. Cobenzl. Chem.-Zeit., 1920, 44, 661–662.

SATIN-WHITE is prepared as follows:—85 kg. of quicklime is slaked with 200 l. of boiling water, the sludge diluted to 450–500 l., and passed through a sieve into a large vat. After cooling, a cold solution of 125 kg. of aluminium sulphate (18% Al_2O_3) in 500 l. of water is slowly added with vigorous agitation, followed by 1100–1200 l. of water to prevent the mass setting. The liquid is finally diluted to 6000 l.; 100 c.c. of the clear solution should not require more than about 25 c.c. of N/10 oxalic acid to render it neutral to phenolphthalein. If more alkaline than this, 4 kg. of sulphuric acid of 40° B. (sp gr. 1.38) is added. Any slight trace of yellow colour is removed by the addition of 100–150 c.c. of a solution of 250 g. of commercial Indanthrene Blue 2GSZ paste in 5 l. of water. The satin-white is then collected in a filter-press. The materials used must be as free as possible from iron, sand, and carbonaceous matter, and the best results are obtained by using slightly basic aluminium sulphate (i.e., that containing only 80–85% of the theoretical sulphuric acid). This may be prepared by treating a solution of the commercial salt with the necessary quantity of lime, allowing the precipitate to settle, and siphoning off the clear liquid for use. Experiments show that the reaction involved in the process is represented by the equation:



—A. R. P.

Bituminous acid-proof coatings for acid-proofing concrete surfaces. Report by Bituminous Materials and Cement Section, U.S. Bureau of Standards. Chem. and Met. Eng., 1920, 23, 287–289.

EITHER bituminous paint, enamel or mastic is used, depending upon the character of the surface to be coated, the strength of the acid, and the desired resistance to changes of temperature and abrasion. The surface to be coated must be clean, dry, and free from dust, and a priming coat should always be applied. Paints are made from bitumen or coal-tar pitch thinned with solvents, the priming material being thinner than the paint. Enamels are mixtures of bitumen of high-melting point thoroughly incorporated with 15–40% of finely-powdered silicious filler; they contain so much bitumen that they melt below 350° F. (177° C.), and are applied hot. The priming material consists of the same kind of bitumen thinned. Mastics are mixtures of hard asphalt, ground with asbestos and finely-powdered silicious mineral filler; when ready for application the mixture contains about 15% of asphaltic binder, 20% of mineral filler, and 65% of properly graded, coarse sand or other aggregate. Mastics are applied hot when the priming coat of thinned asphalt has dried to a tacky condition. Several layers of about 1–32 in., each poured on and spread out with a trowel, are applied one over the other so as to give a final thickness of at least 1 in. General specifications for each kind of material are given,

together with the necessary tests to be applied to ensure compliance with the specifications.

—W. H. C.

“Tunga” varnish resin; Properties and application of —. H. A. Gardner and P. C. Holdt. Paint Manufacturers' Assoc. U.S.A., Circ. No. 102. Aug., 1920. 3 pp.

TUNGA varnish resin (Paint Manuf. Assoc. U.S.A., Circ. 101) is prepared by esterification of rosin with glycerin either as a separate process or during the actual varnish-making process. The resin has a dark reddish-brown colour, sp. gr. 1.132, m.p. (capillary tube) 105°–125° C., acid value (in alcohol-benzol) 16.2, saponif. value 71.59. It is slightly softened by hot alcohol, completely soluble in hot benzol and hot oil of turpentine, insoluble in amyl alcohol, acetone, and petroleum spirit. Tunga resin is suitable for the manufacture of elastic, hard and spar (boat) varnishes. An elastic varnish made from tunga resin and tung oil dried hard in 7 hrs., and the dried film was very resistant to the action of boiling water. Varnishes made from tunga resin with perilla, mixtures of perilla and tung, and perilla and linseed oils have been prepared. With perilla oil there is somewhat more difficulty of manipulation than with tung or linseed oils.

—A. de W.

PATENTS.

Composition impervious to oils, spirits, water, and the like [for coating concrete etc.]. C. H. Iverson and G. S. Roberts. E.P. 149,365, 19.11.18.

A COMPOSITION contains 30–65 pts. by weight of zinc chloride, 30–55 pts. of zinc oxide, and 2–20 pts. of ammonium chloride together with other ingredients such as silica, borax, or powdered glass; magnesium oxide and chloride may replace a portion of the zinc compounds, and agglutinants, such as dextrin, starch, gum, or glue, may also be added. The mixture is made into a paste with water for application to the material to be treated.

—L. A. C.

White lead; Process for producing —. E. Euston. U.S.P. 1,349,334, 10.8.20. Appl., 22.3.19.

APPROXIMATELY neutral lead acetate solution is treated with carbon dioxide gas, basic lead acetate solution is introduced in amount and at such a rate as to maintain a neutral condition of the solution, and the treatment with carbon dioxide gas is continued until a part of the precipitate of basic lead carbonate is converted into the normal salt, and a mixture is obtained to yield a product containing 11.3–14.3% CO_2 .—A. de W.

Insulating and protective coating; Process for the making of an —. E. R. Stowell, Assr. to Stowell Fireproofing Co. U.S.P. 1,350,343, 24.8.20. Appl., 1.7.18. Renewed 10.4.20.

POWDERED silicon carbide (8lb.) is mixed with aqueous sodium hydroxide (1 gall.), and the mixture is allowed to stand in a covered container until the hydrogen gas formed by the action of the alkali on the free silicon is exhausted. The foam-like product is stirred until it is reduced to its original volume, and 1 pint of “50° to 54° sodium silicate” is then added per gall. of composition.—L. A. C.

Lampblack and hydrogen; Preparation of — by decomposition of hydrocarbons. Berlin-Anhaltische Maschinenbau A.-G. G.P. 312,546, 2.6.17.

DECOMPOSITION takes place in a series of retorts, pipes, or chambers heated to successively higher temperatures. For methane the stages are 700°–800°, 900°–1000°, and 1200°–1400° C. In the first stage carbon is deposited as lampblack, and in the last as graphitic carbon.—C. I.

Paint; Preparation of a — from tar and slaked lime. W. Klement. G.P. 318,699, 25.8.17.

A PAINT which furnishes a bright, smooth surface is obtained by mixing tar with milk of lime.

—G. F. M.

Paint for ships' bottoms etc. H. Burstin. G.P. 319,199, 22.11.18.

NEUTRAL or acid naphthenic acid salts of mercury, copper, arsenic, nickel, lead, or other heavy metals, or mixtures of these salts with already known paint materials are employed, either with or without solvents.—G. F. M.

Paint for wood; Preparation of a substance suitable for producing a durable —. K. S. Fuchs. G.P. 320,011, 11.5.19.

THE liquors from the alkaline disintegration of vegetable matter, particularly straw, are cooled, precipitated with acids, and the precipitate filtered off and dissolved in aqueous ammonia. The solution is painted on wood, and the colour is rendered insoluble by heating to 50°–60° C., or by treatment with formaldehyde or ferric chloride.

—G. F. M.

Oil colours and varnishes; Solvent for removing dried —. Tetralin G.m.b.H. G.P. 320,152, 28.5.18.

HYDRONAPHTHALENES, either alone or mixed with other solvents, such as amyl alcohol, are used as solvents for removing dried oil colours and varnishes.—G. F. M.

Solvent for the extraction of resins, fats, essential oils, liquid and solid hydrocarbons, caoutchouc, sulphur, dyestuffs, and the like. Tetralin G.m.b.H. G.P. 320,807, 7.3.16.

THE substances named are dissolved at elevated temperatures, and, if necessary, with the aid of a vacuum, by tetrahydro-, and other more highly-hydrogenised naphthalenes.—G. F. M.

Varnish; Preparation of quickly drying — from tar products. Deutsch-Luxemburgische Bergwerks- und Hütten A.-G., and S. Hilpert. G.P. 320,808, 9.12.16. Addn. to 319,010 (J., 1920, 577A).

THE varnish consists of a mixture of coumarone resin with the resinous organic material obtained from the waste acids which have been used for washing the coal tar fractions boiling above 150° C. For example, the dark brown resin obtained from the waste sulphuric acid which has been used for washing naphthalene is suitable for the purpose.

—G. F. M.

Pencil leads and the like; Manufacturing —. E. C. R. Marks. From American Lead Pencil Co. E.P. 149,072–3, 7.5.19.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

[*Rubber.*] *Investigations on different coagulants.* O. de Vries. Comm. Central Rubber Stat., Buitenzorg, 1920, No. 19, 163–219.

SULPHURIC acid used in small proportions as a coagulant yields a rubber comparable with the product obtained with acetic acid; with a somewhat larger proportion of sulphuric acid a rubber is obtained with an abnormally slow rate of vulcanisation, but this gradually attains a normal value. Alum is best used with undiluted latex in the proportion of 3–12 g. per l.; the product shows a marked decrease in rate of vulcanisation and in viscosity, the effect increasing with the proportion of coagulant. Formic acid gave irregular results probably owing to the variable composition of the commercial article. Hydrochloric acid in

relatively high proportion gave a rubber with a slow rate of vulcanisation, and the product in two years became tacky and no longer capable of vulcanisation. In coagulating with alcohol, approximately 500 c.c. is needed for 1 l. of undiluted latex, coagulation being instantaneous; the product has a normal rate of vulcanisation, and the process possesses some advantages for experimental purposes. (See also Eaton and Whithy, J., 1918, 99A; Eaton, J., 1918, 99A, 630A.)—D. F. T.

Latex and rubber from individual trees. O. de Vries. Comm. Central Rubber Stat., Buitenzorg, 1920, No. 20, 249–271.

THE yield of individual trees varied from 4 to 71 g. of rubber per day, the rubber content of the latex ranging from 17% to 50%; the necessary vulcanisation period varied from 90 to 160 mins., and the viscosity number from 14 to 90.—D. F. T.

Hevea latex; Natural coagulation of — in the absence of air after addition of lime salts. W. Spoon. Comm. Central Rubber Stat., Buitenzorg, 1920, No. 21, 273–291.

THE latex obtained from trees during July and August, when they were leafless, was unusually resistant to spontaneous coagulation in closed vessels, the process being far from complete after 20 hrs., although this period was sufficient earlier and subsequently. During the months in question satisfactory spontaneous coagulation could be ensured by the additional presence of 0.3% of sugar and heating for 7–8 hrs. at 40° C. or of 0.1% of calcium chloride. The rubber obtained with the addition of calcium chloride vulcanised more rapidly than that prepared by the acetic acid process, and was rather less uniform in this respect, but no harmful influence of the calcium salt could be detected.—D. F. T.

[*Rubber.*] *Further data on spontaneous coagulation.* O. de Vries and W. Spoon. Comm. Central Rubber Stat., Buitenzorg, 1920, No. 21, 292–311.

RUBBER obtained by spontaneous coagulation of undiluted latex showed practically the same tensile strength and slope as rubber prepared from the same latex by the addition of acetic acid. With latex of high rubber content yielded by trees after a period of rest, spontaneous coagulation proceeded very satisfactorily, but with the latex obtained after heavy tapping, spontaneous coagulation was much less easy and less complete. The crêpe from rubber obtained by spontaneous coagulation was not so pale in colour as the crêpe ordinarily produced.—D. F. T.

Rubber; Determination of sulphur in vulcanised —. P. Dekker. Gummi-Zeit., 1920, 34, 1020–1022, 1044–1045.

FOR the oxidation of free sulphur in the acetone extract from vulcanised rubber, treatment with concentrated nitric acid (sp. gr. 1.4) and a little potassium chlorate is satisfactory. Oxidation with concentrated nitric acid saturated with bromine, followed by evaporation and fusion with a mixture of sodium carbonate and potassium nitrate (Waters and Tuttle, J., 1911, 1268; Tuttle and Isaacs, J., 1915, 436), is satisfactory for the determination of the combined and total sulphur in vulcanised rubber; Rothe's method of oxidation with nitric acid (sp. gr. 1.48) and magnesium nitrate, followed by evaporation and heating, also yields satisfactory results. In order to avoid the strong heating necessary with the last two methods, and thereby to effect a saving in crucibles or flasks, the following modified process is recommended. The sample (1 g.) is gently warmed with concentrated nitric acid (40 c.c.) and anhydrous magnesium nitrate (6 g.) until the liquid begins to froth; the mixture

is then boiled gently for 1 hr., treated with one or two drops of bromine and boiled for a further $\frac{1}{2}$ hr.; the liquid is then evaporated almost to dryness in a porcelain dish, and after the addition of concentrated hydrochloric acid again evaporated until the residue crystallises on cooling; the residue is dissolved in water and its content of sulphate estimated in the usual manner. With this and the other methods mentioned above there is a tendency to somewhat low results if the rubber contains much sulphur.—D. F. T.

PATENTS.

Rubber and similar materials; Process for vulcanising — W. A. Gibbons, Assr. to American Rubber Co. U.S.P. 1,350,798, 24.8.20. Appl., 11.12.16.

VULCANISATION is effected in a medium consisting of an inert gas inside a chamber communicating with the atmosphere; the entry of air into the chamber is prevented by a continuous current of the inert medium.—D. F. T.

Rubber; Process for vulcanising — E. E. A. G. Meyer, Assr. to Morgan and Wright. U.S.P. 1,350,824, 24.8.20. Appl., 29.4.16.

A SMALL quantity of a concentrated solution of caustic alkali is introduced into the mixture to be vulcanised.—D. F. T.

Solvent for caoutchouc. G.P. 320,807. See XIII.

XV.—LEATHER; BONE; HORN; GLUE.

Tannin analysis; Boldracco and Camilla's modification of the official shacco method of — E. Schell. J. Soc. Leather Trades' Chem., 1920, 4, 220—221. (See J., 1920, 123 A, 523 A.)

THE gelatin-acetic acid reagent proposed by Baldracco and Camilla is designed to prove complete detannisation. The modified method effects complete detannisation by the use of more hide powder, which must absorb a corresponding excess of non-tannins. The proposed modification is less accurate, and the nature of the new hide powder and the proportion of hide powder required for a certain strength of tannin are not sufficiently defined. The tannin absorbed is also in a less pure form than by the official method.—D. W.

Chrome tanning. D. Burton. J. Soc. Leather Trades' Chem., 1920, 4, 205—218.

EXPERIMENTS with violet and green chrome liquors on pieces of limed hide have shown that the violet chrome solutions tan more rapidly than green solutions of the same basicity figure, and chromium and neutral salt content. This is explained by the more acid character of the green solutions. The properties of chrome leather are influenced not only by the percentage of chromium, but also by the degree of neutralisation of the chromium salt on the fibre. The completion of the tannage is best judged from the absence of horniness and curling at the edges of a piece of leather after boiling. The leather must be neutralised before boiling, and though it may shrink after boiling, such shrinkage is no criterion for judging the tannage. The tanning properties of a chrome liquor are determined by the chromium content and the hydron concentration. The basicity figure is no guide to the latter.—D. W.

PATENTS.

Condensation products [tanning substances]; Manufacture of — from N- and O-arylsulpho derivatives of aromatic amino- and hydroxy-sulphonic acids. Ges. f. Chem. Ind. in Basel. G.P. 320,613, 20.5.16. Addn. to 319,713 (J., 1920, 553 A).

SOLUBLE aromatic compounds which have two or

more N-arylsulphamino or O-arylsulpho groups, together with a sulpho group in the molecule, are treated with formaldehyde. The products are more soluble and have more effective tanning properties than the initial materials.—W. J. W.

Hides; Process for tanning — P. Ringbauer. G.P. 321,343, 31.10.19.

THE hides are stretched in a frame in the closed tanning-vessel, which is then exhausted. The dilute tanning liquor is then introduced, without destroying the vacuum, and by agitation is caused to exert a steady pressure on both the hair and flesh sides of the hides.—W. J. W.

Extracting tannin. G.P. 320,688. See XVII.

XVI.—SOILS; FERTILISERS.

Soil; Studies of a Scottish drift — W. G. Ogg and J. Hendrick. J. Agric. Sci., 1920, 10, 333—357.

THE glacial drift soil under examination, which is free from calcium carbonate, was separated into fractions by rubbing up and shaking with water, followed by sedimentation, the usual preliminary treatment with acid and ammonia being omitted. The absorptive power of unit weight for ammonia from a solution of ammonium sulphate was greatest in the case of the "clay" fraction, this being closely approached by the "fine silt" and "silt." Powdered granite was divided into mechanical fractions by the same method, and it was found that these fractions had very similar absorptive powers for ammonia from solutions of ammonium sulphate to those of the soil fractions. After ignition there was a reduction in the absorptive power, but this was more marked in the case of the soil fractions than with the granite fractions. The absorbed ammonia was only gradually washed out by percolating water, and the whole of it could not be removed in this way.—W. G.

Soils; Effect of various soluble salts and lime on evaporation, capillary rise and distribution of water in some agricultural — M. I. Wolkoff. Soil Sci., 1920, 9, 409—436.

IN general the soluble salts used, ammonium, potassium and magnesium sulphates, sodium chloride, calcium nitrate, and potassium phosphate, and also calcium oxide, materially decreased the evaporation of soil moisture, there being apparently a direct relationship between the efficiency of the salts in this respect and the osmotic concentration of the soil solution in the top inch of soil. Sodium chloride and calcium nitrate were most effective and potassium sulphate and phosphate least effective in checking evaporation. Calcium oxide was least effective in sea sand and sandy loam, and most effective in clay loam. Sodium chloride decreased the capillary rise in two of the soils examined, whilst calcium oxide in drab clay and potassium phosphate in silt loam tended to increase the capillary rise. In a silt loam treated with sodium chloride, potassium phosphate, or calcium oxide there was a pronounced tendency for an increase in the water content of the top 8 in. of soil, and soil treated with potassium phosphate or calcium oxide contained more water even in the top inch than the untreated soil, although evaporation was decreased from the treated soil.—W. G.

Soils; Effect of gypsum on bacterial activities in — T. M. Singh. Soil Sci., 1920, 9, 437—468.

BOTH ammonification and nitrification were decreased by applications of gypsum to the soil, whereas nitrogen-fixation was stimulated. The amounts of nitrogen fixed by *B. radicola* were much greater in soil than in solution cultures.

Gypsum increased the process, the greatest increase occurring with the largest application of gypsum. An application of 1000 lb. of gypsum per acre increased the yield of red clover, smaller dressings having, however, no effect on either wheat or leguminous crops. The nitrogen and potassium content of the leguminous crops were not affected by gypsum except in the case of red clover, where there was a very large increase in the potassium content. The applications of gypsum increased the acidity of the soil. The addition of lime as well as gypsum to the soil resulted, in some respects, in a better effect than with lime alone, whilst in others a poorer result was obtained. Thus nitrification was better but not ammonification.—W. G.

Soil; Causes of the injurious effect of potassium and sodium salts on the structure of the —. G. Hager. *J. Landw.*, 1920, 68, 73—105.

RELATIVELY small additions of sodium chloride depress considerably the permeability of soil, the turbidity of the drainage water indicating the occurrence of a peptisation of the highly disperse clay which is to be attributed to electrification of the soil particles owing to interchange of ions. The effects of the liberal application of potassium salts to heavy soils are similarly explained.—T. H. P.

Humic soils; Estimation of the acidity of —. S. Odén. *Int. Mitteil. Bodenkunde*, 1919, 9, 361—374. *Chem. Zentr.*, 1920, 91, IV., 285.

AN estimation of the total acidity of a soil does not show the amount of humic acid, as a number of other acids are also present. The hydrogen-ion content of a soil is first determined by the potentiometer method; concentrated potassium chloride is then added to the test portion of the soil, and the hydrogen-ion content is again measured. The acids in the filtrate are also estimated by titration, and the total acidity and acidity are determined by the method of Tacke or Tacke-Süchting (*J.*, 1908, 171), whereby an indication of percentage of acids present which readily form only slightly soluble calcium salts is obtained.—L. A. C.

Sulphur; Relation of — to soil acidity and to the control of potato scab. W. H. Martin. *Soil Sci.*, 1920, 9, 393—403.

WITH all the different amounts of sulphur applied (300—1200 lb. per acre) there was an increase in soil acidity as measured by the hydrogen-ion concentration, and this was accompanied by a decided increase in the number of clean tubers. In no case, however, was scab entirely eliminated. The amount of sulphur which gave the most satisfactory results depended to some extent on the hydrogen-ion concentration of the water extract of the original soil. Where this was $p_H = 5.8$ or less, the lighter applications (300—500 lb. per acre) gave approximately as good results as the heavier applications (700—1200 lb. per acre).—W. G.

Titanium; Distribution of — in soils and plants. Geilmann. *J. Landw.*, 1920, 68, 107—121.

SOILS contain from traces to 1% of TiO_2 , the usual amount being 0.3—0.6%. Nearly all of a number of plants examined were found to contain titanium, mostly in the green parts. Plant ashes contain from traces to 0.27% of TiO_2 .—T. H. P.

Fertilisers; Carriers of nitrogen in —. C. E. Thorne. *Soil Sci.*, 1920, 9, 487—491.

RESULTS of field trials extending over periods of 16—25 years show that, with only two exceptions in the forty comparisons, sodium nitrate has produced the largest yield, except on limed land, where the yields from ammonium sulphate slightly exceeded those from the sodium nitrate. The other forms in which nitrogen was supplied for com-

parison were linseed oil meal, dried blood, and tankage.—W. G.

Manures; Investigation of the action of —. O. Lemmermann. *Biedermann's Zentr.*, 1920, 49, 291—310.

A CONTINUOUS series of experiments with nitrogenous, phosphatic, potash, and lime fertilisers and stable manure showed that generally the fertilisers had at first but little effect on the crop yields, the exception being the effect of the nitrogenous manures.—W. P. S.

PATENTS.

Soils; Composition for artificially inducing mould action in —. R. W. Parramore. U.S.P. 1,347,401, 20.7.20. Appl., 11.8.19.

A VEGETABLE meal made from dried and ground leaves and stems of green or immature plants is mixed with ground inorganic fertilising material. J. H. L.

Fertiliser, and process for making same. J. N. Carothers, Assr. to N. D. Baker. U.S.P. 1,350,591, 24.8.20. Appl., 14.1.20.

CALCIUM phosphate is treated with nitric acid containing more than 40% HNO_3 , after which sufficient calcium cyanamide containing free lime is added to convert the monocalcium phosphate into dicalcium phosphate.—W. J. W.

Atmosphere surrounding plants; Process for enriching with carbon dioxide the —. F. Riedel. G.P. 312,793, 14.5.16.

WASTE gases from furnaces and the like are purified by removal of dust, soot, oil, tar, sulphur compounds, etc., and led into the atmosphere in the neighbourhood of plants.—L. A. C.

Seeds; Mixtures for treating —. Chem. Fabr. L. Meyer. G.P. 312,797, 17.8.18.

THE germinating power of the seeds is increased by the addition of mercury or silver salts, e.g., mercuric chloride, to the usual mixtures containing tar or tar-oils used for treating them.—L. A. C.

Fertilisers; Nitrogenous —. Norsk Hydro-Elektrisk Kvaestofaktieselskab. E.P. 139,462, 9.2.20. Conv., 24.2.19.

SEE U.S.P. 1,340,708 of 1920; J., 1920, 498A.

XVII.—SUGARS; STARCHES; GUMS.

Cane juice and syrups; Relative importance of some colouring matters in sugar —. F. W. Zerbau. *J. Ind. Eng. Chem.*, 1920, 12, 744—751. (*Cf. J.*, 1918, 778A.)

THE dark colour of sugar cane juice appears to be due almost entirely to the presence of several polyphenol derivatives; when ferric salts are present, as is almost always the case with factory products, the colour is two or three times as dark as with polyphenols alone. A tannin, which gives a green colour with ferric salts, is present, but its influence on the colour of the juice is supplemented by that of the polyphenol derivatives, e.g., anthocyanin and saccharatin.—W. P. S.

Sugar; Prevention of the deterioration of raw — by the use of superheated steam in the centrifugals. N. Kopeloff. *J. Ind. Eng. Chem.*, 1920, 12, 860—862.

MASSECUITE inoculated with micro-organisms known to be active in causing the deterioration of raw sugar (*J.*, 1918, 275A; 1919, 539A) was centrifuged and treated with superheated steam while still in the machine, the temperature of the product thus being raised to 65°—70° C. A diminution of 99.5%

of the bacteria, and 98.3% of the moulds, originally present in the massecuite was thus effected; while in the case of the separated molasses the figures were 84.3 and 50.0% respectively. (Cf. Shorey, J., 1898, 555.)—J. P. O.

Sugars; Iodometric estimation of — H. M. Judd. *Biochem. J.*, 1920, 14, 255—262.

THE polarimetric method is unreliable for the estimation of levulose in fruit juices. The iodometric methods of Colin and Liévin (J., 1918, 745 A) and of Willstätter and Schudel (J., 1918, 556 A) for the determination of dextrose in presence of levulose do not give accurate results, since only about 95% of the dextrose is oxidised, whilst the levulose is definitely attacked. It has been found, however, that a given weight of dextrose or levulose always reacts with a definite weight of iodine; in neither case is the amount affected by the presence of other sugars or by the concentration of the alkali, and the iodine equivalents of mixtures calculated from the constants agree with the observed values. Willstätter and Schudel's method may therefore be used for the analysis of mixtures of dextrose and levulose if the results be interpreted from the calculated iodine equivalents and the copper-reducing power of the mixture. The iodine equivalents of 1 g. of each of the following sugars has been calculated: Dextrose, 1.315 g.; levulose, 0.1028; mannose, 0.977; galactose, 1.418; arabinose, 1.702; rhamnose, 0.9243; lactose, 1.502; maltose, 0.7456 g. Sucrose is not oxidised by iodine under the conditions named.—J. C. D.

Saccharimeters; Testing of — by means of the telescopic control tube. C. A. Browne. *J. Ind. Eng. Chem.*, 1920, 12, 792—796.

THE control tube consists of a telescopic metal tube which can be adjusted by a screw to give a column of solution of any length between 225 and 410 mm. By means of a vernier the exact length of solution at any point within this range can be read on the scale to 0.1 mm. The tube may be used for determining the errors of saccharimeter scales, for comparing the scales of different instruments and their comparative accuracy, for determining the probable error of those who use the instrument, and for determining the influence of personal equation in the observations. The average probable error for a single reading upon the Schmidt-Haensch and Fric saccharimeters is found to be ± 0.0514 and ± 0.0286 , respectively. The author confirms the opinion of Landolt that the quartz-wedge saccharimeter contains inherent defects which prevent it from becoming an instrument of the highest precision.—W. P. S.

Carbonatation sludge cakes [in sugar factories]; Composition of — and new means for their more efficient washing. A. Müller. *Z. Zuckerind. Czechoslov.*, 1920, 44, 374—376.

IN filter-presses operated by a cylinder pump the fluctuations of pressure due to the stroke of the pump necessitate the use of stronger and denser filter cloths than would be required with a uniform pressure, and also facilitate the deposition of sand and coarse particles, which consequently form a relatively permeable layer at the bottom of the filter chambers, and render difficult the effective washing of the upper part of the cakes. By means of a combined pneumatic shock absorber and sand remover devised by the author, which is mounted on the suction or pressure side of the pump, and utilises part of the pressure pulse to effect a rotary motion of the liquid, sand may be removed centrifugally. The relatively great permeability of the layer of sand and coarse particles at the bottom of the filtering chambers may also be counteracted by increasing the resistance of the bottom of the filters, e.g., by means of a strip of non-perforated

or finely perforated metal, or by sewing a strip of denser cloth along the bottom of each filter cloth.

—J. H. L.

Fructose [levulose]; Specific rotation of —. W. C. Vosburgh. *J. Amer. Chem. Soc.*, 1920, 42, 1696—1704.

THE specific rotation of levulose solutions over the concentration range 2.6—20 g. per 100 c.c. is given by $[\alpha]_D^{25} = -(88.50 + 0.145p)$ or $[\alpha]_D^{25} = -(88.50 + 0.150c - 0.00086c^2)$, where p and c represent the concentration of the sugar in % and g. per 100 c.c. respectively. Over the temperature range 15°—37° C. the variation of $[\alpha]_D$ with temperature is given by $[\alpha]_D^t = [\alpha]_D^{25} + (0.566 + 0.0028c)(t - 25)$. For most purposes the rotations with the D line may be converted into those for the line $\lambda = 546\mu$ by $[\alpha]_D^t \lambda = 1.1809[\alpha]_D^t$.—W. G.

Fat associated with starch. T. C. Taylor and J. M. Nelson. *J. Amer. Chem. Soc.*, 1920, 42, 1726—1738.

CORN (maize) starch, carefully freed by extraction from all adherent or associated fat, gave on hydrolysis about 0.56% of "fat by hydrolysis," the two main constituents of which were palmitic acid and an unsaturated compound of unknown constitution. This fat is apparently liberated when the hydrolysis has reached the erythrodextrin stage. It was obtained in considerable amount from the "refinery mud" in the manufacture of dextrose from starch. Starches of other vegetable origin, e.g., from rice, sago, potato, etc., contained amounts of this fat varying from 0.04 to 0.83%.

—W. G.

Levan; Formation of — by mould spores. N. Kopeloff, L. Kopeloff, and C. J. Welcome. *J. Biol. Chem.*, 1920, 43, 171—187.

SPORES of the mould, *Aspergillus sydowi*, form levan in sucrose media. The sucrose is hydrolysed before utilisation and the levulose and dextrose are utilised, the former to the greater extent, in formation of the gum. It appears necessary that the sugars be present in a "nascent" condition for the synthesis to be possible, as little gum formation occurs in media containing reducing sugars from ordinary sources. The synthesis by the enzyme, levanase, is influenced by the reaction of the medium, and has its optimum at about $pH = 7.0$. Levan yields levulose on hydrolysis. (Cf. J.C.S., Oct.)—J. C. D.

Glutamic acid. Skola. See XX.

Alkalimetric titrations. Bruhns. See XXIII.

PATENTS.

Sugar juices and the like; Treatment of —. J. N. A. Sauer. G.P. 322,135, 6.3.19.

THE juices are first subjected to preliminary treatment with a char produced by heating cane bagasse or exhausted beet slices in closed retorts, and then treated with finely powdered decolorising carbon. The latter may be produced from the above-mentioned char by treatment with hot gases in absence of air, or it may be produced directly from cane bagasse or exhausted beet slices by heating in absence of air and simultaneously conducting hot gases through the material. The preliminary treatment of the juices effects a saving in decolorising carbon and facilitates the regeneration of the latter.—J. H. L.

Diffusion process [for extraction of sugar, tannin, etc.]. R. Farkas. G.P. 320,688, 25.5.17.

THE exhaustion of the material in the last unit of a diffusion battery is rendered more complete by introducing the total quantity of fresh solvent for this unit in portions at intervals instead of all at

one time. The corresponding portions of liquor drawn off are collected in a separate receptacle and afterwards introduced together into the preceding unit in the ordinary way.—J. H. L.

Syrup or artificial honey; Preparation of — from crude sugar solutions. W. Meyer. G.P. 319,841, 21.3.18.

CRUDE sugar solutions are inverted by evaporating in acid medium to a high density, and are then diluted, neutralised, filtered, and concentrated: or a strong inverted solution of relatively pure sugar may be mixed with very impure sugar solutions, such as molasses, previous to neutralising and filtering.—G. F. M.

XVIII.—FERMENTATION INDUSTRIES.

Yeast; Degeneration and treatment of —. R. Heuss. Z. ges. Brauw., 1920, 43, 225—227, 233—234, 241—242.

A **BOTTOM-FERMENTATION** brewery yeast used in the production of dark, 4·5% war beers, was much improved by treatment with five times its volume of water containing 0·12% of free potassium carbonate for 15 hrs. at about 4° C., followed by two washings with cold water. Compared with the same yeast washed only with water, the treated yeast was much lighter in colour, and its flavour was mild and free from bitterness. In the first fermentation after the treatment it produced a slightly less rapid attenuation than the control yeast, but in subsequent fermentations this difference was reversed. The break or flocculation of the treated yeast occurred at a later stage of fermentation than in the case of the control yeast. The beers produced were milder in flavour but possessed less palateness than those produced by the control yeast. —J. H. L.

Yeast. Nitrogen metabolism in S. cerevisiae. L. H. Lampitt. Biochem. J., 1919, 13, 459—486.

EXCESS of yeast ensures the removal of the greatest total amount of nitrogen. During active fermentation the greater the coefficient of multiplication the greater the amount of nitrogen assimilated by each cell. Active reproduction may result in a lowering of the nitrogen coefficient, even when a large amount of available nitrogen is present. The final nitrogen coefficient of a yeast is independent of the initial coefficient, and tends to reach a value constant for any particular conditions of reproduction. The amidase of Effront (this J., 1908, 462) does not produce solely a volatile acid by its action on asparagine, but also a non-volatile acid, probably malic acid. If this is so, the acid is probably converted into the ammonium salt before it is fermented to alcohol, for the free acid is not fermented. Propionic acid is not fermented by yeast. Fermentative activity is essential to nitrogen assimilation. The two actions are not proportionate, but the former stimulates the latter, and, once induced, desamination may continue after zymatic activity ceases. Excessive zymatic activity does not ensure rapid nitrogen assimilation. During fermentation yeast continually loses nitrogen to the liquid, and this action has been called "nitrogen excretion." This excretion is dependent on the life of the cell, and takes place even when nitrogen is being assimilated. The substances so secreted can be used by the yeast as a source of nitrogen under suitable conditions. Increase in the amount of sugar available for fermentation increases the rate of excretion, and although zymatic activity is necessary for nitrogen excretion, the two are not proportionate, nor does the cessation of the former result in an immediate cessation of the latter.—J. C. D.

Fermentation; Cold —. G. Fries and R. Heuss. Z. ges. Brauw., 1920, 43, 249—252, 257—260.

HEALTHY bottom-fermentation yeast which had been subjected to alkaline treatment (see preceding abstract) was employed in large scale fermentations of 4% and 10% worts at temperatures of 3°—4° C., instead of the usual temperatures of bottom fermentation, viz., 4°—10° C. The results were unsatisfactory; fermentation occupied 1—3 days longer than usual, and although relatively large amounts of yeast were used for pitching, the yeast crops obtained were very small. Moufang's statement that low fermentation temperatures improve the character of the beer produced was not confirmed. In the alkaline treatment of the yeast losses amounting to about 40% were incurred (cf. loc. cit.)—J. H. L.

Acidity [of worts]; Determination of — and titration in stages. H. Lüers. Z. ges. Brauw., 1920, 43, 252—253.

A MODIFICATION of Reichard's method of titration in stages (J., 1917, 399; 1918, 523 A) is described, in which "neutral red" is used instead of litmus paper as the first indicator, in order to enable both stages of the titration to be carried out in the apparatus previously described by the author (J., 1914, 761). The method, as applied to coloured liquids like wort, is thereby rendered much more precise. The colour standard used for the first titration is a permanent dye solution having the same colour as a Sørensen phosphate solution containing 6·5 pts. of secondary to 3·5 pts. of primary phosphate to which 1/10 of its volume of a 0·02% solution of "neutral red" in 50% alcohol has been added. This phosphate solution has $p_H=7·07$ (absolute neutrality), which differs only slightly from the transition reaction for litmus, $p_H=6·8$. The first titration is carried out in an analogous manner to that described for phenolphthalein (*ibid.*), the left hand sample of wort receiving 1/10 of its volume of 0·02% "neutral red" solution. After the first titration has been completed it is necessary to add a similar quantity of "neutral red" to the right-hand sample of wort, since this indicator retains a yellow colour in alkaline solutions. The colour standard is then replaced by the phenolphthalein colour standard (*ibid.*), the left-hand sample of wort is treated with 1/10 of its volume of 0·05% phenolphthalein solution, and the second titration is proceeded with until the end-point ($p_H=9·18$) is attained.—J. H. L.

Wines; Glycerol in —. L. Mathieu. Bull. Assoc. Chim. Sucr., 1920, 37, 423—425.

THE quantities of sulphurous acid commonly employed in vinification were found to have no perceptible influence on the formation of glycerol. Reduction of the acidity of a must by addition of potassium carbonate appreciably reduced the production of glycerol during fermentation, whilst an excess of carbonate equivalent to 0·65% of sulphuric acid slightly increased the yield of glycerol. —J. H. L.

Wine; Analysis of —. W. Fresenius and L. Grünhut. Z. anal. Chem., 1920, 59, 209—232. (Cf. J., 1920, 555 A).

METHODS are given in detail for the determination of ash, total alkalinity of ash, alkalinity of soluble ash, and phosphoric acid.—W. P. S.

Volatile acids; Determination of — in fermentation products. G. Hinard. Ann. Chim. Analyt., 1920, 2, 239—242.

FIFTY c.c. of the sample is mixed with 10 c.c. of water and distilled, 50 c.c. of distillate being collected and titrated; 50 c.c. of water is then added to the residue in the distillation flask, the mixture distilled as before, and these operations are

repeated several times. When the results are plotted graphically, using the volumes of the distillates (50 c.c.) as abscissæ and the quantities of standard alkali solution used for the successive titrations as ordinates, the asymptote of the curve indicates the volume of alkali solution necessary to neutralise the amount of volatile acids contained in the 50 c.c. of the sample.—W. P. S.

Alcohol; Losses of — in distilleries. *Methods of recovery.* Mariller. Bull. Assoc. Chim. Sucr., 1920, 37, 412—423.

THE alcohol entrained by fermentation gases from closed or open vats may be recovered by using cresol as absorbent (*cf.* Brégeat, E.P. 128,640 and 131,938, J., 1919, 612A, 751A). Cresol may also be used for the absorption of volatile products from the distillation of wood, peat, lignite, etc. It has been successfully applied to the recovery of alcohol and ether in perfumery.—J. H. L.

Yeast growth-promoting stimulus. Emmett and Stockholm. See XIXA.

PATENTS.

Glycerol; Manufacture of — from sugar. Verein. Chem. Werke A.-G. E.P. 138,331, 21.1.20. Conv., 18.6.17. Addn. to 138,099.

IN the process claimed in the chief patent and in E.P. 138,328 (J., 1920, 608A), when the fermentation has attained its height a further quantity of sugar is added, if necessary, together with a small proportion of yeast and alkaline compounds, preferably sulphites, with or without other salts. This effects a considerable saving in respect of yeast, sulphite, and time.—J. H. L.

Spirits and alcoholic liquors; Process and apparatus for ageing —. A. Jarraud. E.P. 141,687, 31.3.20. Conv., 17.4.19.

THE liquid is subjected to electrolysis by means of an anode of non-oxidisable material and a depolarising cathode (*e.g.*, a carbon rod surrounded by manganese dioxide in a porous cell), with a current-density of 0.5—1 milliampère-hr. per sq. cm. of anode surface. If necessary, oak shavings may be interposed in the path of the current in the neighbourhood of the anode. For the treatment of spirits and the like, in new casks, a tubular cathode may be inserted through the bung-hole, and the anode may consist of sheets of metallic foil secured to the outer surface of the cask. Pads of moist cloth may be interposed between the foil and the wood to reduce the resistance of the latter.

J. H. L.

Fat; Production of mould fungi of all kinds rich in —. Reichsausschuss für pflanzliche und tierische Öle und Fette, G.m.b.H. G.P. 320,560, 2.9.17. (*Cf.* J., 1920, 417A.)

IN the production of fat by cultivation of mould fungi in nutrient liquids rich in sugar but of low nitrogen-content, the mould cells are maintained thinly distributed throughout the liquid (*e.g.*, corresponding to a "pitching" of 20 g. of moist pressed mould per litre), local accumulation of cells being avoided. Yeast containing 40—50% of fat may be produced.—J. H. L.

Fat from sulphite-cellulose lyes. G.P. 305,091. See XII.

Calamus root. G. P. 307,623. See XX.

XIXA. FOODS.

Wheat flour; Heat of hydration and specific heat of —. F. Daniels, B. H. Kepner, and P. P. Murdick. J. Ind. Eng. Chem., 1920, 12, 760—763.

THE heat of hydration of various Canadian and American wheat flours ranged from 5.4 to 7.6

B.Th.U. per lb.; flour generally used for bread-making gave a value of 6.5 B.Th.U. per lb.; the heat of hydration decreased when the flour was exposed to the atmosphere, but did not change appreciably with age when the flour was isolated completely from air. The specific heat of flour was found to be 0.43.—W. P. S.

Butter and certain allied products; Volumetric method for the detection and determination of neutralisers in —. L. W. Ferris. J. Ind. Eng. Chem., 1920, 12, 757—759.

THE greater part of the fat is separated from 100 g. of butter by treatment with petroleum spirit and centrifugal action, the aqueous portion is mixed with a known amount of *N*/10 hydrochloric acid and an excess of picric acid solution, filtered, the lactic acid and excess of picric acid are extracted with ether, and the residual solution is titrated with *N*/10 sodium hydroxide solution, using methyl orange as indicator. The difference between the volume of acid added and that of the alkali solution used for the neutralisation will be a measure of the alkalinity of the salts or the amount of acid that has combined with the bases formerly present as alkalis, lactates, and phosphates. The phosphoric acid is then determined in the neutralised solution. In normal milk and cream the alkalinity of the salts bears a definite ratio to the inorganic phosphoric acid, being about 45 c.c. of *N*/1 solution to 1 g. P_2O_5 ; in neutralised cream this ratio is increased to a degree dependent on the amount of alkali added and the P_2O_5 content of the sample. Butter made from neutralised cream has an alkalinity ratio similar to that of the cream from which it was made. If, however, the acidity of the cream is reduced to less than 0.3% it is not sufficient to keep all the phosphate in solution, and the butter may then have a lower alkalinity ratio than the neutralised cream.—W. P. S.

Eggs; Preservation of —, including a bibliography of the subject. H. I. Jones and R. Du Bois. J. Ind. Eng. Chem., 1920, 12, 751—757.

COLD storage of eggs is successful only when the surrounding atmosphere is dry and the eggs are kept dry; low temperature merely inhibits bacterial etc. growth and is not germicidal. The most satisfactory method consists in coating the eggs, and for this purpose a solution of aluminium soap in pure pentane is recommended; if gasoline is used in place of pentane the eggs must be treated previously with dilute sulphuric acid to prevent penetration of the objectionable odour and taste of the gasoline.—W. P. S.

Caseins; Behaviour of different — as regards their increase in viscosity [when heated]. C. Chorower. Chem.-Zeit., 1920, 44, 605—606, 613—614.

WHEN cows' milk is evaporated under reduced pressure, as in the manufacture of condensed milk, and then maintained at about 58° C. for 35—40 mins., the viscosity shows an increase during this heating period of three to four times the value at the commencement. This increase is a function of the temperature, time of heating, and concentration, and is due to changes in the casein. The other constituents of the milk do not appreciably alter in viscosity. The casein of goats' milk, unlike that of cows' milk, does not increase in viscosity when its concentrated solution is subjected to prolonged heating, and admixture of goats' milk with cows' milk reduces the viscosity of the finished condensed product.—W. P. S.

Tyrosine; Colorimetric estimation of — by the method of Folin and Denis. R. A. Gortner and G. E. Holm. J. Amer. Chem. Soc., 1920, 42, 1678—1692.

THE phenol reagent of Folin and Denis (J. Biol.

Chem., 1912, 12, 245) cannot be used for the estimation of tyrosine in protein hydrolysates. Tryptophane (and probably other products of protein hydrolysis), if present, will give a blue colour with the reagent. Indole and its derivatives and ferrous salts also give a blue colour. The intensity of the blue colour produced by tyrosine varies with the relative amounts of tyrosine and reagent present. Protein hydrolysates must not be decolorised by carbon or bone black if they are to be used subsequently for the estimation of amino-acid content, as tyrosine, tryptophane and its decomposition products are adsorbed in appreciable amounts by bone black. Bone black itself contains some easily oxidisable material which dissolves in acid solutions and then gives a blue colour with the phenol reagent.—W. G.

Inulin in the globe artichoke. R. Okey and A. W. Williams. J. Amer. Chem. Soc., 1920, 42, 1693—1696.

In a sample of globe artichokes which contained 6—8% of nitrogen-free extractives as measured by difference after the estimation of moisture, ash, crude fibre, crude protein, and ether extract, the authors found probable inulin, 2.5% and total carbohydrate extracted by water and 1% hydrochloric acid, 4.2%.—W. G.

Vitamines; Water-soluble, anti-neuritic, and growth-promoting B — A. D. Emmett and G. O. Luros. J. Biol. Chem., 1920, 43, 265—286.

The authors' results tend to support the theory that these substances are not identical. (Cf. J.C.S., Oct.)—J. C. D.

Vitamines; Relation of water-soluble, anti-neuritic, and water-soluble B — to yeast growth-promoting stimulus. A. D. Emmett and M. Stockholm. J. Biol. Chem., 1920, 43, 287—291.

FURTHER evidence is given that vitamin B and the anti-neuritic factor are not identical. Possibly the substance stimulating the growth of yeast is also distinct.—J. C. D.

Vitamine content; Studies in — W. H. Eddy and H. C. Stevenson. J. Biol. Chem., 1920, 43, 295—309.

THE authors propose a modification of the processes devised by Bachmann and by Williams (J., 1920, 608A) for the quantitative estimation of extracts containing the vitamin B, and show that its use may throw light upon the relative values of food-stuffs as sources of this vitamin.—J. C. D.

Albumoses. Nagayama. Sec XX.

PATENTS

Milk products; Manufacture of artificial — N. V. A. Jurgens' Vereen. Fabrieken. E.P. 128,544, 23.4.19. Conv., 18.0.18.

ARTIFICIAL milk or cream possessing the natural flavour is prepared by emulsifying vegetable or animal fats or fatty mixtures in skim milk or an artificial albuminous solution the acidity of which has been reduced, by partial neutralisation, to not more than 5° Soxhlet-Henkel (equivalent to 5 c.c. of N/4 alkali per 100 c.c.). Pure cultures of bacteria or other substances may be added to improve the flavour.—J. H. L.

Protein and phosphates of calcium and magnesium; Process for recovering — from acid waste waters. A. Giesecke. U.S.P. 1,348,990, 10.8.20. Appl., 10.3.19.

ACID waste water from corn steeping is neutralised partially with milk-of-lime in order to precipitate a mixture of proteins and calcium and magnesium phosphates.—W. P. S

Jam; Preparation of — from fruit or other vegetable constituents. O. and C. Biemann. G.P. 313,768, 9.8.18. Addn. to 303,995 (J., 1920, 525A).

THE solution of sugar and fruit juice obtained from the diffusion vessel freshly filled with fruit is heated and further sugar is added to bring the concentration up to that of the original sugar solution employed. The solution is then returned to the diffusion vessel containing the extracted fruit instead of fresh sugar solution.—G. F. M.

Syrups and jellies from fruits or other vegetable products; Preparation of — O. and C. Biemann. G.P. 318,266, 9.8.18. Addn. to 303,995 (J., 1920, 525A).

SYRUPS containing a definite proportion of sugar are obtained by passing the juice coming from a diffusion battery through a vessel containing coarse sugar crystals at a definite speed or temperature or both.—C. A. M.

Lupins; Production of a feeding stuff rich in proteins from — H. Thoms and H. Michaelis. G.P. 320,559, 7.9.17. Addn. to G.P. 307,007 (J., 1919, 597A).

THE treatment with alkali precedes extraction of the bitter substances with alcohol.—J. H. L.

Fruit juices; Process of treating — J. G. F. Hieber. E.P. 149,537, 17.9.19.

SEE U.S.P. 1,325,094 of 1919; J., 1920, 170A.

Wheat and other cereals and their products; Apparatus for separating and purifying — J. Higginbottom. E.P. 149,020, 10.10.18 and 9.4.19.

Boiling pans for manufacture of confectionery; Installation of — R. S. and T. N. McColl and T. F. Dryden. E.P. 149,172, 9.12.19.

XIXB.—WATER PURIFICATION; SANITATION.

Cyanogen chloride; Mechanism of the toxic action of — C. I. Reed. J. Pharm. Exp. Ther., 1920, 15, 301—304.

THE symptoms following poisoning with cyanogen chloride are typical of poisoning with hydrocyanic acid. Sodium thiosulphate exerts a protective action in both cases by the formation of non-toxic sodium thiocyanate. In cases of delayed poisoning by cyanogen chloride it is possible that the toxic action of the chlorine also plays a part.—J. C. D.

Chloropierin; Destruction of white ants by — J. Feytaud. Comptes rend., 1920, 171, 440—442.

EXPOSURE at 20° C. for 12 hrs. to an atmosphere containing 2 mg. of chloropierin per l., or for 6 hrs. in an atmosphere containing 5 mg., is sufficient to destroy white ants (*Leucotermes lucifugus*, Rossi) either freely exposed or in fragments of wood 10 cm. thick.—W. G.

PATENTS.

Water-softening compound and method of producing same. G. L. Borrowman. U.S.P. 1,348,977, 10.8.20. Appl., 2.6.16. Renewed, 8.5.19.

A WATER-SOFTENING material consists of a "natural base exchange silicate" stabilised by baking to render it capable of resisting the powdering action of flowing water.—L. A. C

Water purified with chlorine; Removing iron from —. Th. Goldschmidt A.-G. G.P. 319,963, 31.12.13.

APPRECIABLE amounts of iron are retained in a colloidal form by water after the iron treatment following chlorine purification. The iron can be removed by passing the water through a sand filter.—W. J. W.

Boiler-feed water; Process of removing gases from, softening, and preheating —. J. P. Winer. G.P. 320,423, 1.12.16. Addn. to 302,643 (J., 1918, 440 A).

The feed water from the nozzles is sprayed into a part of the receptacle in the upper part of the boiler, separated by a partition, and from this it passes through the external tubular extension to the lower part of the boiler.—W. J. W.

Liquids; Purification of — by means of colloidal silicic acid. O. Biemann. G.P. 320,846, 22.11.16.

LIQUIDS are treated with silicic acid prepared by passing carbon dioxide into dilute sodium or potassium silicate solution and filtering. The colloidal silicic acid obtained may be purified before use by means of water containing carbon dioxide in solution, the product being absolutely tasteless and without smell.—W. J. W.

Sterilising liquids by means of chlorine; Process and apparatus for —. E. Volland. G.P. 305,888, 14.7.15.

THE liquid to be sterilised, supersaturated with chlorine, is transferred in a fine spray to a vessel in which a vacuum is maintained, and is thereby freed from the excess of chlorine.—G. F. M.

Sterilising agent; Preparation of a powerful solid —. A. Pieroni, and Soc. Chim. Lombarda A. E. Bianchi & Co. G.P. 320,795, 9.1.17. Conv., 5.1.16.

A CHLORATE is mixed in the cold in a closed vessel, under pressure, with chlorine and iodine, or materials capable of giving iodine under the action of chlorine and an acid. The reaction, which is carried out in the dark, or in red light, is assisted, if necessary, by heating. The product is crystalline, and probably has the composition KIO_2 . —G. F. M.

Refuse; Treatment of — in refuse destructors and the production and treatment of a fluid slag obtained therefrom. J. B. C. Kershaw. E.P. 149,033, 11.10.19.

A SLAG which is fluid at 1260° – 1370° C. is produced in refuse destructors by mixing the refuse with limestone, chemical waste, road sweepings or other material containing lime, so that the resulting slag contains 40% SiO_2 and 15% $\text{CaO} + \text{MgO}$. If necessary, slack, coke or other combustible material may also be added, and an air-blast may be used so as to increase the temperature in the destructor. A jet of steam may be allowed to play on the stream of molten slag issuing from the destructor so as to form a fine amorphous powder which is ready for use as an aggregate for concrete or mortar. Alternatively, the molten slag may be run into moulds so as to form blocks for building purposes.—A. B. S.

Waste liquors. G.P. 322,461. See V.

Soapy liquors. U.S.P. 1,349,530. See XII.

Treating seeds. G.P. 321,797. See XVI.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Glucosides; Extraction of — from two indigenous orchids; identification of these glucosides with loroglossin. P. Delauney. Comptes rend., 1920, 171, 435–437.

A CRYSTALLINE glucoside obtained from *Orchis Simin*, Lam., and *Ophrys aranifera*, Huds., is shown to be identical with loroglossin obtained from *Loroglossum hircinum*, Rich., by Bourquelot and Bridel (J., 1919, 337 A).—W. G.

Spilanthal, the pungent principle of Para cress (Spilonthes oleracea). Y. Asahina and M. Asano. J. Pharm. Soc., Japan, 1920, 503–515.

CRUDE spilanthal was prepared (yield ca. 1%) from air-dried flower-heads of the plant by Gerber's method (Arch. Pharm., 1903, 241, 270). The base, $\text{C}_8\text{H}_{11}\text{N}$, formed by heating it with alcoholic hydrogen chloride, proved to be isobutylamine. Hydrosplanthal, prepared by hydrogenating crude spilanthal in acetic acid solution in presence of platinum black, is a colourless viscous liquid which solidifies to crystals, m.p. 28° C. It is probably a mixture of two substances, $\text{C}_{13}\text{H}_{27}\text{NO}$ and $\text{C}_{11}\text{H}_{23}\text{NO}$. When heated with alcoholic hydrogen chloride in a sealed tube it yields isobutylamine and a saturated fatty acid, m.p. 28° C., and b.p. 136° – 140° C. at 4 mm; probably a mixture of decolic acid, m.p. 31.5° , and nonoic acid, m.p. 12.5° C.—K. K.

Viburnum prunifolium; Some constituents of —. F. W. Heyl and C. Barkenbus. J. Amer. Chem. Soc., 1920, 42, 1744–1755.

THE dried root bark of *Viburnum prunifolium* ("black haw") contained moisture 7.1%, ash 7.3%, ligroin extract 7.1%, ether ext. 10.4%, and alcohol extract 18.7%. Acetic and valeric acids were present in the free state, as water-soluble complex esters, and as resinous esters insoluble in water. From the resin insoluble in water a new phyto-sterol, m.p., 186° – 187° C.; $[\alpha]_D^{20} = +115^\circ$ (in chloroform) was isolated, giving an acetate, m.p., 223° – 224° C. The fat from this resin contained formic, acetic, caproic, caprylic, myristic, palmitic, oleic, and linolic acids, but no valeric acid. A trace of an amorphous alkaloid was found in the alcoholic extract of the drug.—W. G.

Pseudo-muscarine ("synthetic muscarine"). A. B. Weinhausen. J. Amer. Chem. Soc., 1920, 42, 1670–1678.

THE products obtained in the preparation of "synthetic muscarine" by the action of nitric acid on choline platinichloride vary considerably with the conditions. If the original directions of Schmeideberg and Harnack (Arch. exp. Path. Pharm., 1877, 6, 101) are adhered to the main product is choline nitrate platinichloride. (Cf. J.C.S., Oct.).—W. G.

Ferrous zymophosphate. K. Schweizer. Bull. Assoc. Chim. Sucr., 1920, 37, 464–468.

FOLLOWING Euler's method (cf. F.P. 458,096; J., 1913, 1031) for the preparation of salts of hexose-phosphoric acid, the author obtained a ferrous compound in the form of a greyish-green amorphous powder, tasteless and odourless, and insoluble in water and ordinary organic solvents but decomposed by mineral acids or sodium hydroxide. Administered to guinea-pigs it was in part decomposed in the stomach, but the undecomposed portion passed through the intestines unchanged.

—J. H. L.

Phytic acid; Synthesis of — R. J. Anderson. *J. Biol. Chem.*, 1920, 43,, 117—128.

THE product obtained by interaction of inositol, phosphoric acid, and phosphorus pentoxide appears to be an inositoltetraphosphoric acid, and is not identical with natural phytic acid or inositolhexaphosphoric acid.—J. C. D.

Adrenaline; Colorimetric determination of — W. L. Scoville. *J. Ind. Eng. Chem.*, 1920, 12, 769—771.

A Mixture of water, 20, 1% potassium iodate solution, 5, and N/1 hydrochloric acid 0.25 c.c., is heated to 38° C. and 0.5 c.c. of the adrenaline solution to be tested (this may be a 0.1% solution) is added. A similar standard mixture is made at the same time with pure adrenaline. Both mixtures are heated at 38° C. for 15 mins., then cooled, and the colorations compared. If the coloration of the test solution differs by more than 25% from that of the standard, the determination should be repeated, using more or less of the test solution. If the test solution contains bisulphite, 0.05 g. of sodium bisulphite must be added to the standard solution, which is prepared by dissolving 0.05 g. of pure adrenaline in 0.5 c.c. of N/1 hydrochloric acid and diluting to 50 c.c. The results obtained agree with those yielded by the biological method.—W. P. S.

Phenolphthalein; Iodometric method for the determination of — S. Palkin. *J. Ind. Eng. Chem.*, 1920, 12, 766—769.

UNDER suitable conditions, phenolphthalein when treated with iodine gives a quantitative yield of tetraiodophenolphthalein. The phenolphthalein (not more than 0.25 g.) is dissolved in the minimum amount of 30% potassium hydroxide solution in water, 20 g. of ice is added, and an excess of iodine reagent is introduced; about 10 c.c. is sufficient. (The reagent is prepared by dissolving iodine in 15% potassium iodide solution to make a 10% iodine solution and then discharging the colour of the iodine by the addition of alkali.) The mixture is then treated, drop by drop, with concentrated hydrochloric acid until precipitation is complete, more iodine is added if the supernatant liquid is not brown in colour, the precipitate is dissolved by the addition of 30% potassium hydroxide solution, and the re-precipitation and re-solution are repeated three or four times. The final alkaline solution is treated with 0.5 c.c. of 15% sodium sulphite solution and a few c.c. of hydrochloric acid, and extracted several times with acetone-chloroform (1:3) mixture. The united extracts are evaporated, the residue of tetraiodophenolphthalein thus obtained dried at 100° C. for 20 mins., and weighed; the weight multiplied by 0.3781 gives the amount of phenolphthalein. To determine the latter in tablets which contain chocolate, the sample is ground, extracted for 2 hrs. with petroleum spirit to remove fat, the phenolphthalein then extracted with acetone, the acetone solution evaporated, the residue dissolved in potassium hydroxide solution, and the process continued as described.—W. P. S.

Albumoses; Histamine and a histamine-like substance as decomposition products of — T. Nagayama. *J. Pharm. Exp. Ther.*, 1920, 15, 401—414.

THE proteoses of commerce vary in toxicity. A secondary albumose was prepared from fresh thyroid glands by means of peptic digestion. Under certain conditions hydrolysis with acids causes the appearance of an appreciable quantity of histamine, and also of a histamine-like substance. Attention is called to the large amount of chloro-

form-soluble matter obtained in the hydrolysis of the proteoses studied.—J. C. D.

Aminoacetanilide; Preparation of — A. J. Hill and E. B. Kelsey. *J. Amer. Chem. Soc.*, 1920, 42, 1704—1711. (Cf. Dubsky and Gränacher, *Ber.*, 1917, 50, 1701.)

AMINOACETANILIDE is best prepared by a modification of Majert's process (E.P. 5269 of 1891; *J.*, 1892, 369). 550 g. of chloroacetanilide is added to 11 kg. of 95% alcohol previously saturated with ammonia at 10° C., and the mixture left to stand for 5 days at 20° C. It is then evaporated under diminished pressure to $\frac{1}{4}$ of its volume and poured into 3500 c.c. of water. An oil separates and solidifies. This is the secondary base which is filtered off. The filtrate is evaporated nearly to dryness and the residual material dissolved in hot water. After decolorising, the solution is cooled in ice and saturated with ammonia when aminoacetanilide crystallises out. Satisfactory yields could not be obtained by Majert's second process (*loc. cit.*) from aniline and ethyl aminoacetate hydrochloride.—W. G.

Cinnamic acid; Identification of — Denigès. *Bull. Soc. Pharm. Bordeaux*, 1919. *Ann. Chim. Analyt.*, 1920, 2, 245—246.

A DISTINCT odour of benzaldehyde is obtained when 2 c.c. of cinnamic acid solution (containing as little as 0.02 mg. of the acid) is treated with 1 drop each of ferric chloride solution, 10% sulphuric acid, and hydrogen peroxide solution (2 vol.), and then heated. In the case of cinnamic esters, Peru balsam, etc., a preliminary saponification with a few drops of sodium hydroxide solution is required.—W. P. S.

Glutamic acid and its salts; Decomposition of — on heating. V. Skola. *Z. Zuckerind. Czechoslov.*, 1920, 44, 347—351, 355—360, 363—368, 370—374.

ON recrystallising glutamic acid obtained by Andrlik's method from molasses (*J.*, 1915, 1064), non-crystallisable syrupy mother liquors were obtained, owing to the formation of the corresponding lactam, *l*-glutimic acid (cf. Stanek, *J.*, 1912, 1001). Such syrups were successfully treated by saturating with hydrochloric acid whereby the glutimic acid was converted into the hydrochloride of glutamic acid, which crystallised. The nitrogen of glutamic acid can be determined almost quantitatively by "formol-titration" provided successive additions of formaldehyde are made alternating with neutralisation of the acidity developed by each addition. Glutimic acid scarcely reacts with formaldehyde. (Cf. *J.C.S.*, Oct.)—J. H. L.

Urea; Determination of — by xanthhydrol. Frenkel. *Ann. Chim. Analyt.*, 1920, 2, 234—239.

TEN c.c. of a 10% solution of urino is treated with 35 c.c. of glacial acetic acid and 10% xanthhydrol solution (in methyl alcohol) is added in quantities of 1 c.c. at intervals of 10 mins., until 5 c.c. in all has been added; after 1 hr. the precipitate of dixanthylurea is collected on a tared filter, washed with a small quantity of 95% alcohol, dried at 100° C., and weighed. The weight is divided by 7 to obtain the amount of urea. Other substances (e.g., proteins, sugars, uric acid, etc.) occurring in urine do not interfere. The method may be applied to the determination of urea in blood after this has been treated with a mixture of mercuric chloride, 27, potassium iodide 7.2 g., glacial acetic acid 66, and water 100 c.c., and filtered.—W. P. S.

Chlorocarbonic esters. G. Capelli. *Gaz. Chim. Ital.*, 1920, 50, ii., 8—12.

THESE esters may be prepared in 90% yield by the

following procedure. The phosgene supply is connected, through a washing vessel containing vaseline oil to indicate the rate of flow of the gas, with a 5-l. vessel which acts as a safety vessel and for the condensation of any carbon tetrachloride in the phosgene. This vessel is joined to a 5-l. Woulff's bottle, to which are fitted a graduated funnel containing the alcohol to be esterified and a double surface condenser connected with a draught. The vessels may be joined with short pieces of glass tubing for small preparations, otherwise lead tubing should be used; the joints are best made with thoroughly waxed corks. The reaction vessel is charged with 200 g. of well-dried furnace coke in pieces the size of peas and is immersed in pounded ice, the water forming being removed and replaced by fresh ice. After about 30 mins. phosgene is introduced in a steady stream and is absorbed in considerable amount by the coke, the rate of flow being increased after 20 mins. and the alcohol allowed to fall rapidly in drops. 1 kg. of alcohol may be esterified in 4–5 hrs. After standing in a draught for 20 hrs. to ensure the esterification of all the alcohol and to allow the excess of phosgene to escape, the crude ester is washed with water and ice, dried over calcium chloride and fractionally distilled. The alcohol used need not be absolute, 75% alcohol giving good results. The addition of basic substances is of no advantage.

T. H. P.

Chlorhydrins; Formation of —. L. Smith and E. Sammelsson. *Z. physik. Chem.*, 1920, 94, 691–722.

A 40% yield of perfectly pure α -glycerol monochlorhydrin may be obtained by passing hydrogen chloride into 1 g.-mol. of dry glycerol containing 4 g. of succinic acid at 18° C. until the weight has increased 36 g. The addition of hydrogen chloride should take place in two stages with a pause of a day between them. The best laboratory method for the preparation of an approximately pure (1% β isomer) α -chlorhydrin is to add hydrogen chloride to 1 g.-mol. of glycerol containing 4 g. of succinic acid until the weight has increased 47 g.; the heat of the reaction keeps the temperature at 60°–70° C. After the addition is completed the mixture is kept at the ordinary temperature for a day. If there is no special need for a uniform product, a yield of 65% can be obtained by passing hydrogen chloride into the above mixture at 120° C. until the weight has increased 39 g. (*Cf. J.C.S., Oct.*)—J. F. S.

β -Monochlorhydrin; Glycerol —. L. Smith. *Z. physik. Chem.*, 1920, 94, 723–738.

A mixture of 97 g. of glycerol (95%) and 150 g. of hydrochloric acid is heated at 120° C. for 5 hrs., the mixture is distilled at 15 mm. pressure, and the fraction distilling at 110°–150° C. collected. This is pure chlorhydrin containing 15% of the β -isomer. The mixture is fractionated with a rod and disc still-head under a pressure of 11–15 mm. and after many repetitions the pure β -compound obtained. It has b.p. 124.5°–125° C. at 14.5 mm., sp. gr. 1.3375 at 0°, 1.3207 at 20° C.—J. F. S.

"Cupferron"; Preparation of —. D. R. Kasanof. *J. Ind. Eng. Chem.*, 1920, 12, 799.

In the preparation of "cupferron" (J., 1911, 1090) the yield of the intermediate substance, phenylhydroxylamine, depends greatly on the quality of the zinc dust used to reduce the nitrobenzene, but consistently good yields may be obtained by using amalgamated zinc dust. The latter is prepared, immediately before use, by stirring zinc dust with 2% mercurous nitrate solution containing a few c.c. of nitric acid; after a few mins., the solution is decanted and the zinc dust washed with water.

—W. P. S.

Acetone; Estimation of —, in minute amounts, by titration. R. S. Hubbard. *J. Biol. Chem.*, 1920, 43, 43–56.

THE author has modified Messinger's process (J., 1889, 138) so as to make it applicable to the analysis of very dilute acetone solutions. A process of repeated distillation is described whereby acetone may be separated from comparatively large amounts of alcohol and other substances which interfere with the accuracy of the method.

—J. C. D.

Acetone in expired air; Estimation of —. R. S. Hubbard. *J. Biol. Chem.*, 1920, 43, 57–65.

THE air is passed through wash vessels containing sodium bisulphite which effectively holds back all acetone. Interfering substances are removed by a distillation process (*cf. preceding abstract*), and the residual acetone is estimated either by the Scott-Wilson reagent or by titration.—J. C. D.

Copper; Catalytic activity of — [*on oxidation of alcohols*]. I. W. G. Palmer. *Proc. Roy. Soc.*, 1920, A, 98, 13–26.

ELECTROLYTIC copper has no catalytic effect on the oxidation of ethyl or isopropyl alcohol, even when the metal is alloyed with zinc. Copper prepared by the reduction of the oxide catalyses the reaction with the formation of acetaldehyde or acetone. (*Cf. J.C.S., Oct.*)—J. F. S.

Artemisia glutinosa; Essential oil of —. C. T. Bennett. *Perf. Essent. Oil Rec.*, 1920, 11, 286.

THE essential oil of *Artemisia glutinosa*, produced in Spain, has the following characteristics: Odour fragrant and aromatic, recalling that of a mixture of sage, rosemary, and spike lavender, the odour of borneol predominating; sp. gr., 0.937; rotation, +24°; n_D^{20} , 1.4780; total alcohols as borneol, 17.5%; esters as bornyl acetate, 6.3%; phenols, 8%; aldehydes and ketones, 18%; distillation, 52% between 175° and 200° C. The phenol is probably methylchavicol. The presence of thujone could not be established, but a small quantity of a fragrant aldehyde was isolated.—G. F. M.

Recovery of alcohol etc. Mariller. *See XVIII.*

PATENTS.

Esters; Continuous process for the manufacture of —. U.S. Industrial Alcohol Co., Assees. of A. A. Backhaus. E.P. 130,968, 23.6.19. Conv., 7.8.18.

SULPHURIC acid (0.33 pt. by weight of 62–95%), vinegar (10 pts. of 8% acetic acid), and methyl alcohol (0.8 pt. of 95%) are fed continuously into a column still at points situated respectively at the top, near the top, and near the bottom of the still, which is heated at the lower end by a steam coil. Dilute sulphuric acid passes out at the bottom of the still, and a mixture of methyl alcohol and methyl acetate vapour passes out at the top into a dephlegmator, which returns a portion of the alcohol to the top of the still. The remaining vapour passes into the middle of another similar column still; methyl alcohol flows out at the bottom and is led back into the first still at a point opposite the main methyl alcohol supply, and methyl acetate vapour passes out at the top into a dephlegmator, which removes the remaining methyl alcohol and returns it to the top of the still, and thence into a condenser. If glacial acetic acid is used instead of the dilute acid, the ingredients are mixed together and led into the first still through a common delivery pipe. Other acids and alcohols, e.g., formic acid and ethyl alcohol, may be employed.—L. A. C.

Alkaloids; Isolation of —. Chem. Fabr. vorm. Sandoz. E.P. 134,197, 16.8.19. Conv., 25.10.18. Addn. to 125,396 (J., 1920, 349 A).

THE process described in the chief patent and E.P. 131,283 (J., 1920, 426 A) for the extraction of ergot and hyoscyamine may be employed for the extraction of other alkaloids (pilocarpine, aconitine, and yohimbine) from alkaloidal vegetable matter, and also for the extraction of alkaloids from galenical extracts, inspissated juices, or other commercial preparations, in which case vegetable cellular matter such as powdered leaf which has previously been extracted with alcohol and benzene is mixed with the material before treatment.—L. A. C.

Calcium oxalate; Process for making —. F. M. Dupont and G. A. Hanke. U.S.P. 1,319,947, 17.8.20. Appl., 18.3.19.

A MIXTURE of sodium oxalate in solution and suspension, milk of lime, and carbon monoxide is subjected to heat and pressure.—W. J. W.

Caffeine; Manufacture of —. R. L. Datta. U.S.P. 1,350,092, 17.8.20. Appl., 17.10.19.

IN the production of caffeine from tea extract, a portion of the tannin and albumin is precipitated by addition of milk of lime, and the remainder is precipitated by means of basic or normal lead acetate.—L. A. C.

Propylene and its homologues; Preparation of —. Chem. Fabr. Buckau. G.P. 291,794, 6.12.12.

A MIXTURE of acetylene and methane and/or their homologues is passed under high pressure over a suitable porous non-metallic contact substance at 200°–350° C. Suitable catalysts are titanite or silicic acid and their salts, molybdic acid, tungstic acid, thoria, zirconia, etc. Examples are given of the preparation of propylene from acetylene and methane, ethylethylene from acetylene and ethane, and dimethylethylene from allylene and methane.—G. F. M.

Calamus root; Extraction of useful substances from —. A. Deppe Söhne, and O. Zeitschel. G.P. 307,623, 5.8.17.

DRIED and powdered calamus root is extracted with an organic solvent, and the essential oil separated from the fats in this extract by distillation. The residue containing starch and protein is saccharified and fermented for production of alcohol. About 30% of essential oil and 5% of fatty oil are obtained.—W. J. W.

Hydrastinine; Preparation of derivatives of —. K. W. Rosenmund. G.P. 320,480, 6.12.13.

METHYLENEDIOXYPHENYLISOPROPYLAMINE is treated with formaldehyde, or a substance yielding formaldehyde, and a catalyst, and the resulting condensation product is oxidised; or the condensation product is isomerised by means of a catalyst, alkylated, and the product oxidised. Iodine is used as the oxidising agent. For the preparation of 3-methyl-6,7-methylenedioxytetrahydroisoquinoline (m.p. 65°–67° C.) the product obtained from the calculated quantity of methylenedioxyphenylisopropylamine and formaldehyde is heated at 100° C. with 25% hydrochloric acid. The base gives with methyl-iodide 3-methyldihydrohydrastinine hydriodide, m.p. 240° C. The corresponding hydrochloride, m.p. 230° C., is obtained directly by heating the hydrochloride of the isopropylamine derivative with 2 pts. of formaldehyde solution for 3 hrs. at 130°–135° C. On oxidation with alcoholic solution of iodine in presence of potassium acetate, 3-methyldihydrohydrastinine gives 3-methylhydrastinine hydriodide, m.p. 207° C. The free base melts at 103°–104° C. These products are less poisonous than hydrastinine.—G. F. M.

Medicaments for "avitaminosis"; Preparation of —. R. Bosshard and F. Hefti. G.P. 320,785, 19.3.16.

PROTEIN substances rich in vitamins, particularly yeast or rice bran, are treated with dilute mineral acids at 80° C. until the biuret reaction gives a negative result. The product is filtered, freed from mineral acid, and then evaporated to dryness at the lowest possible temperature, either without further treatment, or after the conversion of the amino-acids present into their calcium or sodium salts. By avoiding too high a temperature or acid concentration the vitamins are by this means brought unchanged into solution.—G. F. M.

***o*-Gurtholmanomethyltri-hydroxyarsenomenthol; Preparation of** —. A. Mauersberger. G.P. 320,797, 12.9.15.

MENTHOLGUETHOLSULPHURIC acid ester, a brown, oily liquid, is condensed with monomethylsodium arsenate, or sodium benzenedisulphonate is treated with sodium ethoxide, sodium arsenate is added to the product, and the resulting compound is treated with mentholsulphuric acid methyl ester. The final product is a glutinous mass which solidifies at about 22° C., and has b.p. 154° C. (decomp.).

—L. A. C.

Nitrogenous compounds from the Corpus luteum; Preparation of —. L. Seitz and H. Wintz. G.P. 320,857, 19.7.14.

THE freshly-peeled *Corpora lutea*, reduced to small pieces, are subjected to successive extractions with alcohol and acetone on the one hand, and ether and chloroform on the other. The evaporated acetone extract is mixed with the alcoholic extract and the mixture united with chloroform extract, if necessary with the addition of a further quantity of chloroform. Finally the two layers, which are formed, are separated and evaporated to dryness. Two substances having antagonistic actions are obtained; the one, which is a lipid, soluble in chloroform and containing 61.2% C, 12.5% H, 4.33% N, 2.29% S, 2.83% P, and 16.85% O, stops menstruation, whilst the other, a water-soluble lecithalbumin, promotes menstruation.—G. F. M.

Aldehydes and ketones; Preparation of —. C. Harries. G.P. 321,567, 13.6.18.

REDUCTION of ozonides by means of potassium ferrocyanide, with or without the addition of an acid, yields aldehydes and ketones with little or no resin formation. A 95% yield of vanillin is obtained by reducing isoeugenolozonide in acetic acid, and nonyl aldehyde is formed from *n*-oleic acid ozonide. The ozonides from tar-oil, Hallenser gas-oil, or unsaturated oils which have been prepared by chlorinating crude paraffin oils and splitting off hydrogen chloride form aldehydes and ketones and only a small percentage of acids.—L. A. C.

Cholic acid; Method of obtaining — from bile. H. Wieland. G.P. 321,699, 10.8.16.

BILE is saponified by boiling with sodium hydroxide for 12 hrs., with removal by evaporation of about $\frac{1}{2}$ of the water, and the solution when cold is slowly acidified while being vigorously agitated with petroleum ether to dissolve fatty acids. The crystalline crude cholic acid when boiled with ether yields about 60% of almost pure cholic acid.

—L. A. C.

Mercury compounds of hydroxybenzenesulphocarboxylic acids and their homologues; Preparation of — which form colloidal solutions in water. Saccharin-Fabr. A.-G. vorm. Fahlberg, List und Co. G.P. 321,700, 8.12.18.

THE desired compounds are formed by mixing

aqueous solutions of a hydroxybenzenesulphocarboxylic acid or a homologue and a mercury salt, or by acting upon 1 mol. of a benzenesulphocarboxylic acid with less than $1\frac{1}{2}$ mols. of mercury oxide in the presence of water, and heating the mixtures or allowing them to stand for some time. Alternatively, a mercury compound of a hydroxybenzenesulphocarboxylic acid slightly soluble or insoluble in water is heated with an acid, or compounds or mixtures of mercury and salicylic acid, or its homologues, are treated with sulphuric acid and subsequently heated or allowed to stand for some time in the presence of a dilute acid. Mercury sulphosalicylate (prepared from 5-sulpho-2-hydroxybenzene-1-sulphonic acid) and mercury sulpho-*m*-cresotic acid are white crystalline compounds insoluble in organic solvents but soluble in water forming viscous colloidal solutions.—L. A. C. *Solvent*. G.P. 320,807. See XIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

[*Photographic*] *tone reproduction; Theory of — with a graphic method for the solution of problems*. L. A. Jones. Communication No. 88 from Eastman Kodak Research Lab. J. Franklin Inst., 1920, 190, 39—90.

THE relationship between the appearance of a photographic print and that of the object represented by it is dependent not only on the qualities (characteristic curves) of the negative and positive materials used in making the reproduction, but also upon the conditions of observation, the chief factor in this respect being the variation of the sensitiveness of the eye to brightness variation with varying degrees of illumination. The various factors are considered in detail, a new system of terminology and symbols being used, and a graphic method of combining curves given for investigation of various relationships when certain of the factors are known.—B. V. S.

PATENTS.

[*Photographic*] *colour-filter negative and process of preparing the same*. J. A. H. Hatt. U.S.P. 1,349,956, 17.8.20. Appl., 28.12.18.

A FIRST negative is prepared by exposure through a suitable colour-filter and a second negative by exposure through a different colour-filter and through the first negative in contact with the sensitive surface and in registration with the image of the picture. The second negative is used for the preparation of the printing plate.—B. V. S.

Celluloid products [photographic films]; Process for treating [recovering silver from] —. F. W. Horton. U.S.P. 1,350,157, 17.8.20. Appl., 13.1.20.

FOR the recovery of silver from photographic films they are treated with a solution of caustic alkali and the metal precipitated from the resulting solution.—B. V. S.

Photographic films; Process of making —. E. M. Flaherty, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,350,274, 17.8.20. Appl., 5.12.18.

A SOLUTION of nitrocellulose and camphor in butyl acetate and a diluent is fed on to a revolving drum on which it is partly dried, and the resulting film then stripped off, dried, and reeled in a continuous operation.—B. V. S.

Ferroprussiate paper; Manufacture of —. E. Bertsch. G.P. 320,981, 6.7.18.

ABOUT 10—20% of an alkali oxalate is added to alkali ferric oxalate solution before treatment with ferrieyanide. The yellow product remains unchanged in the dark, and on exposure to light

changes to emerald green, greenish-blue, bright blue-green, yellow, grey, and finally bright yellowish-grey. The treated paper has an intense blue colour after washing and drying, the lines being white.—W. J. W.

Photographic developing process. G. W. A. Sosna and J. E. Biedebach. E.P. 11,036, 29.7.15.

XXII.—EXPLOSIVES; MATCHES.

Nitro explosives; Modern high —. C. F. van Duin and B. C. R. van Lennep. Rec. Trav. Chem., 1920, 39, 145—177.

NITRO compounds of different types, capable of being used as explosives, were examined with respect to their stability, temperature of explosion, and sensitiveness to shock, an endeavour being made to determine the influence of constitution on these properties. In general the 2.3.4.6-tetranitro derivatives decomposed rapidly either at high temperatures or at the ordinary temperature, this being due probably to the splitting off of the nitro group in position 3. A comparison of tetranitroaniline and tetranitrophenylmethylnitramine, the latter being less stable, indicates that the substance containing the most active nitro group is the least stable. The instability of substances with mobile nitro groups is due to these mobile groups, as is shown by the fact that dipicrylamine is stable, whilst 2.4.6.2'.3'.4'-hexanitrodiphenylamine is not; a methylnitramine group also exerts an unfavourable influence on the stability of a compound, but less than the mobile nitro group. The introduction of a fourth nitro group or of a methylnitramine group considerably increases the sensitiveness of a compound to mechanical shock, whilst the introduction of an amino group lowers it. Etherification of a phenol diminishes its sensitiveness. In determining the explosion temperature it is advisable to take two readings, one where the temperature is raised from 100° C. by 20° per minute and the other where it is only raised 5° per minute. There is apparently no definite relationship between the stability of an explosive and the explosion temperature. Where a substance decomposes at its melting-point, this temperature and its explosion temperature are generally almost identical. For testing explosives for their sensitiveness to shock the authors consider that Kast's method (this J., 1909, 47) is quite satisfactory.—W. G.

PATENTS.

Propellant explosive. O. Silberrad. U.S.P. 1,349,983, 17.8.20. Appl., 10.9.19.

A "FLAMELESS powder" consists of nitrocellulose, 30.7%; nitroglycerin, 41%; vaseline, 3.9%; and starch, 21.4%.—W. J. W.

Explosives and other materials; Process for gelatinising —. A.-G. Siegener Dynamit-Fabrik. G.P. 298,420, 14.3.16.

WARM nitroglycerin is forced by compressed air through a jet, and the issuing stream draws in and mixes with air and collodion wool which has been whirled up into dust.—G. F. M.

Nitrocellulose; Process for safely drying — before gelatinisation. C. Claessen. G.P. 298,565, 14.9.16.

NITROCELLULOSE is mixed, in hot water under air agitation, with not more than 6% of a gelatinising aromatic nitro-compound. It then undergoes a preliminary gelatinisation by pressing it between hot plates into slabs which may be easily dried.—W. J. W.

Trinitrotoluene and other aromatic nitro-compounds; Purification of —. Sprengstoff A.-G. Carbonit. G.P. 299,015, 31.10.13.

THE nitro-compounds may be purified by re-

crystallisation from trichloroethylene or tetrachloroethane. Trinitrotoluene is soluble in 2 pts. of the former.—W. J. W.

Trinitrotoluene; Manufacture of —. R. Polltzer and V. Jelocnik. G.P. 299,661, 19.3.15.

PART of the waste acids derived from the manufacture of trinitrotoluene is revivified with nitric acid, and then used for converting mononitrotoluene into dinitrotoluene, part of the waste acid resulting from this operation being utilised for the manufacture of mononitrotoluene. The remainder of the waste acids from both trinitro- and dinitrotoluene manufacture is utilised for nitration of toluene, being thus freed from nitric acid and dissolved nitro-compounds, and the sulphuric acid in the waste acid from the toluene nitration, after purification and concentration, is available for use in the mixed acid for conversion of dinitrotoluene into trinitrotoluene.—W. J. W.

Explosive. A. Wilhelm. G.P. 300,010, 14.3.15. COTMARONE products, alone or mixed with nitrogen compounds, are suitable as vehicles for liquid oxygen or air for use as explosives. The velocity of detonation and brisance are greater than when natural resins are employed.—W. J. W.

Azides of the heavy metals; Preparation of —. O. Matter. G.P. 310,090, 26.8.17.

AN aqueous solution of hydrazoic acid or a metallic azide is added gradually to a solution of the acetate of a heavy metal. The corresponding azide slowly crystallises in fine regular crystals, which can easily be manipulated and will bear pressures up to 15,000 kg. per sq. cm. without exploding.

—G. F. M.

Picric acid; Manufacture of —. L. G. Badier and L. B. Holliday, Assrs. to L. B. Holliday and Co., Ltd. U.S.P. 1,349,802, 17.8.20. Appl., 30.12.19.

SEE E.P. 9962 of 1915; J., 1919, 305A.

[*Picric acid;*] *Evaporator [for concentrating weak solutions of —]*. J. N. Brooke. U.S.P. 1,349,810, 17.8.20. Appl., 1.7.18.

SEE E.P. 120,951 of 1917; J., 1919, 92A.

XXIII. — ANALYSIS.

Thermocouple for low temperature work; Needle type —. G. F. Taylor. J. Ind. Eng. Chem., 1920, 12, 797—798.

A METHOD of making this kind of thermocouple is described. Silk-insulated copper and "constantan" wires are used; as many lengths (about 1 m.) of each as there are to be junctions (*e.g.*, 1 to 50, or more) are cut, gathered together, and each end of the bundle is slipped through a hard glass tube, the ends of the wires are dipped in "bakelite," the insulation burned away for a length of 1 cm., the ends are twisted together in pairs and soldered. Each junction is then insulated by fusing over it a very small glass tube, and a tube of copper foil, 5 mm. in length, is drawn over the bundle above the junctions so that one end extends within the glass tube. The other end of the couple is made in the same way. A ten-junction thermocouple of No. 36 B. and S. gauge wire is only 1.5 mm. in diameter; it has a value of 0.002169° C. per microvolt over a range of 0° to -2° with a lag of 0.76 sec.—W. P. S.

Specific gravities; Determination of — by the pycnometer. W. Block. Z. angew. Chem., 1920, 33, 198—200.

THE author discusses the determination of sp. gr.

by means of the pycnometer and indicates the precautions to be observed as regards temperature, weighing, influence of atmospheric pressure, etc.

—W. P. S.

Specific gravity; The viscosimeter as a means of determining —. J. Holker. J. Path. Bact., 1920, 23, 185—187.

By combining the formulae of Ostwald-Poiseuille and of Scarpa (J., 1911, 51), in which the specific gravity of the liquid does, and does not, occur, respectively, it is possible to find the specific gravity by the viscosimeter. The results were in satisfactory agreement, and 2 c.c. or less of liquid is sufficient.—J. R. P.

Viscosities; Method of determining several — simultaneously. J. Holker. J. Path. Bact., 1920, 23, 177—184.

THE method of Scarpa (*cf.* preceding abstract) has been modified, and a method devised for determining several viscosities simultaneously. The method depends on the measurement of two times, t_1 and t_2 , the former occupied in drawing up through a vertical capillary sufficient liquid to fill a bulb at the top of this tube, and the latter taken by the liquid in flowing out of the bulb under its own weight. Under constant conditions, the viscosity is then proportional to $t_1 t_2 / (t_1 + t_2)$.

—J. R. P.

Calorimeter; Electrical adiabatic —. E. Cohen and A. L. T. Moesveld. Z. physik. Chem., 1920, 95, 305—327.

AN adiabatic calorimeter is described on the principle of that used by Richards, the main difference consisting in heating the liquid in the surrounding jacket by an electric current instead of by the neutralisation of sodium hydroxide by acid.—J. F. S.

Boiling point determinations under reduced pressure; Little noted sources of error in —. C. von Rechenberg and E. Brauer. Z. physik. Chem., 1920, 95, 184—214.

BOILING point determinations under reduced pressure are best carried out by the dynamic method. The following conditions should be observed: the boiling flask may be of glass or metal; in the latter case a flattened bottom is preferable, whilst in the case of glass a second neck carrying a capillary tube is advantageous. The neck of the flask should be surrounded with a vapour mantle; the boiling tube should be at least 20 mm. wide, the neck and condenser tubes 15 mm. wide. The thermometer bulb should be placed at least 50 mm. above the level of the boiling liquid, and the whole of the mercury thread should be in the vapour. Pressure measurements should be made on the boiling tube. The manometer should have a tube 15 mm. wide and be vertical. Pressures below 5 mm. should not be measured in this way, and the velocity of distillation should never be greater than one drop per second. Temperature and pressure measurements should not be made until 10 c.c. has distilled over. (*Cf.* J.C.S., Oct.)

—J. F. S.

Alundum filtering crucibles; Use of —. D. T. Inglis. J. Ind. Eng. Chem., 1920, 12, 799—800.

AFTER a precipitate in an alundum crucible has been washed as completely as possible in the usual way, it sometimes happens that soluble salts are retained by the upper portion of the crucible; these may be removed by placing the crucible in a suitable support in an ordinary funnel, pressing a moistened rubber stopper over the top of the crucible, applying suction, and pouring water into the funnel.—W. P. S.

Membrane filters; Chemical analysis with —. L. Moser and Kittl. Chem.-Zeit., 1920, 44, 637—638.

ALTHOUGH membrane filters are very useful for the separation of colloidal precipitates from liquids, their application to quantitative analysis, as recommended by Zsigmondy and Jander (J., 1919, 928A) is limited. The precipitate penetrates the surface of the membrane to a slight extent so that the whole of the precipitate cannot be removed for weighing, and it is not practicable to ignite the precipitate and membrane filter together.

—W. P. S.

Litmus paper; Preparation of red —. W. Fresenius and L. Grünhut. Z. anal. Chem., 1920, 59, 233—234.

ONE hundred g. of powdered litmus is boiled for 15 mins. with 500 c.c. of 94% alcohol, the mixture filtered, the insoluble portion dried at 100° C., and then ground with 500 c.c. of cold water; this mixture is stirred occasionally during 24 hrs., filtered, and a portion of the filtrate is set aside whilst the main portion is treated with dilute sulphuric acid (sp. gr. 1.11) until the colour changes to red. The solution is boiled for 15 mins., evaporated water being replaced, and, if the colour changes to blue, a trace more acid is added. A strip of paper is then dipped in the solution and dried. If too much acid has been used a quantity of the reserved portion of the filtrate may be added. When the desired tint after drying has been obtained, strips of paper are dipped in the solution and dried in an atmosphere free from acids and ammonia. Litmus paper thus prepared should give a blue coloration when treated with a drop of a mixture of 50 c.c. of boiling water and 1 drop of N/4 alkali solution.—W. P. S.

Alkalimetric titrations; Influence of atmospheric carbon dioxide on — using phenolphthalein as indicator. G. Bruhns. Z. Zuckerind. Czechoslov., 1920, 44, 331—335.

WHEN approximately N/100 sodium hydroxide solution is titrated with N/100 hydrochloric acid, using phenolphthalein as indicator, less of the acid is required than is the case with methyl orange as indicator. The difference may amount to about 10% of the volume of acid used, and is due to the absorption of carbon dioxide from the atmosphere during the titration. This influence of carbon dioxide is of importance in the determination of the alkalinity of sugar syrups; it may be diminished by reversing the procedure and adding the alkali solution to the acid.—W. P. S.

Water; Preparation of ammonia-free — [for analytical purposes]. G. C. Baker. J. Ind. Eng. Chem., 1920, 12, 798—799.

DISTILLED water is passed downwards through a layer of permutite, 12 in. deep and about 8 in. in diameter; this quantity of permutite will remove all the free ammonia from 100 gals. of water, but increases the mineral matter content. The activity of the permutite may be regenerated by treatment with 10% sodium chloride solution and washing as usual.—W. P. S.

Copper; Rapid determination of small amounts of — by the iodide method. H. F. Bradley. J. Ind. Eng. Chem., 1920, 12, 800.

THE precipitate of cupric sulphide, containing not more than 0.05 g. of Cu, is washed until free from chloride (complete removal of iron is not necessary), moistened with a few drops of zinc nitrate solution, and ignited; the residue of oxides is dissolved by warming with 1 c.c. of dilute hydrochloric acid (1:2), the solution neutralised with 5% potassium hydroxide solution, acidified with acetic acid, treated with a small quantity of phosphate solu-

tion to prevent interference by iron, 2.5 g. of sodium iodide added, and the liberated iodine titrated.—W. P. S.

SEE also pages (A) 650, *Paraffin wax* (Freund and Palik); 652, *Phenol* (Chapin); 655, *Alum* (Harvey), *Arsenic* (Robinson and Winter); 658, *Porosity of ceramic materials* (Bertrand and Boulanger); 659, *High-speed steel* (Slawik); 660, *Lead and silver refinery slays* (Stahl), *Zinc* (Olivier); 663, *Oxidised fatty acids* (Stadlinger), *Cottonseed oil* (Smith); 665, *Sulphur in rubber* (Dekker); 666, *Tannin analysis* (Schell); 667, *Acidity of soils* (Odén); 668, *Sugars* (Judd), *Saccharimeters* (Browne); 669, *Titration in stages* (Liers), *Wine* (Fresenius and Grünhut), *Volatile acids* (Hinard); 670, *Butter etc.* (Ferris), *Tyrosine* (Gortner and Holm); 671, *Vitamine* (Eddy and Stevenson); 673, *Adrenaline* (Scoville), *Phenolphthalein* (Palkin), *Cinnamic acid* (Denigès), *Glutamic acid* (Skola), *Urea* (Frenkel); 674, *Cupferron* (Kasanof), *Acetone* (Hubbard).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

- Baumgartner. Filtering media for liquids. 27,123. Sep. 23. (Austria, 6.11.15.)
 Biancardi. 26,986. See XIX.
 Conder and Vivian. Crushing machines. 26,623. Sep. 17.
 Hargreave. Apparatus for drying, concentrating, and filming solids, liquids, etc. 26,214. Sep. 13.
 Hayhurst. Filtering apparatus. 27,258. Sep. 24.
 Hayhurst. Liquid evaporators. 27,259. Sep. 24.
 Hofmann. Apparatus for drying pulverulent etc. substances. 26,657. Sep. 17.
 Metcalfe. Continuous kilns of the tunnel type. 26,556. Sep. 16.
 Noeggerath. Pumping or propelling semi-solids by viscosity pumps. 26,557. Sep. 16.
 Smallwood. Furnaces. 26,219. Sep. 13.
 Speight. Drying-cylinders, calenders, etc. 26,313. Sep. 14.
 Topf n. Söhne, and Wettig. Drying apparatus. 26,941. Sep. 21.
 Tregoning and Vivian. Grinding machinery. 26,397. Sep. 15.
 Walker. Pulverising mills. 26,358. Sep. 14.

COMPLETE SPECIFICATIONS ACCEPTED.

- 8571 (1919). Anderson. Furnaces or kilns. (150,762.) Sep. 22.
 14,097 (1919). Merz. Apparatus for evaporating liquids or recovering gases or vapours developed therefrom. (150,785.) Sep. 22.
 14,093 (1919). Merz. Extracting water from materials. (150,786.) Sep. 22.
 14,405 (1919). Thompson (Union Espagnole de Fabr. d'Engrais de Prod. Chim. et de Superphosphates). Centrifugal pumps, especially for acids etc. (127,831.) Sep. 22.
 14,888 (1919). Aube. Pulverising apparatus. (151,072.) Sep. 29.
 3333 (1920). Jewell. Stills. (150,940.) Sep. 22.
 8340 (1920). Akt. Myrens Verksted. Apparatus for drying moist solids. (140,473.) Sep. 22.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

- American Coke and Chemical Co. Coke ovens etc. 26,667. Sep. 17. (U.S., 3,10,19.)
 Davidson. 26,276. *See* XXIII.
 Davis and others. 27,157. *See* XXIII.
 George. Decomposing heavy hydrocarbon oils into lighter oils. 26,340. Sep. 14. (U.S., 30,9,19.)
 Hamer. Removal of water from oil. 27,216. Sep. 24.
 Herbert. Fuels for internal-combustion engines. 27,203. Sep. 24.
 Holst, Oosterhuis, and N. V. Philips Gloeilampenfabr. Removing gas residues and purifying inert gases in electric vacuum tubes. 26,994. Sep. 22. (Holland, 23,9,19.)
 Hutchins. Apparatus for destructive distillation of carbonaceous materials. 26,448. Sep. 15.
 Lessing. Treatment of coal. 27,162. Sep. 23.
 Pick and Strafford. Manufacture of solid fuel and distillation of tar. 26,568. Sep. 16.
 Soc. Franco-Belge de Fours à Coke. Treatment of gases from gas-producers. 26,451. Sep. 15. (Belg., 11,3,20.)
 Thompson (Gish). Treating hydrocarbons for explosive engines. 26,405. Sep. 15.
 Wallace. Apparatus for distilling carbonaceous materials. 27,278. Sep. 24.
 Wallace. Carbonising carbonaceous materials. 27,279. Sep. 24.
 White (Arrowood). Preparing powdered fuel for combustion. 27,028. Sep. 22.
 Wilputto. Coke-ovens etc. 26,789, 27,023, 27,038, 27,046. Sep. 20 and 22.
 Zwillingler. Coke-ovens. 26,461. Sep. 15.

COMPLETE SPECIFICATIONS ACCEPTED.

- 14,121 (1919). Lynn and Rambush. Generation and utilisation of producer gas. (150,787.) Sep. 22.
 32,709 (1919). Andre. Recovery of combustible material from furnace and boiler ashes. (151,192.) Sep. 29.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

- Atack. Process for oxidising hydrocarbons. 26,698. Sep. 18.
 Pick and Strafford. 26,568. *See* II.

COMPLETE SPECIFICATION ACCEPTED.

- 11,439 (1919). Dayton Metal Products Co. Hydrogenation of benzene. (133,666.) Sep. 22.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

- Donington. 26,542. *See* XVII.
 Dreyfus. Manufacture or treatment of products made with cellulose derivatives. 26,733. Sep. 18.
 Great Northern Paper Co. Paper-making. 27,169. Sep. 23. (U.S., 28,3,19.)
 Roerig. Manufacture of acetylcellulose. 27,158. Sep. 23.
 Soc. d'Impression des Vosges et de Normandie. Production of cotton goods to imitate wool or silk with a moiré effect. 26,821. Sep. 20. (Fr., 18,9,19.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 5596 (1919). Kirschbraun. Manufacture of waterproof paper or felted sheets. (151,029.) Sep. 29.
 14,028 (1919). Milne. Stationary digesters etc. used in paper-making etc. (150,782.) Sep. 22.

- 19,802 (1919). Clayton. Apparatus used in making artificial silk. (150,889.) Sep. 22.
 20,156 (1919). Johnson. Process for retting flax. (151,143.) Sep. 29.
 1125 (1920). Great Northern Paper Co. Paper-making. (137,815.) Sep. 29.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

- Brandwood, Pearce, and Sherwood. Cop-dyeing machines. 27,301. Sep. 25.
 Poulson. Sizing textile fabrics etc. 27,197-8. Sep. 24.

COMPLETE SPECIFICATION ACCEPTED.

- 11,231 (1919). Calico Printers' Assoc., and Rouse. Printing cotton fabrics. (151,056.) Sep. 29.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

- A.-G. f. Stickstoffdünger. Manufacture of nitrocalcite capable of being sprinkled and stored. 26,269. Sep. 13. (Ger., 12,9,19.)
 Bacon (Oldbury Electro-Chemical Co.). Manufacture of alkali formates and oxalates. 26,661-3. Sep. 17.
 Commin. Apparatus for producing cyanogen compounds or cyanides. 27,015. Sep. 22.
 Johnson (Badische Anilin u. Soda Fabr.). Manufacture of hydrochloric acid. 27,122. Sep. 23.
 Lunden and Thorssell. Production of ammonia from cyanides. 26,677-8. Sep. 19. (Sweden, 6,10,19.)
 Reed. Manufacture of sulphuric acid. 26,802. Sep. 20.
 Roiboul. 26,357 and 27,183. *See* VIII.
 Soc. Anon. l'Azote Français. Recovery of nitrous vapours. 27,161. Sep. 23. (Switz., 5,7,20.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 15,205 (1919). British Thomson-Houston Co. (General Electric Co.). Manufacture of nitric acid. (150,836.) Sep. 22.
 15,561 (1919). Thorssell and Lunden. Production of cyanides. (151,098.) Sep. 29.
 21,040 (1919). Deuts. Molybdänwerke. *See* X.
 24,129 (1919). Burgess Laboratories. Manufacture of zinc chloride. (133,319.) Sep. 29.
 26,740 (1919). Brusa, and Borelli & Co. Apparatus for making mercuric oxide from mercurous nitrate. (150,917.) Sep. 22.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

- Carborundum Co. (Linbarger). Refractory articles. 27,346. Sep. 25.
 Harvey, and Harvey Glass Furnace Co. Furnaces for melting glass. 26,561. Sep. 16.
 Loy. Furnaces for burning ceramic and refractory products. 27,049. Sep. 22.
 Rheinberg. Production of reflecting surfaces on glass by means of metals of the platinum group. 26,523. Sep. 16.
 Riddle. Porcelain. 27,118. Sep. 23.
 Roiboul. Fusing and casting silica, alumina, etc. 26,357. Sep. 14. (Fr., 15,6,20.)
 Roiboul. Manufacture of filaments or threads of silica, alumina, etc. 27,183. Sep. 24. (Fr., 16,6,20.)

COMPLETE SPECIFICATION ACCEPTED.

- 29,759 (1919). Mount. Glass-melting furnaces. (151,184.) Sep. 29.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Berry. Manufacture of artificial stone, cement, etc. 26,833. Sep. 20.
 Melsom, and Meldia Manufacturing Co. Manufacturing jointing material. 26,377. Sep. 14.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Alexander (Cobb Electro Reduction Corp.). Reduction of ores. 26,234. Sep. 13.
 Alexander (Luckenbach Processes, Inc.). Reagent for concentrating ore by flotation. 26,233. Sep. 13.
 Bacon and Kilby. Recarburising molten steel in open-hearth furnaces. 26,768. Sep. 20.
 Boorne. Tin-oxidising furnaces. 27,132. Sep. 23.
 Briolais and Correa. Alloy for soldering aluminium. 27,032. Sep. 22.
 Griggs. Open-hearth furnaces. 27,120. Sep. 23.
 Lavandeyra. Aluminium alloys. 26,508. Sep. 16.
 Metallbank u. Metallurg. Ges. Production of alloys. 26,627. Sep. 17. (Ger., 22,12,19.)
 Naismith. Basic open-hearth furnaces. 27,273. Sep. 24. (U.S., 17,4,16.)
 Thornton (Hagan Co.). Heat treatment. 26,830. Sep. 20.

COMPLETE SPECIFICATIONS ACCEPTED.

14,267 (1914). Lance. Treatment of the ores of certain metals. Sep. 29.
 15,213 (1919). Fish. Process of making steel. (151,092.) Sep. 29.
 21,040 (1919). Deuts. Molybdänwerke. Extraction of molybdenum compounds from yellow lead ores. 131,898.) Sep. 22.

XI.—ELECTRO-CHEMISTRY.

APPLICATION.

Harris and Rose. Electrolytic cell. 26,246. Sep. 13.

COMPLETE SPECIFICATIONS ACCEPTED.

13,029 (1919). Svenska Ackumulator Akt. Jungner. Electrodes for primary cells and storage batteries. (128,197.) Sep. 22.
 14,192 (1919). Wild and Barfield. Electric furnaces. (150,793.) Sep. 22.
 14,462-3 (1919). Pouchain. Negative plates for batteries and accumulators. (150,810-1.) Sep. 22.

XII.—FATS; OILS; WAXES.

APPLICATION.

Chaviara, Clayton, and Gill. Production of emulsions. 26,844. Sep. 21.

COMPLETE SPECIFICATION ACCEPTED.

14,340 (1919). Withers (National Electro Products, Ltd.). Hydrogenation of oils. (150,802.) Sep. 22.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATION.

Peachey and Skipsey. Vulcanisation of rubber etc. 27,019. Sep. 22.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Carmichael and Ockleston. Process of tanning. 26,757. Sep. 20.
 Klotz and Weiss (Dufaux). Substitute for horn. 26,730. Sep. 18.

XVI.—SOILS; FERTILISERS.

COMPLETE SPECIFICATIONS ACCEPTED.

10,528 (1918). Partington and Parker. Fertilisers. (151,024.) Sep. 29.
 29,378 (1919). Saves. Granulating calcium cyanamide to render it suitable for use as a fertiliser. (135,847.) Sep. 29.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATION.

Denington. Converting cellulosic and ligneous material into sugars. 26,542. Sep. 16.

XVIII.—FERMENTATION INDUSTRIES.

COMPLETE SPECIFICATION ACCEPTED.

11,673 (1919). Takamine. Diastatic composition and process of making same. (131,579.) Sep. 22.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Biancardi. Filter and purification tank. 26,986. Sep. 22.
 Ellison. Manufacture of disinfectant etc. 26,972. Sep. 22.
 Magrath. Softening, sterilising, etc., water. 26,223. Sep. 13.
 Thomson. Extraction of proteids from whey. 26,239. Sep. 13.
 Thomson. Preparation of proteid substances from natural solid proteids. 26,240. Sep. 13.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Imray (Soc. Chem. Ind. in Basle). Manufacture of soluble derivatives of camphoric acid. 26,924. Sep. 21.
 John. Manufacture of condensation products of formaldehyde and carbamide or carbamide derivatives. 26,381. Sep. 14. (Austria, 6,3,18.)

COMPLETE SPECIFICATIONS ACCEPTED.

15,034 (1919). Soller, Hotz, and British Cellulose and Chemical Manuf. Co. Production of acetaldehyde. (151,086.) Sep. 29.
 15,376 (1919). Commercial Research Co. Manufacture of chlorhydrins, bromhydrins, or olefinic hydrocarbons. (123,578.) Sep. 29.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Amiot. Photographic sensitisers. 26,365. Sep. 14. (Fr., 15,9,19.)
 Rheinberg. Light-sensitive collodion coating mixtures and films. 26,522. Sep. 16.
 Shepherd. Photographic processes. 26,335. Sep. 14.

COMPLETE SPECIFICATION ACCEPTED.

14,631 (1919). Greene and O'Malley. Colour photography. (150,819.) Sep. 22.

XXIII.—ANALYSIS.

APPLICATIONS.

Davidson. Gas calorimeters. 26,276. Sep. 13.
 Davis, Hollings, and South Metropolitan Gas Co. Apparatus for determining calorific values of coal gas etc. 27,157. Sep. 23.

I.—GENERAL; PLANT; MACHINERY.

Evaporator design; Studies in —. III. W. L. Badger and P. W. Shepard. Chem. and Met. Eng., 1920, 23, 390—393. (See J., 1920, 647 A.)

THE effect of hydrostatic head on heat transmission is dealt with, and the following conclusions are arrived at:—When treating water or other non-foaming, non-viscous liquids, heat transmission attains a maximum when the tubes are from one-third to one-half submerged; the shape of the bottom of the evaporator and hence the quantity of liquid below the bottom tube-plate compared with that above influences the result, the greater the amount of this bottom liquid the less being the influence of variation of hydrostatic head. As the liquor head increases above the top tube-plate, the heat transmission falls off along a straight line. These changes are only partly due to the variation of hydrostatic head, as variations in the velocity of circulation have considerable influence on the result.—W. H. C.

Coal-saving in the chemical industry. D. Brownlie. Chem. Trade J., 1920, 67, 247—250, 311—314, 343—347.

SEVERAL tables are given showing the results of tests made of the boiler plants of 60 works engaged in various branches of the chemical industry, together with a description of the methods used and a discussion of the results obtained. The average efficiency was found to be only 58%, and the poor results are attributed to the following causes:—Boiler grates are often too long, and too little fuel is burned per sq. ft. of grate area; the coal is frequently not tested, nor is a continuous record of the evaporation obtained always kept; impure feed water is not always treated, and often too little advantage is taken of the economisers even where they are installed. The measurement of the feed water and of the steam produced, the weighing and analysis of the fuel, and the installation of some form of continuous CO₂ recorder are the means indicated, together with a careful and regular consideration of the figures so obtained, by which an improvement may be effected.—W. H. C.

Ultra-filtration on an industrial scale. G. Schmitt. Chem.-Zeit., 1920, 44, 657—658, 669—671.

A TYPE of filter for the filtration of fine or colloidal suspensions is described, consisting of a perforated metal cylinder surrounded by numerous rings of fine wire gauze, which can be pressed together by means of a screw, so that the size of the filter pores can be regulated during the filtration. A screw conveyor within the cylinder forces the liquid through the filter, the actual filtering medium being a uniform layer of the deposit from the liquid itself. A medium suitable for colloidal filtration is formed by passing fibrous or pulverulent material through the filter. Instead of wire gauze, thin perforated metal rings may be used; or the spaces between the wire gauze rings may be packed with asbestos, cement, plaster of Paris, etc., to form a compact outer cylinder. Filtration is preferably effected at a pressure of 10 atmospheres. In another modification cylindrical cores are wrapped with wire in several places, the liquid then passing through the spaces between these wires, which for certain purposes may be covered with yarn, asbestos, cellulose, etc. These filtering surfaces behave like osmotic membranes, with the additional advantage of strength. For the filtration of large volumes of liquid they may be arranged vertically or horizontally in groups, or they may be used separately as revolving presses, the screw conveyor then being replaced by a central ram. In this way a pressure

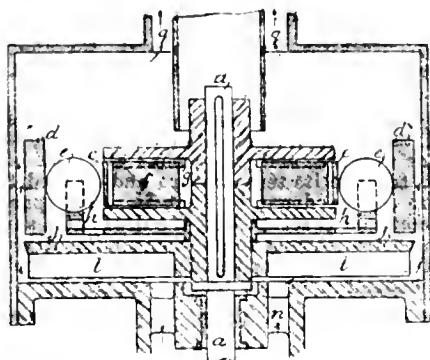
of over 300 atm. is obtainable. These presses are particularly suitable for the dehydration of clay for ceramic purposes, for the separation of precipitated pigments, the removal of bacteria from water supplies, separation of organic dyestuffs from the liquids in which they have been formed, and in the varnish and sugar industries. Oil may be simultaneously expressed from oil seeds at high pressure and filtered, and gases freed from suspended matter, with or without electrical precipitation.—C. A. M.

Distillation and rectification. Gay. See XVIII.

PATENTS.

Grinding machines [with air separation]. F. L. Smith & Co. E.P. 127,589, 29.5.19. Conv., 30.5.18.

In the fig. *d* is a fixed grinding ring, *e* grinding balls, and *c* an inner segmental grinding ring driven by the shaft, *a*, and forced against the balls by the



centrifugal force of loading blocks, *f*. The cage, *h*, is merely to separate the balls and is not driven. A disc, *j*, rotating with the shaft, has vanes, *l*, on its underside which draw air through ports, *n*, and force it (together with the ground material) through the narrow space between the outer grinding ring, *d*, and the outer casing. The velocity of the air here is sufficient to lift all the partly ground material, but on arrival at the larger space above the balls the velocity decreases so that the coarser particles fall back to be re-ground, and only the finely-ground material is blown out through *q*.

—B. M. V.

Surface condensers. R. N. Ehrhart. E.P. (A) 131,595, 18.8.19, and (B) 134,852, 6.11.19. Conv., 21.8 and 8.11.18.

(A) A RECTANGULAR condenser shell is provided with a vapour inlet, a condensed liquid discharge, and two outlets for the non-condensable fluid. Two nests of horizontal tubes are disposed within the shell so as to leave a downwardly tapering passage between them. Each nest of tubes is divided by a longitudinal baffle plate into two substantially triangular banks of tubes through which the steam passes in the direction of their decreasing cross-section. (B) The nests of tubes are arranged so as to leave a central converging passage from the vapour inlet to the condensed liquid outlet and to expose two sides of each nest to the incoming condensable fluid. Outlets for the non-condensable gas are provided midway between the top and bottom of each nest of tubes, which is further divided at this level by a substantially horizontal baffle plate, so that part of the condenser wall and a baffle plate form one of several converging passages leading to the non-condensable gas outlets.—W. H. C.

Drying processes in drying plants; Control of —.

G. Crida. E.P. 140,104, 11.3.20. Conv., 11.3.19.

THE substance being dried is supported in a counter-balanced container, the weight being recorded on a chart. Besides indicating, the pointer of the instrument also makes contact with either side of a double flexible conducting strip which is bent into such a position on the chart that the rate of drying at any instant may be maintained according to a pre-determined curve of maximum efficiency. For example, a reversible electric motor controlled by the contact arm may be used to control the heat supply.—B. M. V.

Drying processes and apparatus therefor. T.

Boberg, and Techno-Chemical Laboratories, Ltd. E.P. 149,055, 3.5.19.

THE wet material is spread upon a heated surface (e.g., a rotating drum), to which it is caused to adhere until it is sufficiently dry, when it is removed. The apparatus is enclosed in a casing and the steam given off is withdrawn, and after having its temperature raised by being compressed is utilised as the heating medium.—W. H. C.

Drying processes and apparatus therefor. T.

Boberg, and Techno-Chemical Laboratories, Ltd. E.P. 150,068, 27.5.19.

PULPY material of high water content, e.g., peat, is spread in a thin film on a slowly rotating heated drum or the like, and the vapour given off is compressed to raise its temperature and applied to the interior of the drum to heat it. The partially dried material is scraped off the drum at a suitable point and transferred to another similar dryer. If desired the drying may be effected in three or more stages instead of in two stages.—B. M. V.

Drying apparatus. J. L. Cloudsley. E.P. 149,774, 19.5.19.

AN apparatus for drying such material as broken stone or sand consists of a number of superposed trays, alternately convex and concave, the material flowing down the slope and over the outer edge of the former and down the slope and through a central opening of the latter. The trays are vibrated vertically by means of a central shaft, to which both sets or either set may be rigidly attached; in the latter case the second set is carried by resilient supports. The slope of the convex plates is much flatter near the circumference than near the centre.—B. M. V.

Dryer. J. O. Tensfeldt. U.S.P. 1,349,590, 17.8.20. Appl., 18.11.19.

A DISTRIBUTING system for dry hot air is arranged above a drying chamber, below which is a chamber for receiving the moisture-laden air. The air is withdrawn from this latter chamber, dried, and returned to the distributing system, whence it passes downwards through the material being dried.—W. H. C.

Desiccating fluid substances; Process for —.

R. W. G. Stutzke, Assr. to Crown Maltose Co. U.S.P. 1,350,247, 17.8.20. Appl., 8.6.16.

THE fluid is injected as a spray into a superheated atmosphere of its own vapour, the evaporated liquid is withdrawn from the system at such a rate as to maintain a sub-atmospheric pressure, and the desiccated solids are collected.—J. H. L.

Filtering and/or mixing oils and other liquids;

Apparatus for —. R. H. Cox. E.P. 149,038, 30.4.19.

THE liquid is fed through a hollow shaft into a horizontal drum, the peripheral wall of which is formed of two layers of wire gauze or perforated metal with one or more layers of felt or other suit-

able material between. The drum is rotated at high speed in a tank in which the liquid collects after passing through the gauze and fabric.—W. H. C.

Distilling and fractionating liquids [e.g., petroleum]; Apparatus for —. J. Y. Johnson. From H. L. Allan. E.P. 149,776, 20.5.19.

PETROLEUM or other hydrocarbon is fractionally distilled in a series of rectifiers which are connected at the top by atmospheric condenser and reflux pipes. Each rectifier is provided with a number of vertical open-ended tubes through which the liquid passes upwards from a header at the bottom and overflows at the top into an upper chamber formed by a horizontal partition midway of the height of the rectifier. The liquid then passes through a connecting pipe to the bottom header of the next rectifier. The lower portions of the vertical tubes may pass through a compartment bounded by two horizontal partitions, and superheated steam may be passed into this compartment. The vapour passes into the next rectifier by the air condenser pipes at the top.—W. F. F.

Heated air; Arrangements for supplying — to furnaces. W. H. Owen. E.P. 150,065, 27.5.19.

THE air for combustion is heated by the waste gases: after an initial heating the hot air is divided, part going direct under the fire-bars, the other part being further heated to a much higher temperature and supplied above the fire-bars.—B. M. V.

Kilns. W. Somerville. E.P. 150,157, 23.7.19.

IN a kiln for burning lime, dolomite, etc., outlets for carbon dioxide are provided just below the level of the fuel bed, leading to damper-controlled uptakes, which rejoin the main chimney above the main dampers. The fire is only allowed to burn upwards at starting.—B. M. V.

Foam in liquids; Method of producing —. E. Wirth-Frey. U.S.P. 1,351,089, 31.8.20. Appl., 8.4.19.

DURING the heating of liquids the steam generated is drawn off and re-introduced under pressure, out of contact with the atmosphere, in order to produce foam.—W. J. W.

Separating substances from liquids [e.g., wax from mineral oils]; Process for —. P. T. Sharples. U.S.P. 1,351,265, 31.8.20. Appl., 28.6.19.

TO separate from a liquid a substance which is soluble in it at normal temperatures, but insoluble at lower temperatures, the mixture is cooled to a temperature at which the substance separates, and centrifuged with a liquid which is heavier than the substance and non-miscible with it. The substance floats on the heavier liquid and is separately discharged. The process is applicable to the separation of wax from mineral oils by means of brine.—W. F. F.

Material from solution; Apparatus for recovering —. W. D. Mount, Assr. to Nitrogen Products Co. U.S.P. 1,352,210, 7.9.20. Appl., 27.6.18.

THE solution is heated in a horizontal cylindrical casing provided with a combined agitator and conveyor which can be rotated in one direction to agitate the contents of the casing and in another direction to discharge the material at one end of the casing.—B. V. S.

Fire-extinguishing liquid. S. S. Sadtler, Assr. to C. B. Ford and J. M. Castle. U.S.P. 1,316,191, 16.9.19. Appl., 28.4.17.

CARBON tetrachloride is mixed with less than 10% of a saturated hydrocarbon adapted to lower the freezing point and raise the flash-point of the composition comparatively to the freezing and flash-

points of carbon tetrachloride. One example of such a mixture is carbon tetrachloride with about 5% of petroleum spirit (sp. gr. 0.759–0.766); this mixture has a flash-point of 113.5° C. and a freezing point of 10° C.—W. H. C.

Evaporating apparatus. S. M. Duck. E.P. 150,007, 7.2.19.

SEE U.S.P. 1,298,470 of 1919; J., 1919, 398 A.

Furnace. A. Smallwood. U.S.P. 1,351,801, 7.9.20. Appl., 31.7.18.

SEE E.P. 120,259 of 1917; J., 1919, 1A.

Crushing machines. J. D. Ochterbeck. E.P. 128,936, 24.6.19. Conv., 12.1.14.

Centrifugal disintegrating machines. W. Brealey. E.P. 149,751, 16.5.19.

Shaft kilns and the like; Discharge mechanism for —. Gebr. Bühler. E.P. 137,269, 11.6.19. Conv., 31.12.18.

Furnaces; Forced draught —. H. C. Mann, and The Chemical Engineering and Wilton's Patent Furnace Co. E.P. 150,451, 11.6.19.

Refrigerating plant; Condensers for —. A. G. Wood and E. Hall. E.P. 149,797, 29.5.19.

Heat exchange apparatus [; Tubular —]. W. Smith and R. Stephenson. E.P. 149,882, 24.12.19.

Depositing soda etc. U.S.P. 1,350,128. See VIII.

Treating liquids. U.S.P. 1,351,522. See XVIII.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Lignite; Comparison of methods for producing tar and gas from —. H. Holzwarth. Braunkohle, 1920, 19, 189–192. Chem. Zentr., 1920, 91, IV., 374–375.

Four methods of treatment of lignite containing 8% of bitumen have been compared, viz.: (1) The moist lignite was directly gasified in producers; (2) the lignite in the form of briquettes containing 15% of moisture was gasified with recovery of tar and by-products; (3) partial carbonisation in rotary ovens to obtain tar and burning coke; (4) partial carbonisation in Thyssen rotary ovens for production of burning coke and subsequent gasification of the coke in producers. The following results were obtained, calculated to the raw, undried lignite:—

	1.	2.	3.	4.
Consumption of raw lignite in production of 1 million calories in the gas	700 kz.	1805 kg.	925 kg.	800 kg.
Tar yield in production of 1 million calories in the gas	28 kg.	31 kg.	29.3 kg.	63 kg.
Ratio raw lignite consumed : tar produced	25	5	31.6	12.5

—A. J. H.

Heavy mineral oils; Determination of the specific heat of —. H. S. Bailey and C. B. Edwards. J. Ind. Eng. Chem., 1920, 12, 891–894.

THE oil (250 g.) is introduced into a vacuum-jacketed calorimeter provided with an electrical resistance coil and a stirrer, and a current of measured strength transmitted. The temperature, voltage, and amperage are read at intervals of

1 min. between the limits of temperature required, the current then cut off, and the system allowed to cool with the agitator still running, and the falling temperature noted at intervals of 5–10 mins. through the range. The specific heat is calculated by means of the equation

$$\text{Sp. ht} = C/W = V \times A \times 60 \times 0.239 / W(t + r) + c/W,$$

where C is the heat capacity of the oil, W the weight of oil, V the average voltage, A the average amperage, 0.239 cal. per joule, c is the water equivalent of the calorimeter, t the rise of temperature in ° C. per min., and r the radiation in ° C. per min. from the cooling curve. The specific heat of aniline thus determined agreed closely with the recorded value.—C. A. M.

Coal; Economic selection of —. A. L. Booth. Iron and Steel Inst., Sept., 1920. [Advance proof.] 9 pp.

FOR the selection of a suitable coal for industrial use microscopical examination may be of value when used in conjunction with chemical analysis. The author classes coals in three main types—"humic," "spore," and "cannel" coals (Lomax, Trans. Inst. Mining Eng., 1911, 42, (i.), 1; J., 1913, 276). The extent to which these types merge into one another is judged from the microscopical examination. For steam raising humic coals, containing a fair proportion of cuticle or spores, are the most suitable. Spore coals are necessary for producer-gas work. For direct-fired furnaces the hard coals, which are almost true cannels, are used. Coal from the same seam was found to be usually very uniform, even when delivered on dates several years apart.—W. P.

Gasoline from natural gas. III. Heating value, specific gravity, and specific heat. R. P. Anderson. J. Ind. Eng. Chem., 1920, 12, 852–857.

FORMULÆ are given showing the relationship between the cal. per g.-mol. of normal paraffin hydrocarbons and the number of carbon atoms per mol., and between the B.Th.U. per lb., per cub. ft., and per gall., and the number of carbon atoms per mol. and sp. gr. in liquid form respectively. In so far as gasoline is a mixture of normal paraffin hydrocarbons, the relationship between the B.Th.U. per lb. or per gall. and the sp. gr. (G.) may be expressed by the formulæ—B.Th.U. per lb. = $23,330 - 3500 G$; B.Th.U. per gall. (at 15.5° C.) = $14,200 + 153,500 G$. These formulæ may be applied directly to natural-gas gasoline and straight-run gasoline. The probable values of the heating value and sp. gr. of the vapour and vol. of vapour per gall. have been calculated for four grades of gasoline, and figures are given showing the change in the heating value and sp. gr. caused by the extraction of different amounts of gasoline from different grades of natural gas.

—C. A. M.

Java petroleum; Catalytic hydrogenation of lava-rotatory fractions of —. W. Steinkopf and F. Beiersdorf. J. prakt. Chem., 1920, 101, 75–78.

NO inversion of the sign of rotation was produced by the catalytic hydrogenation of Java petroleum fractions, and the very slight alterations observed in the magnitude of the specific rotation appear to indicate that optical activity is caused by the presence of a saturated compound which is not affected by hydrogenation. The rotation cannot be due to the presence of cholesterylene, since this, when catalytically hydrogenated, yields dextro-rotatory cholestane. The optical activity of the oils is more pronounced as the quantity of unsaturated substances in them diminishes.—H. W.

Asphalt and coke content of petroleum; Relations between the —. W. Steinkopf and H. Winternitz. J. prakt. Chem., 1920, 101, 82–92.

PETROLEUMS with a high content of hard asphalt

give also a high yield of coke. Soft asphalt can, however, play a considerable rôle in the formation of coke, since certain oils which have a particularly small hard asphalt content show a high coke number. About 40–60% of the soft asphalt is converted into coke. The differences between the coke yields determined at the ordinary pressure and in a vacuum are only considerable if the oil has a high asphalt content.—H. W.

Coal-saving. Brownlie. See I.

Ether-air mixtures. McClelland and Gill. See XX.

Mixtures of gases. Weaver and Palmer. See XXIII.

PATENTS.

Fuel; Manufacture of — and apparatus therefor. J. Armstrong. E.P. 149,449, 20.5.19.

POWDERED fuel, e.g., coal, peat, sawdust, etc., is compressed with a binder to form a long continuous bar, in which deep narrow grooves are cut by revolving blades, running in a bath of tar, oil, water, or other liquid which is thus deposited in the grooves. On combustion the liquid produces vapours which open up the structure of the briquette and thus promote satisfactory combustion.—A. G.

Peat; Method of treating —. W. W. Blair. U.S.P. (A) 1,349,713 and (B) 1,349,714, 17.8.20. Appl., (A) 20.6.18, (B) 14.12.16. (B) Renewed 13.5.19.

(A) FRESHLY dug peat is macerated, pressed into blocks, dried, and impregnated with a fuel-oil. (B) Raw peat (unmacerated) is compressed into blocks, which are dried and impregnated with a fuel-oil.—A. G.

Water-gas; Producing — and carbonising coal. H. L. Doherty. E.P. 132,483, 10.5.19. Conv., 7.9.18.

A COLUMN of fuel is subjected to a cross blast of air to produce a high temperature zone, and a longitudinal blast of steam for the production of water-gas. The current of steam may be started before the cross blast is entirely cut off. The furnace comprises a fuel shaft communicating with regenerators by way of transverse flues opening into the shaft at an intermediate zone, the shaft at this zone being restricted in sectional area and formed (e.g. by arches), so as to deflect the transverse blast downwards between the flue openings. The fire arches are connected by controlled passages for leading a portion of the gas of the transverse blast direct from one flue to the other.—A. G.

Gas producers. S. B. Sheldon. U.S.P. (A) 1,350,472, (B) 1,350,473, (C) 1,350,474, and (D) 1,350,475, 24.8.20. Appl. (A, B) 11.6.18, (C) 6.9.18, (D) 4.11.18.

(A) POKERS are mounted on opposite sides of the centre of a revolving beam, thus disturbing the fuel in the coking zone. (B) Fuel is fed by means of a plunger from the floor of a water-cooled hopper, located directly above the top cover of the producer, on to a water-cooled distributor, actuated externally. (C) The cover of the producer is mounted on wheels and can rotate on a track running round the periphery of the main structure. A superstructure capable of rotation is mounted independently of the top and carries the producer-operating mechanism. (D) The body of the producer is capable of rotation whilst the top is stationary and is sealed to prevent escape of gas. The top has a large number of poker openings and vertical pokers which fit loosely in the openings by ball and socket joints, so as to lag as the body rotates. The ball-and-socket members are pierced so that the poker shanks can move reciprocally. The pokers are raised by actuating mechanism and fall by gravity

at regular intervals, so that the fuel bed is maintained in a porous condition.—A. G.

Acetylene or like explosive gases dissolved in liquids; Porous charges for containers for storage of —. J. Pintsch A.-G. E.P. 135,511, 21.11.19. Conv., 20.5.14.

THE charge consists partly, but not wholly, of porous materials capable of swelling, e.g., cork, elder-pith, or like substances of vegetable origin.—A. G.

Fuel; Process of producing liquid — and product thereof. E. C. R. Marks. From U.S. Industrial Alcohol Co. E.P. 149,398, 7.5.19.

10 g. of calcium carbide is added to 50 c.c. of ethyl alcohol (95%). Part of the acetylene produced by the dehydration of the alcohol dissolves in the alcohol, whilst the balance is passed into a mixture of 50 c.c. of gasoline and 40 c.c. of kerosene. The dehydrated alcohol is filtered off from any calcium hydroxide produced and is then mixed with the gasoline and kerosene, thus producing a liquid suitable for motor fuel or for burners.—A. G.

Wax; Process of expressing — from petroleum distillates. W. C. and F. E. Wells. U.S.P. 1,350,482, 24.8.20. Appl., 23.4.14.

THE distillates are fed to a woollen fabric strainer, uniformly and adequately supported by a structure that does not cause material obstruction, so that, under sufficient pressure, the oils are expressed from the distillate.—A. G.

Gasoline; Process for cleaning —. M. Leitch, Assr. to De Laval Separator Co. U.S.P. 1,351,047, 31.8.20. Appl., 23.9.18.

GASOLINE contaminated with water and other impurities is mixed with a saponifying agent and the resulting emulsion is broken, whereby it separates into a layer of pure gasoline and other layers containing the soap, water, and dirt free from gasoline.—W. F. F.

Volatile [hydrocarbon] liquids from gases; Process for obtaining —. L. F. Bayer. U.S.P. 1,351,151, 31.8.20. Appl., 11.10.17.

THE gas is mixed with a hydrocarbon oil and the mixture compressed. Another liquid is artificially refrigerated and added to the mixture of gas and hydrocarbon oil under pressure, whereby the volatile constituents of the gas are condensed, the condensed liquid mixing with the oil; the refrigerating liquid is subsequently separated from the mixed hydrocarbons.—J. S. G. T.

Distilling mineral oil; Apparatus for —. E. V. Stone. U.S.P. 1,351,266, 31.8.20. Appl., 14.11.18.

MINERAL oil is distilled in a rotating retort which is slightly inclined to the horizontal. The retort contains a number of iron rods of equal length which break up carbon deposits by their impact due to the rotation of the retort, and the carbon is discharged at the lower end to a conveyor.—W. F. F.

Fuel and process of forming same. D. Markle. E.P. 126,972, 16.5.19. Conv., 11.9.17.

SEE U.S.P. 1,287,382 of 1918; J., 1919, 128 A.

Petroleum and like oils; Treatment of —. E. W. Wynne. U.S.P. 1,351,458, 31.8.20. Appl., 28.8.18.

SEE E.P. 127,918 of 1918; J., 1919, 567 A.

Feeding [powdered] fuel to káns; Hopper for —. G. A. Collins. E.P. 150,161, 31.7.19.

Gas producers; Rotary grates for —. H. Nielsen and F. D. Marshall. E.P. 150,261, 21.8.19.

Distilling petroleum. E.P. 149,776. See I.

Separating wax from mineral oils. U.S.P. 1,351,265. See I.

Nitric oxide in gas engines. G.P. 321,981. See VII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

PATENTS.

Retorts and the like [for low-temperature distillation]. Merz and McLellan, A. C. Michie, and E. G. Weeks. E.P. 149,733, 14.5.19.

AN apparatus for feeding fuel to low-temperature distillation retorts comprises one or more lateral conduits projecting horizontally into the retort at a level below the normal surface of the fuel. The conduits are open at the top within the retort, and each contains a screw conveyor which causes the fuel to overflow into the retort without the creation of dust and consequent fouling of the condensers which occurs when the fuel is dropped into the retort from a hopper.—W. F. F.

Wood and other carbonaceous material; Process and apparatus for carbonising —. J. F. Wells. E.P. 149,773, 19.5.19.

WOOD, vegetable matter, peat, coal, lignite, and other carbonaceous materials are carbonised in a vertical retort to which air is admitted through the grate to burn the volatile products at the bottom. The hot gases pass upwards through the charge to distil the lighter volatile constituents in the upper part. The carbonised material is withdrawn at the bottom at the moment when the last portion of the volatile product is being consumed and is replaced by the descending fuel.—W. F. F.

Vegetable charcoal; Production of highly active granular —. Verein Chem. Fabr. in Mannheim. G.P. 309,221, 15.4.17.

WOOD in small pieces of appropriate size is impregnated with solutions of electrolytes other than caustic potash, potassium carbonate, and similar compounds of potassium, and carbonised at a dull red heat. It is then lixiviated, impregnated with alkaline solutions (e.g., potassium carbonate), and carbonised at a bright red heat.—J. S. G. T.

Distillation of wood or coal; Process for removing tar from products of —. E. A. Barbet. U.S.P. 1,351,363, 31.8.20. Appl., 6.11.18.

SEE E.P. 120,558 of 1918; J., 1920, 57 A.

III.—TAR AND TAR PRODUCTS.

Alkali fusions. II. Fusion of sodium benzene-disulphonate with sodium hydroxide for the production of resorcinol. M. Phillips and H. D. Gibbs. J. Ind. Eng. Chem., 1920, 12, 857—860.

THE best yields of resorcinol are obtained by the fusion of 16 mols. of sodium hydroxide with 1 mol. of sodium benzene-*m*-disulphonate for 2 hrs. at 310° C., in the presence of not more than 2—4% of water. (Cf. J.C.S., Nov.)—C. A. M.

Naphthalene and anthraquinone; Nitro-derivatives of —. S. N. Dhar. Chem. Soc. Trans., 1920, 117, 1001—1001.

THE two isomeric tetranitronaphthalenes were obtained by nitrating 1,8-dinitronaphthalene with a mixture of equal volumes of concentrated sulphuric and nitric acids. The hitherto unknown 1,2,6,8-tetranitro compound separates out from this mixture. It does not melt below 300° C. Two trinitro-

β -naphthoic acids melting at 220° C. and 215° C. respectively were obtained by the nitration of β -naphthoic acid. As the result of the nitration of anthraquinone under various conditions four dinitro compounds were obtained, of which two were identified as 1,5-dinitroanthraquinone, and the hitherto unknown 1,3-dinitroanthraquinone, m.p. 210° C., respectively.—G. F. M.

s-Xylidine; Synthesis of —. H. L. Haller, E. Q. Adams, and E. T. Wherry. J. Amer. Chem. Soc., 1920, 42, 1840—1842.

s-XYLIDINE may be prepared from mesitylene by way of mesitylenic acid and its amide, but a more satisfactory process is a modification of that of Willgerodt and Schmierer (Ber., 1905, 38, 1472), starting from *m*-4-xylidine. (Cf. J.C.S., i., 670.) —W. G.

Halogenation. Dhar. See XX.

PATENT.

Reduction of nitro-compounds. E.P. 150,412. See XX.

IV.—COLOURING MATTERS AND DYES.

Cyanine dyes. II. Synthesis of o-amino-cinnamylidene-quinaldine methiodide. W. H. Mills and P. E. Evans. Chem. Soc. Trans., 1920, 117, 1035—1040.

o-NITROCINNAMIC aldehyde was condensed with quinaldine in presence of zinc chloride with the formation of *o*-nitrocinnamylidene-quinaldine, which was reduced to the amino-compound with iron and acetic acid, and the quaternary methiodide of this base was then prepared, the amino-group being temporarily protected by acylation. The compound forms deep reddish-brown solutions, which are turned pale yellow by mineral acids. It dyes wool and silk a rich reddish-brown, but the colour is not fast to light. Its absorption spectrum differs from that of the isocyanines, and the compound shows little analogy in properties to these substances. It has also no appreciable sensitising action on a gelatino-bromide photographic plate.—G. F. M.

Catechin; Constitution of —. I. M. Nierenstein. Chem. Soc. Trans., 1920, 117, 971—979.

THE methylated reduction product obtained from catechin tetramethyl ether by Kostanecki and Lampe (Ber., 1907, 40, 720), and variously regarded as 2,4,6,3',4'-pentamethoxy-3-ethylidiphenylmethane and as 2,4,6,3',4'-pentamethoxy- α -diphenylpropane, is not identical with either of these substances, which have now been synthesised from 2,4,6,3',4'-pentamethoxy-3-ethylbenzophenone and 2,4,6-trimethoxyphenyl-3,4-dimethoxystyryl ketone respectively. The acid, $C_{19}H_{20}O_8$, however, which is obtained by the oxidation of Kostanecki's methylated reduction product, $C_{20}H_{26}O_8$, is shown to be identical with 2,4,6,3',4'-pentamethoxydiphenylacetic acid synthesised from the corresponding diphenylmethyl chloride by the Grignard reaction. It is accordingly deduced that the methylated reduction product is the pentamethoxy- α -diphenylpropane. Catechin itself would therefore be represented by a slight modification of Perkin's chroman formula (Trans. Chem. Soc., 1902, 81, 1172).—G. F. M.

Dyes; Table for the identification of — by means of the spectroscope. J. Moir. J. S. Afr. Assoc. Anal. Chem., 1920, 3, 9—13.

THE table given shows the position of the centre of the absorption-band in the spectra of a large number of dyes as observed in a wave-length spectroscope. The positions range from 425 for Aurantia to 668 for Tetramethylthionine.—W. P. S.

Ultra-filtration. Schmitt. See I.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

[Paper pulp;] *Curtaiment of bleaching powder consumption by the addition of sulphur to the digester liquor [in manufacture of —].* G. K. Spence. Paper, 1920, 26, 19—21.

TABLES of the results of 35 experimental digestions, with and without the use of sulphur, are given. In all cases the sulphur was used at the rate of 1.1% on the weight of stock. It was found that 1.1 lb. of sulphur effected a saving of only 0.92 lb. of bleaching powder (35% avail. Cl). This is a small advantage, since claims that the sulphur leads to increase of yield and strength are not yet proved.—A. J. H.

[Parchment] *paper pulps; Mucilage of —.* F. B. Seibert and J. E. Minor. Pulp and Paper Mag., 1920, 18, 939—942. (Cf. Schwalbe and Becker, J., 1919, 858 A; 1920, 330 A.)

THE presence of hydrocellulose is favourable to the development of mucilage during the beating of paper pulp, and this explains why parchment pulps having a high copper number require less beating than pure cellulose pulps. It is advantageous to subject pulp to a cold acid treatment before beating. This increases the amount of hydrocellulose present, and also the strength of the paper produced.—A. J. H.

PATENTS.

Flexible [fireproof] material for aeroplanes. N. A. T. N. Feary. E.P. 149,745, 15.5.19.

NON-CONDUCTING flexible fabrics are made fireproof and also generally waterproof without loss of tensile strength by coating them with a metal by electrolytic deposition.—A. J. H.

Artificial silk and staple fibres; Treatment of —. A. Lanffs. G.P. 322,141, 3.12.18.

IF suitable organic substances such as sugars, glycerol, and soluble starch be added to the water used for precipitating the fibres, the harmful effects of any lime still present (most of the lime is removed by a preliminary treatment of the water) is much diminished and, especially in dyeing, more even results are obtained.—A. J. H.

Pyroxylin products; Method of making —. J. G. Jarvis. U.S.P. 1,351,652, 31.8.20. Appl., 13.3.19. Renewed 23.6.20.

A FILM is produced by spreading a plastic solution of nitrocellulose and acetylcellulose on a carrier and topping this with a fluid mixture made by the addition of acetone and collodion to a mixture of acetylcellulose, naphthalene, and equal parts of acetone and ethyl acetate. A solution, *e.g.* of chloral hydrate is finally added when the mixture is homogeneous in order to reduce the inflammability of the film.—A. J. H.

[Celluloid] *films; Obtaining products from —.* W. C. Pierson. U.S.P. 1,352,216, 7.9.20. Appl., 27.11.17.

THE celluloid is dissolved in acetone, and thrown out of solution, by the addition of water, as a thick mass which is then washed, dried, and pulverised.—B. V. S.

Paper-stock; Method of reclaiming —. C. Baskerville and C. M. Joyce. U.S.P. 1,351,092, 31.8.20. Appl., 7.10.19.

WASTE inked paper is saturated in a mixture of kerosene, borax, soap, and water.—W. J. W.

Wood-pulp liquor; Method of and means for the de-acidification of raw —. B. Grätz. E.P. 150,571, 26.11.19.

THE liquor is allowed to flow along a trough through which the ashes from lignite or other brown coals are transported in an opposite direction by means of a mechanical conveyor. The cost of neutralisation is small, and the liquor obtained may be used for preparing artificial stone.—A. J. H.

Soda-cellulose waste lyes; Process for treating —. H. Achenbach. G.P. 322,771, 16.2.19.

THE waste liquor passes down a tower, in which it is concentrated from about 5° B. (sp. gr. 1.037) to about 10° B. (sp. gr. 1.075) by means of furnace gases. It can then be evaporated in the usual apparatus without frothing.—L. A. C.

Hemp; Process for manufacturing an elastic imitation —. T. Honda, Assr. to H. Nishimura. U.S.P. 1,351,385, 31.8.20. Appl., 3.7.19.

SEE E.P. 130,590 of 1919; J., 1920, 444 A.

[Paper pulp;] *Beating and refining engine [for —].* T. H. Nash. U.S.P. 1,351,962, 7.9.20. Appl., 3.6.19.

SEE E.P. 135,733 of 1919; J., 1920, 60 A.

Proofing of fabrics. R. Pilkington, and J. Mandleberg and Co., Ltd. E.P. 149,791, 26.5.19.

Coated fabric. U.S.P. 1,352,163. See XIV.

p-Cymene as solvent. Wheeler. See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Printing fabrics with basic dyestuffs. A. Stiegler. Sealed note No. 2035, Bull. Soc. Ind. Mulhouse, 1920, 86, 173—174. Report by C. Sunder. *Ibid.*, 174—175.

THE fixation of basic colours in printing may be advantageously carried out by using lead acetate instead of antimony salts. Cloth is printed with a paste containing 30 g. of basic dye, 100 g. of lactic acid, 500 g. of starch-tragacanth thickening, 180 g. of 50% tannic acid solution, 20 g. of lead acetate, and 170 g. of water; steamed for 1 hr., washed and soaped, and finally chemicked. The lactic acid prevents the formation of the lead colour lake in the printing paste. The colours so obtained are pure, bright, and as fast to washing and light as those obtained with antimony salts. The shades are also heavier. For naphthol-prepared cloth, a steaming for 5 min. is sufficient. Sunder reports favourably on the process but, owing to the sensitiveness of lead salts to hydrogen sulphide, suggests that antimony salts be used in the soap bath.—A. J. H.

PATENTS.

Scouring fibres of vegetable origin; Process for — with the object of accelerating and facilitating subsequent bleaching. E. T. J. Watremez. E.P. 139,457, 25.11.19. Conv., 24.2.19. Addn. to 135,516 (J., 1920, 568 A).

FOR scouring in the cold it is advisable to omit completely the reducing salt from the bath (*loc. cit.*), which may be made by adding in the cold 1 kg. of zinc oxide to a mixture of 40 l. of caustic soda of 30°—35° B. (sp. gr. 1.262—1.320) and 30 l. of Eau de Javelle of 16°—18° B. (sp. gr. 1.125—1.143) and diluting to 1000 l. The fibres are steeped in this solution for 12—24 hrs., rinsed, immediately soured at the boil, rinsed, and then subjected to the bleaching process proper.—A. J. H.

Raw [grege] silk; Process of dyeing —. E. L. Maupai. U.S.P. 1,352,014, 7.9.20. Appl., 14.6.20.

GREGE silk is hardened by immersion in a solution of a metallic salt, washed, and dyed with vat colours.—A. J. H.

Silk fabrics; Machines for finishing —. G. P. Vuono. E.P. 149,842, 27.8.19.

Drying of fabrics [after finishing]. W. Backer. E.P. 150,631, 3.3.20.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Pyrophosphoric acid; Third and fourth dissociation constants of —. I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 474—478.

A COLORIMETRIC method is described for the determination of the fourth dissociation constant of pyrophosphoric acid. Thymolphthalein is used as an indicator, and standard solutions of known hydrogen ion concentration consisting of carbonate-bicarbonate mixtures. The value $K_4 = 4.6 \times 10^{-9}$ was found. Other measurements in mixtures of the trisodium and the tetrasodium salts gave $K_4 = 3.6 \times 10^{-9}$. For the third dissociation constant, $K_3 = 7.6 \times 10^{-7}$ was found.—W. S. M.

Alkali chloride solutions; Behaviour of concentrated — during evaporation, in presence of alkali hydroxides. C. Chorower. Z. angew. Chem., 1920, 33, 201—203.

IN connexion with the recovery of potassium salts from vinasses ash by treatment with lime and barium hydroxide, followed by suitable decomposition of the salts, the solubility of alkali chlorides in presence of hydroxides was investigated. An experimental liquor had the following composition:—Cl, 3.559%; OH, 0.9278%; K, 4.792%; Na, 0.933%; S, 0.0591%; SO_4 , 0.0321%; CO_2 , 0.0705%. The liquor was evaporated in a 4-l. vessel, fresh liquor being added at intervals, and the separated salts removed periodically. At various stages, as deposition of salts ceased, the boiling point was taken, after which the specific gravity was determined in a modified pycnometer provided with an asbestos filter, and the sample then analysed. Samples taken after 15, 20, 35, 41, and 48 l. respectively of solution had been evaporated showed boiling points increasing from 110° to 118° , and sp. gr. from 1.295 to 1.370. The chlorine ions diminished from 12.58 to 7.661 g. per 100 g. of solution, and the potassium ions remained almost constant at approximately 17 g. All other ions increased progressively. The figures showed the value Na concentration/OH concentration to be a constant, and the product $Cl \times OH$ also attained a constant value. The author discusses the explanation of the rising Na value and stationary K value by the application of the phase rule.—W. J. W.

Halogens; Solubility of — in the corresponding hydroacids and alkali salts. II. E. Oliveri-Mandalà. Gaz. Chim. Ital., 1920, 50, II., 89—98.

THE solubilities of chlorine in sodium chloride solutions and hydrochloric acid, and of bromine in hydrochloric and hydrobromic acids, have been investigated. (Cf. J.C.S., ii., 614.)—T. H. P.

Carbon dioxide; Determination of — in water-insoluble carbonates. C. S. Robinson. Soil Sci., 1920, 10, 41—47.

DETERMINATIONS were made of the carbonate content of samples of limestone, marl, and soil. The first method used was that of Van Slyke (J., 1919, 40 A), in which the sample was decomposed in a partially evacuated flask containing standard

barium hydroxide. The second method used was a gasometric one, originally proposed for soluble carbonates by Van Slyke (J., 1917, 91D). A special form of finely-graduated cup nitrometer was used, with a tube and special stopper holding the weighed sample introduced between the bottom of the nitrometer and the levelling tube which contained mercury. The apparatus having been rendered air-free, hydrochloric acid is introduced from the cup into the nitrometer and allowed to run down and mix with the sample. After all the gas has been evolved, its volume is read and its weight calculated from tables prepared for the conditions of the method. The gasometric method was found to be the more accurate of the two, but the first method is to be preferred for samples which are low in magnesium carbonate.—J. H. J.

Thiocyanates; Volumetric determination of — with potassium permanganate. R. Meurice. Ann. Chim. Analyt., 1920, 2, 272—273.

THIOCYANATES may be determined by adding to their sulphuric acid solution an excess of standardised permanganate solution and then titrating the excess with hydrogen peroxide solution. The excess of permanganate added should be at least one-half of that required for the actual oxidation of the thiocyanate. Direct titration of thiocyanates with permanganate solution yields low results.—W. P. S.

Sulphuryl chloride; Synthesis of — in presence of organic compounds. G. Cusmano. Gaz. Chim. Ital., 1920, 50, II., 70—80.

IN general, ketones with one carbonyl group increase the velocity of combination of chlorine with sulphur dioxide to form sulphuryl chloride, this property disappearing when a halogen, nitro- or sulphy-group is introduced into the molecule at any distance from the carbonyl group; the presence of carboxyl is without effect. In $\alpha\alpha$ -diketones, one of the carbonyl groups may influence the other in the same way as a negative group does. The lack of accelerating action exhibited by the quinones and certain other compounds is ascribed to the formation of stable compounds with either the chlorine or the sulphur dioxide.—T. H. P.

Barium peroxide; Reactions of — with other oxides. Investigations on some rapid low temperature reactions by means of heating curves. J. A. Hedvall and N. von Zweigbergk. Z. anorg. Chem., 1919, 168, 119—136.

THE reactions between barium dioxide and a large number of other oxides were studied by means of heating curves, as described in a previous paper (this J., 1919, 11 A). Of the oxides examined only SnO , SnO_2 , and ZrO_2 had no action at all. Purely catalytic decomposition of the barium dioxide was brought about by MgO , CaO , CuO , CdO , La_2O_3 , and CeO_2 . In other cases the decomposition of the dioxide was followed by combination with the barium oxide formed, e.g., with ZnO , Al_2O_3 , and TiO_2 . Cu_2O and Bi_2O_3 were oxidised by the barium dioxide, and with the following oxides oxidation occurred followed by combination with the reduced barium oxide:— As_2O_3 , Sb_2O_3 , Sb_2O_5 , Cr_2O_3 , U_3O_8 , MnO , Mn_2O_3 , Mn_2O_5 , MnO_2 , Fe_2O_3 , NiO , and CoO . In some cases the reaction took place with great violence, notably with Cu_2O , V_2O_5 , Sb_2O_3 , Cr_2O_3 , MoO_3 , and MnO . (Cf. J.C.S., 1920, ii., 35.)—E. H. R.

Hydrogen peroxide; Electrolytic production of —. F. W. Skirrow and E. R. Stein. Trans. Amer. Electrochem. Soc., 1920, 69—79. [Advance copy.]

RESULTS obtained by the electrolytic decomposition of sulphuric acid, and of sodium and potassium bisulphate solutions, showed that the current efficiencies with which persulphuric acid was formed

were about the same in all three solutions. Platinum, with the possible exception of manganese dioxide, alone affords a suitable anode material. Sodium persulphate solutions can be produced of a much higher concentration than can persulphuric acid, without loss of current efficiency. Distillation of potassium persulphate with sulphuric acid and water gave reasonably high yields—about 80%—of hydrogen peroxide. In the case of sodium persulphate, in no case did distillation yield more than 9% of the theoretical amount of hydrogen peroxide. Distillation of persulphuric acid or sodium persulphate solutions would need to be preceded by extremely careful removal of all catalytic impurities.

—J. S. G. T.

Liquid air; Rectification of —. E. Barbet. *Chim. et Ind.*, 1920, 4, 147—150.

In liquid air rectifiers in which no means are provided for drawing off argon, it is impossible to attain absolute purity of the nitrogen or oxygen. It is fallacious to assume that nitrogen, showing 0.1–0.2% O₂ on analysis, is of 99.9–99.8% purity, as this content of oxygen implies an even higher proportion of argon, the latter being more volatile and not so readily arrested by the reflux condensers. Removal of argon from the rectifying column may be effected with even more facility than the separation of amyl alcohol from ethyl alcohol, and the employment of regulators and thermometers for this purpose is essential. Argon of any selected concentration may be drawn off continuously at certain plates of the column, preferably the lower ones, without tedious manipulation of the rectification process or multiplication of control tests.

—W. J. W.

Weak acids and bases. Kolthoff. *See* XXIII.

Potassium and sodium. Quartaroli. *See* XXIII.

Mixtures of gases. Weaver and Palmer. *See* XXIII.

PATENTS.

Sulphuric acid; Utilisation of the waste gases evolved in the concentration of —. H. Petersen. G.P. 302,534, 23.2.17.

THE waste gases are cooled by using them to effect a preliminary concentration of cold sulphuric acid, and are then passed into the lead chambers, in place of the water or steam commonly employed.

—J. S. G. T.

Furnace for carrying out endothermic gas reactions [e.g., production of nitric oxide]. Elektrochem. Werke, G.m.b.H. E.P. 116,503, 5.6.18. Conv., 23.3.17. Addn. to 5281 of 1911 (J., 1911, 812).

In an electric arc furnace constructed in accordance with the former patent, a double-walled, water-cooled metallic tube passes through the upper cover of the furnace. The gases from the furnace pass directly from the arc zone into this tube, which is of such dimensions that the gases leave it at about 1200°–1500° C. The concentration of nitric oxide is thereby increased to 2.5%, compared with 1.5% hitherto attained.—J. S. G. T.

Nitric oxide; Production of — in gas engines. F. Gerhardt. G.P. 321,981, 9.12.15. Addn. to 303,255 (J., 1919, 891 A).

THE exhaust gases of the engine are utilised to pre-heat the air or gas stream employed to produce turbulence of the charge.—J. S. G. T.

Gaseous reactions; Catalytic process for effecting — more especially for the production of oxides of nitrogen from mixtures of ammonia and air or oxygen. Permutit A.-G. G.P. 298,981, 2.5.15.

THE gaseous mixture is passed over material of the

nature of aluminate-silicates, in which the aluminium has been displaced by one or more elements, the oxides of which are soluble in alkali; in addition, or alternatively, the alkali of the aluminate-silicate may be entirely replaced by one or more heavy metals. Natural base-exchanging silicate minerals or artificially produced aluminosilicates of heavy metals, such as chromium, manganese, cobalt, nickel, etc., may be employed. A product containing cobalt and tungsten when employed as catalyst affords practically a quantitative yield of chlorine by the oxidation of hydrochloric acid; and a product containing nickel and boron may be used for the oxidation of a mixture of sulphur dioxide and air to sulphur trioxide.

—J. S. G. T.

Ammonia; Synthetic production of —. H. C. Greenwood. U.S.P. 1,312,534, 12.8.19. Appl., 15.2.18.

HYDROGEN and nitrogen are passed over a heated catalyst at a velocity of a million litres or more per hour for each litre of catalyst space. Using 1½ million litres of the mixture at 100 atm., yields of about 15 kg. of ammonia per hr. per litre of catalyst space may be obtained. It is advisable to use 10–20% excess of nitrogen.—W. J. W.

(A) *Ammonia; Production of* —. (B) *Ammonia synthesis and catalyst therefor*. (C) *Ammonia synthesis*. (D, E, F) *Production of ammonia*. J. C. Clancy, Assr. to The Nitrogen Corp. U.S.P. (A) 1,352,174, (B) 1,352,178, (C) 1,352,179, (D) 1,352,180, (E) 1,352,181, and (F) 1,352,182, 7.9.20. Appl., (A) 23.3.16, (B) 17.3.19, (C) 8.9.19, (D, E, F) 14.1.20.

A GASEOUS mixture containing nitrogen and hydrogen is brought under high pressure into contact with a catalyst comprising (A) an alkali metal cyanamide; (B) a metal capable of forming a nitride at an ammonia-forming temperature, together with a compound of an alkali metal and carbon or an alkali metal intimately associated with carbon, manganese, and iron; (C) a compound containing an atom of carbon united to two nitrogen atoms (at a temperature below 550°); (D) a cyanamide composition including cobalt; (E) a cyanamide composition containing manganese, or (F) a cyanogen compound containing potassium.—C. I.

Cyanogen compound of an alkali metal; Process of obtaining the —. J. C. Clancy, Assr. to The Nitrogen Corp. U.S.P. 1,352,175, 7.9.20. Appl., 13.7.17.

AN alkaline-earth carbonate mixed with a cyanide is acted upon by free nitrogen yielding an alkaline-earth cyanide. This is converted into an alkali cyanide by interaction with an alkali carbonate.

—C. I.

Metallic cyanides; Process and means for producing —. C. P. Hidden, Assr. to Nitrogen Products Co. U.S.P. 1,352,192, 7.9.20. Appl., 18.11.18.

A MIXTURE of carbon, alkali carbonate, and catalytic material is caused to travel down a furnace-heated retort in presence of an ascending current of nitrogen. The heating is regulated so that no appreciable formation of cyanide takes place until the charge has reached the lower portion of the retort, at which stage the temperature is suddenly raised, the cyanide produced being rapidly cooled as it emerges to prevent loss from volatilisation.

—W. J. W.

Nitrogen; Process for fixing atmospheric —. C. P. Hidden, Assr. to Nitrogen Products Co. U.S.P. 1,352,193, 7.9.20. Appl., 5.7.19.

FOR the fixation of nitrogen by means of a porous charge, constituents of which are capable of combining with free nitrogen to form a cyanide and

carbon monoxide, the charge is first heated to a cyanide-forming temperature by direct gaseous convection, the gas employed being such as to prevent formation of cyanide. Nitrogen, preheated to the reaction temperature, is then introduced, after which cool nitrogen is passed in to remove the carbon monoxide formed, and at the same time to cool the product below the temperature at which carbon monoxide reacts on the cyanide.—W. J. W.

Potassium sulphate; Manufacture of —. Fabr. de Prod. Chim. de Thann et de Mulhouse. E.P. 137,519, 30.12.19. Conv., 30.12.18.

POTASSIUM sulphate is prepared by the action on finely-powdered potassium chloride of sulphuric acid of such strength (70–72% H_2SO_4) as to render the moist mixture as pulverulent as possible. The mixture is heated in three stages, first at 100°–120° C., with stirring, then at 300° C., and finally at 800° C. By using a slight excess of sulphuric acid a sulphate is obtained almost or quite free from chloride, especially if iron is replaced by brick in the furnaces used for the last two stages. Potassium bisulphate may be substituted for sulphuric acid.—C. I.

Rock salt brine or solutions of rock salt in water; Removal of calcium and magnesium from —. F. A. Freeth and L. A. Munro. E.P. 119,707, 15.2.19.

THE usual process for the purification of brine (L. Mond, E.P. 4175 of 1874 and 3599 of 1878) is modified by adding the sodium carbonate and calcium hydroxide with gentle stirring, the reagents being maintained at 25° C. The precipitate thus formed settles readily.—C. I.

Hypochlorites; Electrolytic apparatus for the direct production of —. P. Pestalozza. E.P. 150,214, 19.2.19.

A VAT is subdivided into a number of cells by transverse partitions, the lower portions of which are constructed of the same material as the vat, the remainder being constituted of electrically conducting material serving as anode for one cell and cathode for the adjacent cell. The several cells communicate with one another by apertures in the partitions, these being staggered. A double series of cooling pipes, constituted preferably of glass, and built up of sections connected together by elastic couplings, is immersed in the cells, the one series traversing the even-numbered cells and the other the odd-numbered cells.—J. S. G. T.

Alumina; Process for the manufacture of —. J. Morel. E.P. 149,769, 17.5.19.

ALUMINIUM sulphate is obtained by treating bauxite with a mixture of sulphur dioxide, air, and steam at a temperature not above 800° C. in a Hargreaves or other suitable furnace. The mass is treated with hot water, the solution filtered, concentrated, heated to boiling point in a closed exhaust apparatus, and a solution of sodium thiosulphate added, $\text{Al}_2(\text{SO}_4)_3 + 3\text{Na}_2\text{S}_2\text{O}_3 = \text{Al}_2\text{O}_3 + 3\text{SO}_2 + 3\text{S} + 3\text{Na}_2\text{SO}_4$. The iron remains as ferrous sulphate in solution. The mixture of alumina and sulphur is washed, dried, and calcined. Part of the sulphur dioxide produced is utilised in the first stage of the process and part in regenerating sodium thiosulphate from sodium sulphide produced by reduction of sodium sulphate by carbon. The first stage of the process can be accelerated by passing the gas mixture over an oxide of iron catalyst or adding to the bauxite 1–2% of aluminium or other chloride.—C. I.

Aluminium nitrate; Preparation of —. J. J. Hood. E.P. 149,783, 20.5.19.

NORMAL aluminium nitrate is prepared by double

decomposition of aluminium sulphate with calcium, barium, ammonium, or potassium nitrate.—C. I.

Lead arsenate; Method of making —. E. O. Barstow, Assr. to The Dow Chemical Co. U.S.P. 1,349,707, 17.8.20. Appl., 28.8.18.

LEAD oxide, in excess, is mixed with lead acetate and the resulting basic lead acetate is separated. A solution of the basic acetate is continuously mixed with arsenic acid, lead arsenate being precipitated.—A. G.

Soda, etc.; Process of depositing —. W. F. Watterson. U.S.P. 1,350,128, 17.8.20. Appl., 8.9.19.

THE crystals are formed around a thin stick of relatively small mass compared with the mass of the crystals which grow upon it. The whole, including the stick, is finally broken up.—W. H. C.

Zirconium oxide; Method of obtaining —. L. E. Barton, Assr. to The Titanium Alloy Manufacturing Co. U.S.P. 1,351,091, 31.8.20. Appl., 24.9.19.

MATERIAL containing zirconium oxide is melted with a carbonaceous reducing agent, the zirconium cyanonitride produced being then caused to react with an alkali sulphate.—W. J. W.

Thorium; Recovery of —. L. W. Ryan, Assr. to Lindsay Light Co. U.S.P. 1,351,439, 31.8.20. Appl., 29.5.18.

THORIUM is precipitated as a metaphosphate from a dissolved thorium salt.—C. I.

Alkali-metal hydroxides; Process of producing —. S. S. Sadtler. U.S.P. 1,351,693, 31.8.20. Appl., 21.3.19.

SODIUM hydroxide is produced by interaction of disodium phosphate (284 pts.) and calcium hydroxide (148–222 pts.).—C. I.

Hydrogen sulphide; Process of manufacturing —. K. Ikeda. U.S.P. 1,351,755, 7.9.20. Appl., 12.11.17.

SULPHUR and fuel gas are caused to interact in the presence of a catalyst.—C. I.

Oxide or oxides of manganese; Method of producing —. B. F. Lovelace. U.S.P. 1,352,208, 7.9.20. Appl., 24.4.19.

A SOLUTION of ammonium carbonate is electrolysed in a single compartment cell at a temperature below 35° C., one of the anodes containing manganese.—B. V. S.

Sodium bicarbonate and hydrogen; Method of producing —. A. Nagelvoort, Assr. to The Nitrogen Corp. U.S.P. 1,352,211, 7.9.20. Appl., 7.2.20.

IN the ammonia-soda process the ammoniated brine is treated with carbon dioxide diluted with hydrogen. The presence of the hydrogen keeps down the temperature and thus favours the formation of large crystals of sodium bicarbonate. The separated hydrogen is collected for use in the synthetic preparation of ammonia.—B. V. S.

Hydrogen peroxide; Process for stabilising solutions of —. A. Queisser. G.P. 321,616, 20.4.19.

WATER-SOLUBLE complex salts of salicylic acid with borax or disodium phosphate are added to solutions of hydrogen peroxide.—L. A. C.

Carbon and the like; Production of substantially pure —. R. Williams, Assr. to Nitrogen Products Co. U.S.P. 1,352,162, 7.9.20. Appl., 24.1.18.

CARBON monoxide is converted into carbon dioxide and free carbon by heating at a temperature below

700° C. in the presence of a catalyst, the latter being agitated to facilitate the conversion.

—D. F. T.

Aluminous materials; Process for purifying —. Carborundum Co., Ltd. From O. Hutchins, E.P. 150,116, 18.6.19.

SEE U.S.P. 1,310,342 of 1919; J., 1919, 631 A.

Nitrogen; Apparatus for fixation of —. E. C. R. Marks. From Nitrogen Products Co. E.P. 150,127, 23.6.19.

SEE U.S.P. 1,321,892 of 1919; J., 1920, 21 A.

Nitric oxide. E.P. 122,173. See XI.

VIII.—GLASS; CERAMICS.

Refractories; Physical characteristics of specialised —. (Part 3) *Electrical resistivity at high temperatures*. M. L. Hartmann, A. P. Sullivan, and D. E. Allen. Trans. Amer. Electrochem. Soc., 1920, 1—15. [Advance copy.] (See also J., 1920, 365 A.)

THE electrical resistivity of nine commercial refractories was determined. Fireclay bricks of grade A quality and coarse texture, bauxite bricks, and silica bricks with a lime-bond gave regular resistivity curves. Carborundum bricks containing 95% of "carbofrax C" and 5% of bond gave similar curves, the resistivity being constant when the bricks were maintained at 1500° C. "Carbofrax B," on the contrary, showed a rapid decrease of resistivity with increasing time of heating. Recrystallised carborundum ("Refrax") gave quite regular curves, the resistivity being low at all temperatures. With magnesia bricks there was a marked difference between the resistivity curves during heating and cooling and a peculiar flattening of the heating curves (indicating a constancy of resistance) between 1000° and 1150° C., which appears to indicate a physical change in the structure of the magnesia. The cooling curves did not show this flattening. The difference between the resistivity curves during heating and cooling of bricks of natural zirconia is very marked. On heating, the resistivity fell very rapidly until a temperature of 1200° C. was reached, after which it fell very slowly, indicating that a permanent change occurs on heating zirconia to 1200° C. Coarse-grained chrome bricks possessed low resistivities at all temperatures, the resistivity remaining practically constant between 1100° and 1200° C., increasing between 1200° and 1350° C., and again decreasing from 1350° to 1500° C. The cooling curves differ greatly from the heating curves. A table showing the resistivities of the materials examined when cold and at various temperatures between 800° and 1500° C. is given.

—A. B. S.

Porcelain [fired at] Seger cone 7—9. T. Hertwig. Sprechaal, 1920, 53, 363—365.

PORCELAINS maturing at cone 7—9 should be used extensively in the future on account of the saving in fuel as compared with those fired at cone 13—14, but to obtain the best results the ovens must be re-designed and additional fires employed. All German hard porcelains may be biscuit-fired at cone 9 when very finely ground. In soft French porcelains the clay:quartz ratio is less important than in the harder German product. Porcelains fired at cone 7—9 have a slight bluish tinge if quartz is used, but this disappears if flint is substituted. A number of formulæ for bodies and for glazes of various colours are given. The materials used for a body for glazed porcelain should not be ground for more than 24 hrs., as if it is too finely ground the glaze subsequently applied will craze.

Materials for bodies which are not to be glazed may be ground for 70—100 hrs.—A. B. S.

Ultra-filtration. Schmitt. See I.

PATENTS.

Clay; Preparation of — for shaping in the dry state under high pressure with the aid of heat. Chem. Lab. für Tonindustrie und Tonindustriezeit. H. Seger & E. Cramer. G.P. 321,930, 20.1.15.

THE plasticity of clay is destroyed by heating to a temperature above that ordinarily used for drying but below the firing temperature. The product when subjected to high pressure and then fired forms a strong mass suitable for bricks.—A. B. S.

Enamel coating; Producing an — on vessels and the like. C. Wagner. G.P. 321,840, 12.4.19.

A MIXTURE of borax, bronzite, and black copper oxide applied to the surface of the vessels gives on firing a crystalline bronze appearance of red, yellow, or green colour.—L. A. C.

Glass surfaces; Process for treating —. T. Ueda. E.P. 144,737, 12.6.20. Conv., 20.11.17.

SEE U.S.P. 1,306,505 of 1919; J., 1919, 579 A.

Drying crucibles [for melting steel and other substances]; Apparatus for —. L. Vergniaud. E.P. 149,703, 22.10.18.

IX.—BUILDING MATERIALS.

Portland cement, hydraulic lime, and volcanic ashes; Long-time tests of —. I. Hiroi. J. Coll. Eng., Tokyo Imp. Univ., 1920, 10, 155—172.

PARTICULARS are given of an investigation commenced over 20 years ago in connection with the manufacture of concrete blocks for harbour works. No case of decomposition of cement by the chemical action of sea-water alone has been observed, except where defective cement was used. It is concluded that Portland cement of good quality, properly gauged, can be made to resist the action of sea-water for an unlimited time. "Neat" cement briquettes attained their maximum strength in less than a year and then rapidly became weaker, in some cases losing their tensile strength entirely in four or five years, though still remaining intact and even showing increased compressive strength. The tensile strength in air and in fresh water increased similarly at first, but after the initial rise the changes were irregular, although showing a tendency to gradual increase. "Neat" hydraulic lime gave similar results in air and in fresh water, but attained a maximum strength in salt water after three years, after which a decrease occurred, with total disintegration in 6—7 years. Prolonged tests of mortar made with 1 part of cement to 2 of sand showed progressive increases in strength with age, no sign of disintegration being observed; the ultimate tensile strengths attained were about 85 and 50 kg. per sq. cm. in air and in sea-water respectively. Similar tests with hydraulic lime mortar indicated an ultimate maximum tensile strength of about 20 kg. per sq. cm. With 1:3 cement-sand mortar the mean final strengths after 22 years in air, fresh water, and sea water respectively were as follows: Tensile, 70, 42, 37; crushing, 620, 500, 350 kg. per sq. cm.; 1:3 hydraulic lime mortars gave similar results. Tests of the crushing strength of mortar briquettes made with coarse beach sand, "standard" sand, and sand which passed through a sieve with 220 meshes per sq. cm. confirmed the superiority of the coarser sands, which gave results 50% or more higher than the fine sand. The strength of mortars in sea

water appears to be nearly in inverse ratio to the proportion of sand present; the most regular increases in strength were shown with 1:2 mortar. The use of volcanic ashes in mortar is of no advantage in air, but in sea water considerably increased strength results from using properly proportioned mixtures of suitable ash with cement and sand. Comparative tests of briquettes prepared by ramming the mortar mixtures into moulds and by pouring indicated that the early superiority of rammed mortar is maintained throughout during use. The proportion of water used appears to have no ultimate effect on the strength of mortar.

PATENTS.

Cement and the like; Rotary kilns for burning — J. S. Fasting. E.P. 149,761, 17.5.19. Conv., 14.6.18.

THE inner surface of a horizontal rotary kiln is increased between the feeding end and the sintering zone by corrugating the surface in the form of circumferential rings. The rings absorb heat from the heating gases and transmit it by conduction to the material treated.—W. F. F.

Kilns. E.P. 150,157. See I.

Wood-pulp liquor. E.P. 150,571. See V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Blast furnaces; Variations in the heat supplied to — and their effect on the fuel consumption. W. W. Hollings. Iron and Steel Inst., Sept., 1920. [Advance proof.] 12 pages.

WHEN an addition is made to the supply of heat to a blast furnace the economy in fuel exceeds the theoretical equivalent of the heat added. It is shown that any variation in the heat supplied involves a secondary series of changes in the heat developed and absorbed within the furnace, and the ultimate calorific effect is the sum of an infinite geometric series of which the primary variation is the first term. The theory is the outcome of an attempt to explain the unsatisfactory results obtained at the Brymbo steel works with a dry air blast, and it is shown that for countries with such a low average moisture as England it does not pay to instal the dry-blast. Crushing of the charge to a uniform size may be expected to result in a considerable saving of fuel. The process of blowing with air slightly enriched with oxygen is not likely to result in an appreciable economy.

—T. H. Bu.

Electric furnace; Consumption of energy by the — in the manufacture of pig iron etc. A. Coutagne. Rev. Mét., 1920, 17, 450—468.

A LARGE amount of experimental data obtained in various operations using a four-phase electric furnace of 800—1000 kw. capacity is given. The energy required for the reduction and fusion of iron in the manufacture of pig iron from ore containing 45—55% Fe and from turnings and scrap containing 90% Fe, for the reduction and fusion of manganese in the manufacture of 75% ferromanganese, and for the reduction and fusion of silicon in the manufacture of ferrosilicon of various grades from iron scrap and turnings is fully considered. In the silicon experiments the questions of capacity of furnace and the richness of the alloys produced are dealt with, as is also the loss of silicon as silica by volatilisation. The consumption of energy required in the manufacture of the ternary alloys of iron, silicon, and manganese from ore and scrap iron is also given. Comparison of the experimental data with theoretical data shows that the useful electric

thermic effect for an electric furnace of 800—1000 kw. capacity is near 60% and for a furnace of 1000 kw. capacity about 70%. Comparative thermal balance sheets show that the electric furnace working with and without gas compares favourably with a blast furnace working under similar conditions.—J. W. D.

Steels; Electric — C. G. Carlisle. Iron and Steel Inst., Sept., 1920. [Advance proof.] 23 pages.

ANALYSES of electric furnace slags and the accompanying metal confirm the contention that the slag must be free from metal oxides before sulphur in any considerable degree can be transferred from metal to slag. The elimination of sulphur is greater the smaller the furnace. Phosphorus cannot be reduced in like degree. Production of sound metal depends upon the control of slags especially during the refining period. Results of tests on electric steels made to Air Board specifications are given. On an equal number of test-pieces a greater percentage of success was obtained than with Siemens steels of the same composition. Compression tests in which the load was applied quickly in order to find out if the steel would resist shock without breakdown showed up clearly the value of the electric steels, and workshop practice over a long period bore out the compression test results. Electric steels gave higher crushing loads and yield points than crucible steels. Particulars of practical tests with electric steel dies and other tools are given. A high-speed drill of electric steel gave record results. Tests on an aeroplane exhaust valve of electric steel showed a marked increase in strength at temperatures over 800° C. In the author's opinion a 15—20-ton electric furnace would, under steady conditions, produce as cheaply as a 50-ton acid Siemens furnace.—T. H. Bu.

Steel; Decaridation of — with hydrogen. J. H. Whiteley. Iron and Steel Inst., Sept., 1920. [Advance copy.] 14 pages.

HEATING a section of a steel plate in hydrogen at temperatures below 950° C. failed to remove the banded structure or micro-ghost lines (J., 1920, 452 A). Samples of Armeo iron containing numerous visible particles identified as iron oxide (FeO) and iron oxide-sulphide eutectic were subjected to the action of hydrogen at various temperatures, the experiments being made with small sections and also with strips 1/400 in. thick. Oxide was removed very slowly below 950° C., but the rate increased with the temperature. At 1300° C. the thin strips appeared to be decaridised in 1 hr. With thin turning the speed of decaridation was greater, probably due to the numerous cracks. It was found that one steel was decarburised and another carburised under the same conditions in hydrogen. To determine correctly oxygen as oxide in steel a temperature of 1000°—1200° C. is necessary. The drillings or turnings should be as fine as possible, and the possibility of the evolution of carbon monoxide and dioxide must be borne in mind.—T. H. Bu.

Cast steel; Studies of the macrostructure of — F. G. Allison and M. M. Rock. Chem. and Met. Eng., 1920, 23, 383—389.

THE influence of the temperature at the time of pouring and the conditions of cooling etc. as revealed by the inspection of photographs of polished and etched surfaces and the conditions of dendritic growth are discussed. A number of photographs are included in the paper.—W. H. C.

Carbon steels heated in vacuo; Surface changes of — E. H. Hemingway and G. R. Ensminger. Amer. Inst. Min. Eng., Aug., 1920. Chem. and Met. Eng., 1920, 23, 366.

AFTER heating a steel containing 1.01% C in *vacuo*.

for 6 hrs., at 1000° C., there appeared on the surface, when examined at 500 magnifications, delicate pearlitic grains representing a structure stable below the critical point. Independent of this structure was a second system consisting of deeply marked polygonal crystals representing γ -iron boundaries, and also a third system independent of either, which was thought to represent the boundaries of former γ -crystals which had been absorbed by crystalline growth. This outside layer of carbon-free iron was very thin, although with steels of lower carbon content a layer of greater depth was obtained. A study of the deposits found inside the vacuum tube suggested that this outside layer consisted of ferrite volatilised at the high temperature and redeposited below the temperature at which solid solution exists. Any iron oxide existing on or in the steel would tend to cause decarburisation by breaking down the cementite.

—J. W. D.

Steel; Nitrogen in — and the erosion of guns.

H. E. Wheeler. Amer. Inst. Min. Eng., Aug., 1920. Chem. and Met. Eng., 1920, 23, 366.

SMALL steel cylinders of various compositions were heated in ammonia for 20 hrs. at 650° C. and then cooled in the same atmosphere. Nascent nitrogen from such treatment forms Fe_3N , which is stable below 450° C., and penetrates the steel. Carburisation also occurs in low-carbon steel by pyridine or hydrocarbon impurities in the gas, while decarburisation takes place in hypereutectoid steels. In alloy steels the alloying elements do not combine with the nitrogen, but affect the penetration only as they affect the carbide. White cast-iron becomes coated with a layer of solid Fe_3N containing 1% C. Cold work greatly assists the penetration, the work apparently shattering the cementite so that it is more easily dissolved and retained in solid solution by the combined action of heat and nitrogen. These results were so similar to the phenomena accompanying gun erosion that it is suggested that the hard white layer on the inside of an eroded gun is a locally cold-worked austenitic case due to the penetration of nitrogen at the high pressure and temperature produced during explosion.—J. W. D.

Flakes [in nickel steel]; Cure of — by proper heat treatment. C. S. Crouse. Chem. and Met. Eng., 1920, 23, 329–332.

FLAKES and streaks in nickel steel forgings due to cooling strains were practically eliminated by casting in octagonal instead of in square moulds, stripping the ingots, and cooling slowly and uniformly in ashes instead of quickly in air. Flakes due to strains set up in the forgings during heat treatment were reduced considerably by adopting the following modified procedure. All forgings were preheated to 500°–700° F. (260°–370° C.) before charging. Large forgings were then raised to 1475° F. (800° C.) in 8–12 hrs., held at that temperature for 1½ hrs., and then quenched in water for 1½ min. On withdrawing from the water they were charged into a tempering furnace heated to 900°–1050° F. (480°–570° C.), raised to that temperature in 2–4 hrs., maintained uniform for 3 hrs., then withdrawn to a closed pit and cooled to below 600° F. (315° C.), with the pit covers closed. Small forgings were subjected to a similar treatment except that they were quenched in water for 1 min. only from a temperature of 1500° F. (820° C.) after being kept for 2 hrs. at this temperature. By the above heat treatment failures were reduced to 15% of the total.—J. W. D.

Nickel-chromium steels; Temper-brittleness of —.

R. H. Greaves and J. J. A. Jones. Iron and Steel Inst., Sept., 1920. [Advance proof.] 48 pages.

On reheating tough and brittle specimens of various

nickel-chrome steels in pairs to temperatures within the embrittling range, identical Izod impact figures were reached in a period of time which increased the lower the reheating temperature. The embrittling range was lower for electric than for open-hearth steels. Tempering within or below the embrittling range introduced two opposing tendencies—a softening effect tending to increase the impact figure and an embrittling effect tending to decrease it. Short periods of tempering (less than 30 mins.) did not induce brittleness. The order of susceptibility of various steels to develop temper brittleness is: acid open-hearth steel, electric steel, crucible steel. Of nickel chromium steels the softer steels are the least susceptible. As the temperature of hardening is raised the impact figure of the tempered steels becomes distinctly lower. Vanadium had no effect on susceptibility, and chromium steels showed the same order of brittleness as nickel-chromium steels. Molybdenum steels showed very slight susceptibility. Nickel steels showed various degrees of susceptibility, and carbon steels with less than 0.045% S and P showed no tendency to develop brittleness. Even in the unhardened state nickel-chromium steels showed a tendency to brittleness. Heating in carbon dioxide caused a decided reduction of susceptibility. Heating and cooling curves showed no certain indication of a critical point in the embrittling range. The density of steel after water cooling is a trifle less than after slow cooling. The electrical resistivity of tough and of brittle material is identical. There is no apparent difference in the hardness of the two varieties. The elastic limit of the water-cooled specimens is generally lower than that of the slowly cooled steel.—T. H. Bu.

Uranium steels. E. Polushkin. Rev. Mét., 1920, 17, 421–437.

A SERIES of experiments carried out on steels containing increasing quantities of uranium up to 7% show that the Acl point is not affected, while the Ar1 point is not affected up to 2% U, but is slightly lower with increased quantities. Uranium is present in the steels either as oxide or carbide. Under the microscope the oxide has a dark-grey colour with a slightly bluish tint, and the carbide on heat tinting acquires characteristic blue, green, and violet colours, which become bluish-grey if the heating is prolonged. The carbide is hard, well-crystallised, contains small white crystals, and does not enter into solid solution with iron even at 1250° C. It has a tendency to produce a pearlitic structure which becomes dark with badly formed lamellae as the quantity of uranium increases. The formula of the carbide is probably UC. Mechanical tests indicate that with low carbon content uranium raises the elastic limit and the ultimate stress, while the ductility remains unaffected or is slightly increased. When the carbon exceeds 0.6%, however, the increase in the elastic limit and ultimate stress is accompanied by a lowering of the ductility. Uranium increases the hardness but does not influence the impact or alternating tests. The properties of alloy steels were apparently not greatly improved by the addition of uranium, although nickel-uranium steels appeared to have a ductility superior to that of ordinary nickel or nickel-chromium steels.—J. W. D.

Galvanised iron; Deterioration of — by smoke.

Wislicenus. Z. angew. Chem., 1920, 33, 204.

Tests by Siedler (Rauch und Staub, 1920, 10, 43) show that galvanised iron is unsuitable for roofing in districts where it is exposed to smoke and gases from factories, railways, and house chimneys. Sulphur dioxide, in presence of moisture and oxygen, attacks it rapidly; on the other hand, dry sulphur dioxide has only a slight effect. A mixture of sulphur dioxide and carbon dioxide is very cor-

rosive; moist carbon dioxide alone is less so. Microscopical examination of corroded galvanised iron shows the presence of minute cavities and of sulphate containing ferric oxide, the formation of which is due to galvanic action or to actual solution of the zinc coating. Soot may be a contributory factor to corrosion by acting as a catalyst, and deterioration may also be brought about by the action of sulphuric acid formed by oxidation of the sulphurous acid in the gases.—W. J. W.

Zinc; Extraction of — in the open-hearth furnace with simultaneous recovery of zinc oxide. R. W. Müller. *Stahl u. Eisen*, 1920, 40, 1193—1196.

A CHARGE made up of 2000 kg. of pig iron, 3000 kg. of scrap and bars, and 8000 kg. of galvanised iron scrap, together with 100—150 kg. of coke, was melted in a 12—15-ton open-hearth furnace. The zinc was volatilised and oxidised and passed out with the outgoing hot gases as zinc oxide vapour. The gases leaving the furnace at 1500° C. were drawn by means of an exhaustor through a tubular cooling arrangement and were cooled to 150° C. From the exhaustor the gases passed through a settling chamber which served the double purpose of cooling the gases still further, and of reducing their velocity before passing to the filtering apparatus. A large proportion of the zinc oxide was deposited on the floor of this chamber, the remainder being recovered in the filtering chamber, which consisted of a large number of iron pipes containing bags of fine texture. The zinc oxide recovered had the composition 96.61% ZnO, 2.07% PbO, 1.28% Fe₂O₃, 0.04% MnO, 0.19% SO₂. —J. W. D.

Tin plating from alkaline tin baths by the use of addition agents. F. C. Mathers and W. H. Bell. *Trans. Amer. Electrochem. Soc.*, 1920, 41—44. [Advance copy.]

COPAIBA balsam, rosin, and to a lesser extent similar organic fatty acids or acid-containing substances were found to be satisfactory addition agents in producing smooth, adherent electrolytic deposits of tin from sodium stannite baths. The baths gradually deteriorated so that finally satisfactory deposits could not be obtained.—J. S. G. T.

Lead plating from sodium hydroxide lead baths by the use of addition agents. F. C. Mathers. *Trans. Amer. Electrochem. Soc.*, 1920, 35—39. [Advance copy.]

GUM sandarac, gum albanum, resin, and oleic acid were found to be satisfactory addition agents for diminishing the formation of loosely adhering crystals on the lead deposits from electrolytic baths of lead acetate dissolved in excess of sodium hydroxide solution. Other gums, resins, and fatty acids were also satisfactory. A suitable concentration of bath is: 7.3% of crystallised lead acetate, 20% of sodium hydroxide, 0.3—1% of addition agent. As high a temperature as possible should be employed. The anode current density should not exceed 1 amp. per sq. dm. A cathode current density of 1.5—2 amps. per sq. dm. may be employed. The deposits are less resistant to sulphuric acid than those afforded by acid lead baths. —J. S. G. T.

Antimony; Determination of — in lead-antimony alloys. L. Bertiaux. *Ann. Chim. Analyt.*, 1920, 2, 273—278.

FIVE g. of the sample (e.g., antifriction metal) is dissolved by heating with 40 c.c. of concentrated sulphuric acid and 10 g. of potassium sulphate, the solution cooled, diluted with 200 c.c. of water, 50 c.c. of hydrochloric acid and 2 drops of a 0.1% solution of Poirrier's Orange are added, and the

mixture is titrated with standardised permanganate solution. As soon as all the antimony has been oxidised the next drop of permanganate solution added reacts with the hydrochloric acid yielding free chlorine which at once bleaches the colouring matter. Lead, bismuth, tin, copper, and arsenic do not interfere; iron is titrated together with the antimony. The iron may be determined colorimetrically with thiocyanate in the titrated solution and an allowance made for its quantity.—W. P. S.

Solid state of aggregation; Theory of the —. A. Van der Werth. *Z. physik. Chem.*, 1920, 95, 129—138.

SPECIFIC heat and mechanical solidity both depend on molecular attraction and furnish a measure of the resistance with which a substance opposes any force which tends to disrupt it. It is possible to draw quantitative conclusions in connexion with the mechanical properties of solid substances, e.g., metals, from simple thermal data. (*Cf. J.C.S.*, ii., 583.)—J. F. S.

After-corrosion of firearms. Huff. See XXII.

Steel. Dovey. See XXIII.

PATENTS.

Electrical furnaces [for melting and refining steel].

F.I.A.T. Fabbr. Ital. Automobili Torino. E.P.

(a) 121,485, 12.12.18, and (u) 134,810, 10.3.19.

Conv., 12.12.17 and 2.11.18.

(A) The electrodes employed in an electric furnace for melting and refining steel etc. are protected from the point where they receive current to their entrance into the furnace, against the action of the outside air, by a telescopic tube the movable member of which is secured to a terminal clamp, and slides airtight against a fireproof ring secured to the fixed member, which is provided with a double wall for water circulation. Fireproof insulating rings are provided inside the fixed member. Means are provided for tilting the furnace for purposes of pouring etc., and for removing the electrode holders together with the roof of the furnace. The furnace hearth is made conducting by the addition of suitable substances to the refractory employed. (B) The electrodes are secured to their supports by means of an angle bar. The cooling cylinders rest upon the furnace roof and slide within guide rings provided at the lower ends of the uprights of the electrode holders.—J. S. G. T.

Electric furnaces. D. de Luca. E.P. 150,532, 2.9.19.

AN electric furnace adapted for the production of cast iron from scrap etc. comprises a metal frame covered with sheet metal and five arches of refractory brickwork, four of which are arranged to form the side walls of the furnace and rest on the fifth, which forms the base. The corners and sides of the furnace are reinforced by angle irons. The arches are lined with flat brickwork, and the intermediate space is filled with a protective refractory material, the brickwork lining being also covered with a protective coating. A filling of binding material is arranged between the arches and the metal sheet enclosing the entire brickwork. A tapping hole is arranged on one side of the furnace. The movable cover is provided with openings and devices for mounting the electrodes. —J. S. G. T.

Steel treatment; Process of —. **Steel treatment apparatus.** D. J. Simpson, Assr. to N. S.

Amstutz. U.S.P. (A) 1,351,494 and (u) 1,351,495, 31.8.20. Appl., 5.4.15. Renewed 29.12.19.

(A) STEEL is subjected to a continuously rising heat up to 1370° F. (740° C.) for a carbon content of 0.3% or 1270° F. (680° C.) for 0.65% of carbon; the application of heat is then arrested, the steel

subsequently heated to above the upper critical point, and finally quenched in a gaseous cooling medium. (b) A furnace for carrying out the process is described.—J. W. D.

[Iron-silicon] alloy. J. G. Homan, Assr. to W. H. Keller. U.S.P. 1,352,367, 7.9.20. Appl., 3.1.20.

AN alloy having an electrical resistance higher than that of iron consists of iron with 1–6% Si and 0.2–1% Mn, the amount of manganese added being directly proportional to the silicon content.

—A. R. P.

Silicon-iron alloys; Prevention of graphite precipitation in the manufacture of acid-resistant —. Maschinenfabr. Esslingen. G.P. 321,879, 4.9.18. Addn. to 306,001 (J., 1918, 628A).

THE silicon-iron alloy is melted in such an apparatus, that an increase in the carbon content is prevented, and not in a cupola, as described in the chief patent.—L. A. C.

Arsenious ores; Treatment of —. H. J. Wilson. E.P. 149,366, 3.1.19.

CRUSHED ore containing arsenic is fed by gravity into a roasting furnace through a preheater consisting of a tower having within it a tubular concentric passage through which the volatile products from the furnace pass to the condensing plant. The spent ore from the furnace passes through an air preheater. The impure arsenious oxides obtained are treated in an electro-thermal furnace consisting of a cylinder lined with vitreosil or fused quartz and wound with nickel chromium resistance wire and having a series of annular louvres over which the arsenious oxide is caused to pass; the resublimed oxide passes through the louvres into a central duct in communication with a condensing plant. The whole apparatus is portable and is intended for use at the place where the ore is mined.

—J. W. D.

Metals and metallic articles; Preservation of —. N.C.F. Jensen. E.P. 149,371, 19.2.19.

METALS or metallic articles are immersed in a mixture of mineral lubricating oil with 5% of paraffin, drained, subjected to a temperature of about 300° C. until the surface assumes a greyish tint, and then immersed in a mixture of equal parts of whale oil and mineral lubricating oil.—J. W. D.

Furnaces for melting and refining metals. H. E. Cohen and J. B. Catterall. E.P. 150,024, 22.5.19.

A FLAT-BED flame-heated furnace has at one side one or more doors, a fume-collecting hood adjacent to the doors, and adjustable side collectors, one for each door, adapted to envelop the doors so as to collect the fume and lead it to the hood. In the crown of the furnace is an opening communicating with the main hood.—J. W. D.

Furnace; Metallurgical —. D. E. Griffiths. U.S.P. 1,350,865, 24.8.20. Appl., 21.10.18. Renewed 16.2.20.

A ROTARY furnace has a stationary burner with downwardly directed ports for gaseous fuel and air. A blast-pipe with a number of tuyères extends longitudinally within the wall of the furnace. The rotation of the furnace automatically opens and closes communication between the tuyère openings and the blast-pipe. The walls of the furnace and the burner are cooled.—T. H. Bu.

Smelting-furnace. R. G. and W. R. Ward, Assrs. to Independent Mines Smelting Co. U.S.P. 1,351,451, 31.8.20. Appl., 4.10.16.

A RELATIVELY long chamber has the lower part of its walls curved downwards and inwards to form a furnace-hearth, which is provided with a central

elongated opening having a width greater than its vertical depth. Co-acting burners are arranged below the hearth in opposed pairs, the flames uniting at a point beneath the hearth opening and the concentrated flames being projected through the opening to points considerably above.

—J. W. D.

Electric arc welding. F. J. Heyes. E.P. 150,372, 2.4 and 6.8.19.

AN aluminium rod for electric arc welding of aluminium is provided with a flux consisting of a coating of cryolite or cryolite and salt held in position by aluminium wire, sheet, or gauze, or by means of a light cotton or like fabric. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 6138 of 1909; J., 1910, 161.)—J. W. D.

Ores; Process of treating —. L. B. Skinner. U.S.P. 1,350,286, 17.8.20. Appl., 10.2.19.

A MIXTURE of an oxidised complex zinc ore containing precious metals with an excess of carbonaceous reducing material is charged on to the hearth of a reverberatory furnace, where it is smelted at a temperature of at least 1200° C. The molten matte and supernatant slag produced are protected by a non-oxidising atmosphere, the fume resulting from the reduction and volatilisation of the zinc being led out of the furnace. The treatment is continued until the tapped slag contains not more than 6% Zn.—T. H. Bu.

Metallic oxides; Process and apparatus for reducing —. W. L. Morrison. U.S.P. 1,350,419, 24.8.20. Appl., 22.1.18.

THE oxides in a state of division fine enough to allow them to be carried in suspension by a gas are subjected to the action of hot reducing gases, the reduced metal is precipitated together with slag-forming material, and the metal is subsequently separated from the slag.—J. W. D.

Ores; Apparatus for agglomerating —. E. W. Davis. U.S.P. 1,350,440, 24.8.20. Appl., 19.11.18.

A SERIES of trucks to receive the material to be sintered are mounted on a track. A reciprocating feeding arrangement mounted beside the track engages with one of the trucks automatically upon its forward stroke and passes it by upon its return stroke. During the travel of the trucks an air-tight communication is formed between each truck and an adjacent vacuum chamber.—J. W. D.

Carbonising articles; Process for — and apparatus for same. M. D. Wilbur, F. P. Cannon, and T. J. Murphy. U.S.P. 1,350,483, 24.8.20. Appl., 26.7.17.

THE articles are packed in a non-fusible carbonising material which is quickly raised in temperature and maintained at a predetermined temperature for a certain length of time, an electric current being passed through the articles and the material between electrodes spaced apart from the articles by the carbonising material.—A. G.

Tungsten; Alloying —. J. H. Deppeler, Assr. to Metal and Thermit Corp. U.S.P. 1,350,709, 24.8.20. Appl., 28.10.19.

TUNGSTEN powder is compressed into dense, coherent, self-sustaining tablets, which are added to the alloying metal.—J. W. D.

Metals and alloys; Process of treating —. C. B. Foley. U.S.P. 1,350,714, 24.8.20. Appl., 29.6.17. Renewed 19.1.20.

AN induced electric current is passed through the molten metal and at each locality of heat addition by the current a hydrostatic head is maintained

which exceeds the vapour tension of the mass at the temperature generated. (*Cf.* E.P. 114,851 of 1916; J., 1918, 308 A.)—J. W. D.

Alloy. V. P. Taylor. U.S.P. 1,350,891, 21.8.20. Appl., 4.8.19.

THE alloy contains 80–100 pts. of aluminium, 8–17 pts. of copper, and tungsten.—T. H. Bu.

Zinc alloy. A. Tedesco, Assr. to Soc. Anon. Stabilimenti Biak. U.S.P. 1,350,892, 21.8.20. Appl., 11.7.19.

AN alloy of high strength contains 85–93 Zn, 3–12% Al, and 0.5–4% Sn.—T. H. Bu.

Aluminium alloys; Method for manufacturing —. A. Tedesco, Assr. to Soc. Anon. Stabilimenti Biak. U.S.P. 1,350,893, 21.8.20. Appl., 11.7.19.

ZINC-COPPER-ALUMINIUM alloys containing a high percentage of aluminium are made by separately preparing an alloy containing 66% Zn, 9% Cu, and 25% Al, and adding it to the aluminium bath in amount up to 30% of the final alloy.—T. H. Bu.

Alloy. F. Milliken, Assr. to F. Milliken, S. F. Weaver, and J. M. Repplier. U.S.P. 1,351,673, 31.8.20. Appl., 8.10.19.

AN alloy consists of copper, 41–52%, nickel, 22–28%, and lead, 22–30%.—W. J. W.

Alloys having high mechanical, chemical, and electrical resistance. A. Tedesco, Assr. to Soc. Anon. Stabilimenti Biak. U.S.P. 1,351,811, 7.9.20. Appl., 6.3.19.

AN alloy of the following composition: 35–44% Cu, 1.5–4.75% Fe, 3–6% W, and the remainder Ni. —J. W. D.

Alloy. Z. Jeffries and W. A. Gibson, Assrs. to The Aluminium Castings Co. U.S.P. (a) 1,352,271, and (b) 1,352,272, 7.9.20. Appl., 30.7.19.

AN aluminium alloy containing: 2–5% Cu, 4–25% Zn, and (a) 0.6–1.8% Fe, or (a) 0.5–1.5% Fe with an appreciable amount of magnesium.—A. R. P.

Metallic alloy and method of making same. T. D. Stay, Assr. to The Aluminium Castings Co. U.S.P. 1,352,322, 7.9.20. Appl., 6.3.17.

AN aluminium alloy containing 88–90% Al, 10–12% Cu, and 0.1% Ti, having a low shrinkage, and relatively great hardness, with a fine grain and good bearing qualities, is made by first preparing an alloy of titanium and copper and adding this to the aluminium.—J. W. D.

Aluminium; Process for electrically depositing —. G. L. Williams, Assr. to H. W. Campbell. U.S.P. 1,351,144, 31.8.20. Appl., 11.3.18.

ALUMINIUM is deposited by the electrolysis of aluminium sulphate dissolved in an anhydrous solvent which is normally liquid, and which, while heated, is of higher resistance than the aluminium sulphate to be electrolysed.—J. S. G. T.

Babbitt-metal product, and process of making same. L. Sapery. U.S.P. 1,351,404, 31.8.20. Appl., 21.5.20.

BABBITT-METAL, prior to melting, is coated with a thin film of another metal which has a considerably higher melting point and is without injurious action on the bearing qualities of Babbitt-metal. This film protects the Babbitt-metal from oxidation during melting.—J. W. D.

Magnesium powder; Process for the manufacture of —. D. S. Nicol, Assr. to Shawinigan Electro-Metals Co., Ltd. U.S.P. 1,351,865, 7.9.20. Appl., 23.7.17.

MAGNESIUM, melted in an atmosphere of a gas that

will not combine with it at high temperatures and pressures, is forced into a nitrogen atmosphere, where it is disintegrated into fine particles by means of a nitrogen jet directed into the stream of molten metal.—A. R. P.

Nickel and copper; Method of separating — from copper-nickel matte or materials. R. C. Stanley, Assr. to The International Nickel Co. U.S.P. 1,351,877, 7.9.20. Appl., 5.2.19.

THE copper is partly separated from the nickel compounds of the matte by furnace treatment; the residual nickel-rich material is converted into impure metallic nickel containing an appreciable amount of copper, and pure nickel is recovered from this by electrolytic treatment.—A. R. P.

Nickel-plating. E. N. Todd and W. R. King, Assrs. to The Hanson and Van Winkle Co. U.S.P. 1,352,328, 7.9.20. Appl., 28.12.14. Renewed 12.2.20.

A MIXTURE for electroplating comprises sulphate of nickel and ammonium, with an admixture of gum tragacanth.—J. W. D.

Zinc-bearing ores; Method of treating —. F. K. Cameron, J. A. Cullen and R. W. Hyde, Assrs. to American Smelting and Refining Co. U.S.P. 1,352,399, 7.9.20. Appl., 17.7.17.

THE zinc is converted into sulphate, and an alkali chloride is added in quantity sufficient to transform the zinc sulphate into chloride and to form a double halide salt, which is then crystallised from the solution.—J. W. D.

Steel; Chrome-nickel —. J. R. Speer. E.P. 150,020, 25.4.19.

SEE U.S.P. 1,314,022 of 1919; J., 1919, 778 A.

Tin ores; Removal of iron from oxide or roasted sulphide — by treatment with acids. M. Chiapponi, R. Hesse, and G. von Rauschenplat. E.P. 7186, 21.3.14. Conv., 28.3.13.

SEE U.S.P. 1,196,049 of 1916; J., 1916, 1022.

Corrosion of tubes and machinery parts of copper and copper-containing alloys; Prevention of selective —. F. von Wurstenberger and H. A. Frei. E.P. 127,823, 2.6.19, and 130,323, 30.6.19. Conv., 1.6 and 24.7.18.

SEE U.S.P. 1,335,209—10 of 1920; J., 1920, 415 A.

Nickel and copper; Manufacture of —. International Nickel Co., Assees. of R. C. Stanley. E.P. 138,600, 12.1.20. Conv., 5.2.19.

SEE U.S.P. 1,351,877 of 1920; preceding.

Ores; Apparatus for the concentration of —. F. J. Lyster, Assr. to Minerals Separation North American Corp. U.S.P. 1,352,072, 7.9.20. Appl., 18.11.16.

SEE E.P. 104,366 of 1916; J., 1917, 507.

Electroplating non-conducting substances with copper. U. Unno. U.S.P. 1,352,331, 7.9.20. Appl., 21.11.17.

SEE E.P. 106,184 of 1916; J., 1917, 721.

Cleaning guns and the like; Compound for — and method of preparing same [in tablet form]. Commercial Electrolytic Corp., Assees. of R. Laughlin. E.P. 133,019, 27.5.19. Conv., 21.9.18.

Case-hardening, tempering, and the like [; Mixture for preventing — locally]. S. Whyte. E.P. 150,035, 21.5.19.

Copper wire; Process for continuous production of — by electrolytical methods. S. O. Cowper-Coles. E.P. 150,063, 26.5.19.

Furnaces for heat treatment of metals. C. F. Gaunt, C. M. Walter, M. van Marle, and Gibbons Bros., Ltd. E.P. 150,099, 7.6.19.

Furnaces [for heat treatment of long articles]. A. Smallwood. E.P. 150,467, 24.6.19.

Annealing metal sheets. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 150,437, 4.6.19.

Ores; Water concentration of —. W. M. Martin. E.P. 150,113, 16.6.19.

Flexible [fireproof] material. E.P. 149,745. See V.

Depolarisation of cathodes. G.P. 322,192. See XI.

XI.—ELECTRO-CHEMISTRY.

Nickel resistors; Deterioration of —. F. A. J. Fitzgerald and G. C. Moyer. Trans. Amer. Electrochem. Soc., 1920, 31—34. [Advance copy.]

NICKEL wire employed as a substitute for nichrome as a resistor in electric furnaces was found to have a very short life, the wire becoming very brittle. The deterioration was not due to the action of carbon monoxide, but to that of sulphur. The effect of sulphur is very marked and rapid above 500° C. Nickel wire made brittle by sulphur may be softened by heating in an oxidising atmosphere. The action of sulphur upon nichrome is not so marked as in the case of nickel. If precautions are taken to prevent reaction with sulphur, nickel wire seems to behave satisfactorily as a resistor.

—J. S. G. T.

Hydrogen peroxide. Skirrow and Stein. See VII.

Electrical resistivity of refractories. Hartmann and others. See VIII.

PATENTS.

Electrical energy; Transforming the kinetic energy in gases into — and utilising the latter for carrying out gas reactions [e.g., production of nitric oxide]. C. Petersen. E.P. 122,173, 30.12.18.

A STRONG magnetic field is established within the reaction chamber by means of a powerful magnet, and the reacting gases are introduced tangentially. The gases traverse spiral paths, and axial and radial electrodes arranged within the reaction chamber become electrified owing to the ions produced by the motion of the gas in the magnetic field. The electrical energy produced may be utilised for the production of nitric oxide, or for effecting other reactions between the gases passing through the magnetic field.—J. S. G. T.

Electric [rotary] muffle furnace. J. R. C. August. E.P. 149,893, 18.10.19.

A MUFFLE is mounted coaxially within a chamber closed at one end and provided with a central aperture at the other. The annular space separating the muffle from the outer container is divided into an inner and an outer space by a coaxial fire-clay cylinder. An electric heating unit composed of powdered material or a heating coil is placed in the inner annular chamber, and the outer annular space is filled with insulating material. A movable container affording a close fit with the muffle is placed within the latter. The inserted end of the container is closed, and the other end projects a little beyond the outer casing of the furnace and is closed with a removable gas-tight cover. The muffle and outer casing rotate together.

—J. S. G. T.

Furnace; Electric —. J. Thomson. U.S.P. 1,351,977, 7.9.20. Appl., 19.11.19.

An electric furnace is provided with a zigzag

carbon resistor formed of two limbs, the ends of which are connected with carbon terminals which pass out from the heating chamber. The two limbs may be connected in series by means of a metallic conductor connected with carbon terminals outside the furnace chamber.—J. S. G. T.

Furnace; Electric —. F. on Schlegell and W. B. Lewis. U.S.P. 1,352,040, 7.9.20. Appl., 4.4.19.

An electrical contact piece projects through the wall of the furnace below the tap-hole into the melting chamber. The outer end of this contact piece is upturned and provided with a cooling chamber above the portion projecting into the furnace.

—J. S. G. T.

Cathodes; Depolarisation of — in electrolytic cells. P. Venturini. G.P. 322,192, 13.11.15.

JETS of the electrolyte are projected against the cathode surface by compressed air introduced into the bath. The electrolyte is thus maintained at uniform concentration, the cathode freed from hydrogen by oxidation, and the surface of the electrolytically deposited metal maintained clean and in contact with concentrated electrolyte.

—J. S. G. T.

Accumulator plates; Production of a protective coating for the active material used in—.

"Metallatom" G.m.b.H. G.P. 322,250, 31.1.18.

A PROTECTIVE, porous, metallic coating is squirted on to the surface of the plates. This is effected by squirting a mixture of the metal forming the skeleton framework of the plate together with indifferent substances such as kaolin, clay, infusorial earth, ashes, etc., or metals or alloys, which are subsequently rendered inactive or removed.

—J. S. G. T.

Filter electrode and process for making the same.

H. O. Trauns Forschungslaboratorium G.m.b.H. G.P. 322,600, 6.7.18.

THE electrode is constituted of a perforated or slotted cylinder of metal, carbon, or graphite connected with a channel for conveying the electrolyte. The cylinder is surrounded by gauze or a perforated cylinder. Coils of wire may be wound upon the latter, or the outer covering may consist of metal cuttings, "wool," or powder held between cylinders of gauze or perforated plates. The whole is held together with a single nut. In making the electrodes compounds of the heavy metals, insoluble in water, may be introduced into the outer covering and reduced to the metal electrolytically. The electrode affords intimate contact between its large surface and a gaseous, liquid, or emulsified solid electrolyte, with simultaneous thorough mixing with reagents, the electrolyte meanwhile streaming through the porous electrode under the influence of an excess or diminished pressure. The electrode can be used as cathode for purposes of reduction, hydrogenation, etc., or as anode for oxidation or halogenation. It may also be employed in accumulators or galvanic cells.—J. S. G. T.

Zinc battery elements [; Production of — by electro-deposition]. S. O. Cowper-Coles. E.P. 150,597, 8.1.20.

Electrolytic cell. E. A. Allen. U.S.P. 1,351,886, 7.9.20. Appl., 19.12.18.

Furnace for gas reactions. E.P. 116,503. See VII.

Hypochlorites. E.P. 150,214. See VII.

Manganese oxides. U.S.P. 1,352,208. See VII.

Electrical furnaces. E.P. 121,485, 134,810, and 150,532. See X.

XII.—FATS ; OILS ; WAXES.

Unaponifiable matter; Determination of — and characteristics of bottlenose oil. W. Fahrion. Chem. Umschau, 1920, 27, 131–134, 146–147.

THE author describes a modified method for determining unaponifiable matter in fats. Samples of technical marine animal oils yielded 8–22% of unaponifiable matter, which was identified as cetyl alcohol (m.p. 50° C.), and attributed to the presence of bottlenose oil. A commercial sample of the latter had: Saponif. value, 7.1; iodine value, 86.5; unaponif. matter 37.2%, with iodine value, 61.7. The unaponifiable matter determined by the method of Hönig and Spitz (two extractions of the soap with petroleum spirit) was 38.8%. The fatty acids (60%) remained fluid, and had mol. weight 285.5. They were thus mainly oleic acid; no doeglic acid was found.—C. A. M.

Chinese wood oil; Influence of the free fatty acid content in the valuation of — by the Browne polymerisation test. P. E. Jameson. Analyst, 1920, 45, 328–330.

THE time required to polymerise Chinese wood oil by heat increases with the fatty acid content, and in Browne's method (J., 1912, 731) the presence of 1.9–4.5% of free fatty acid extends the time by 1–2.5 mins. If the oil is mixed with dry calcium hydroxide and filtered, it will then, if genuine, polymerise within 12 mins. when heated at 282° C.—W. P. S.

Wool fat. VII. The "oily acid." I. Lifschütz. Z. physiol. Chem., 1920, 110, 29–40.

THE author's experiments indicate that the acid portion of wool fat contains no oleic acid or other unsaturated fatty acid. After repeated purification, the so-called "oily acid" gives no reaction for oleic acid with chromic and sulphuric acids, and the low iodine value previously ascribed to this "oily acid" probably depends on the presence of an acid formed by the oxidation either of cholesterol or, as the absorption spectrum indicates, of ischolesterol.—T. H. P.

Ultra-filtration. Schmitt. See I.

Colchicum seed oil. Grimme. See XX.

PATENTS.

Unsaturated fatty acids; Polymerisation of — at low temperature. De Nordiske Fabriker, De-Nofa, Aktieselskap. E.P. 127,814, 19.5.19. Conv., 4.6.18.

POLYMERISED fatty acids and their salts are obtained by converting unsaturated fatty acids, free from glycerin, into soluble salts, which are then heated under pressure with considerable quantities of alkaline agents at about 180°–211° C.—C. A. M.

Fats and oils; Process for splitting — in the manufacture of glycerin and fatty acids. W. J. Mellersh-Jackson. From Twitchell Process Co. E.P. 149,748, 15.5.19.

SEE U.S.P. 1,319,027 of 1919; J., 1919, 953 A.

Oleaginous substances; Process for extracting — from oleaginous-substance-containing materials. J. MacGregor and H. Austin. U.S.P. 1,351,483, 31.8.20. Appl., 24.6.18.

SEE E.P. 118,461 of 1917; J., 1918, 662 A.

Vulcanised oils. U.S.P. 1,315,246. See XIII.

XIII.—PAINTS ; PIGMENTS ; VARNISHES ; RESINS.

Pigments; Diffusing power of —. W. K. Lewis and F. P. Baker. J. Ind. Eng. Chem., 1920, 12, 890–891.

THE diffusion power of a pigment is defined as the weight in g. required to obscure an object behind a suspension of the pigment in a given liquid. A suspension of the pigment, of known concentration, is poured into a vertical cylinder with a plate glass base, and kept uniform by stirring or blowing, until the filament of an incandescence lamp held just below the bottom can no longer be seen. The following results in g. per sq. cm. were obtained with pigments ground in bleached linseed oil:—White lead, 260; carbon black, 2200; lampblack, 1830; red iron oxide, 318; red lead, 137; zinc oxide, 560; whiting, 295; barytes, 170; and lithopone, 560.

—C. A. M.

Turpentine oil; Determination of petroleum spirit in —. A. Allina and H. Salvaterra. Chem.-Zeit., 1920, 44, 673–674, 697–698.

TWENTY c.c. of the sample, 10 c.c. of water, and 20 g. of potassium bromate are placed in a distillation flask and hydrobromic acid (sp. gr. 1.59) is added in small quantities at a time, while the mixture is cooled, until a permanent yellow coloration is obtained. A further quantity of hydrobromic acid is then added, without cooling, so that the total amount introduced is 50 c.c. The mixture is maintained at 40°–50° C. for 1 hr. under a reflux condenser, and then steam-distilled. The distillation is stopped as soon as drops of oil heavier than water come over, the water is separated from the distilled oil, the latter shaken with fuming sulphuric acid, and, after separation of the two layers, the volume of the distilled petroleum spirit is noted. The treatment with sulphuric acid is necessary in order to remove small quantities of brominated terpenes etc. from the distilled petroleum spirit.

—W. P. S.

Ultra-filtration. Schmitt. See I.

PATENTS.

Coating compositions or paints. M. R. Isaacs. E.P. 150,551, 8.10.19.

A WEATHER-RESISTANT water paint is produced by mixing casein or casein glue with lime, together with a fluoride and plaster of Paris; additional materials such as clay, cement, oil, and a preservative such as naphthalene may also be introduced.

—D. F. T.

Gum [resin] from grass trees; Extraction of —. H. J. Pooley and J. L. Strevens. E.P. 150,638, 12.3.20.

THE outside portions of the grass tree (*Xanthorrhoea*) or the crude resins from this source, are extracted with a mixture of benzene and its homologues, boiling below 180° C.; the resin thus obtained contains only relatively small proportions of colouring matter and tannin and is capable of direct application as a raw material for the preparation of substitutes for shellac, and for other industrial purposes.—D. F. T.

Vulcanised oils; Process of treating — and products thereof. W. O. Snelling. U.S.P. 1,315,246, 9.9.19. Appl., 22.5.15.

A VULCANISED oil product, similar in its elastic and plastic properties to gum chicle, is obtained by depolymerising a vulcanised oil and re-polymerising the product to a plastic material without additional vulcanisation. For example, the soft,

plastic product obtained by allowing a mixture of a suitable oil (linseed, castor, soya), an aliphatic alcohol or hydroxy-fatty acid (hydroxystearic acid), and sulphur chloride to solidify, is heated at 100° C. for several hours.—W. H. C.

Varnish for balloons. Ballonhüllen-Ges.m.b.H. G.P. 321,264, 25.1.18.

An oil, e.g., castor oil, is treated with sulphur chloride, the product is dissolved in amyl formate, freed from hydrochloric acid by treatment with an alkali, and added to a balloon varnish, giving a waterproof, airtight, and elastic covering.—L. A. C.

Size. G.P. 321,382. *See* XV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Coagulation of Hevea latex; Bacteriological study of the —. Denier and Vernet. Caoutchouc et Gutta-Percha, 1920, 17, 10,491—10,493.

EXAMINATION of samples of Hevea latex collected with care for the avoidance of extraneous contamination revealed the presence of micro-organisms of 27 different types, only one of which was anaërobic. Cultures were grown in various media, and observation made of the behaviour of the organisms towards dambosc, lactose, sucrose, milk, albumin, and gelatin. Only one micro-organism was found capable of attacking dambosc (the inositol present in the latex), and this was the only organism invariably present in the samples.

—D. F. T.

Vulcanisation; Application of amino-derivatives of furfural to —. Vulcazone. A. Dubosc. Caoutchouc et Gutta-Percha, 1920, 17, 10,495—10,505.

FURFURAMIDE ("vulcazone"). $C_5H_7O_2N_2$, the condensation product of furfural and ammonia, analogous to hydrobenzamide, is recommended as a catalyst for the vulcanisation process. It is stated to be quite non-poisonous, and the tendency to dissociation into its constituents can be avoided, if necessary, by previously transforming the furfuramide, by the action of heat, into its isomeride furfurine, which is also an effective vulcanisation catalyst.—D. F. T.

Vulcanisation; Theory of the acceleration of —. A. Dubosc. Caoutchouc et Gutta-Percha, 1920, 17, 10,511—10,514.

In the presence of a nitrogenous accelerator during vulcanisation, thiocyanic acid of the formula $H:C:S:N$ is formed, which then undergoes fission into hydrocyanic acid and hexavalent sulphur; the hydrocyanic acid again combines with the free divalent sulphur with formation of thiocyanic acid, and hence of more hexavalent sulphur, this cycle of changes recurring continuously. The hexavalent sulphur, possessing three times as many free valencies as ordinary divalent sulphur, is trebly active as a vulcanising agent.—D. F. T.

PATENTS.

Rubber; Treatment of raw —. Dunlop Rubber Co., J. V. Worthington, and A. W. T. Hyde. E.P. 150,043, 22.5.19.

In order to produce rubber of a uniform degree of softness, thereby avoiding the need for wide variations in the time of "working" on the rolls, the rubber is heated for a suitable time in an inert atmosphere, e.g., of "live" steam. Measurement of the viscosity of the rubber supplies a satisfactory method for the control of the treatment.—D. F. T.

Coated fabric and process of producing the same. J. A. Wilson, Assr. to Duratex Co. U.S.P. 1,352,163, 7.9.20. Appl., 19.12.19.

A WATERPROOF fabric is produced by coating a textile material first with a spreading of rubber, and then with a layer of a vulcanising varnish consisting of a vegetable drying oil mixed with sulphur. The coated fabric is then embossed and vulcanised.—D. F. T.

Waterproofed material; Manufacture of — [using reclaimed rubber]. M. Fränkel und Range, and W. Golombek. E.P. 4451, 20.2.14. Conv., 14.8.13.

See G.P. 278,717 of 1913; J., 1915, 437.

XV.—LEATHER; BONE; HORN; GLUE.

Glutin; Benzene derivatives in — and protein-content of gelatin. E. Salkowski. Z. physiol. Chem., 1920, 109, 32—48.

THE products formed by putrefaction of best commercial gelatin include traces of an indole derivative, about 0.11% of aromatic hydroxy-acids, succinic acid, and at least 1.3% of hydrocinamic acid. The presence of a protein in the gelatin has also been demonstrated. (Cf., J.C.S., i., 684.)—T. H. P.

PATENT.

Size; Manufacture of — from horny material. F. Schmidt. G.P. 321,382, 4.12.17.

A PRODUCT, suitable, e.g., for adding to lime-wash colours, is prepared by heating horny material for a long time in an autoclave above 200° C., with or without the addition of acid or alkaline salts.

—L. A. C.

XVI.—SOILS; FERTILISERS.

Oxidising power of soil from limed and unlimed plots and its relation to other factors. J. R. Neller. Soil Sci., 1920, 10, 29—37.

SAMPLES from plots of loam soil, limed and unlimed, were incubated at 20° C., and examined for bacterial activity by means of carbon dioxide production, ammonia and nitrate accumulation, nitrogen fixation, and bacterial numbers. To determine the amount of carbon dioxide produced, 0.75 g. of soya-bean hay was added to each sample of soil; for the ammonia and nitrate tests 110 mg. of nitrogen as dried blood; and for the fixation of nitrogen, 1% mannitol solution. The moisture content was made up to 50% saturation in each case. The oxidising power of the soil from the limed plots, as judged by the carbon dioxide production, was 40% greater than from the unlimed plots. Ammonia accumulation showed no differences. Nitrate production, nitrogen fixation, and bacterial numbers were all higher in the limed samples. The history of the plots showed that the previous crop production of the plots was closely related to the present oxidising power of the soils in all cases, and less closely to the nitrate accumulation and bacterial numbers, but had no relation to the ammonia accumulation.

—J. H. J.

Acid soil; Effect of dicalcium silicate on an —. B. L. Hartwell and F. R. Pember. Soil Sci., 1920, 10, 57—60.

POT experiments were conducted to trace the effect of commercial dicalcium silicate and hydrated silica on a loam soil growing a crop of lettuce. The two substances were added singly and in combination with limestone and acid phosphate (superphosphate). It was found that the silica compounds could replace limestone in counteracting acid con-

ditions in the soil, but there was no evidence of any specific effect of the silica.—J. H. J.

Calcium in soils; Proposed method for the estimation of total — and the significance of this element in soil fertility. O. M. Shedd. *Soil Sci.*, 1920, 10, 1—14.

THE proposed method is more rapid and accurate than that already in use for the determination of calcium in soils, and one in which the production of a precipitate with ammonia, which occludes calcium, is avoided. After fusing a 1-g. sample with fusion mixture, the silica is separated in the usual manner, the filtrate is made slightly alkaline with ammonia and then just acid with hydrochloric acid, raised to the boil, and precipitated with 1—2 g. of powdered ammonium oxalate. The mixture is allowed to stand on the top of the steam bath for a few hours, and then at room temperature overnight. The precipitate is filtered off and ignited, dissolved in hot dilute hydrochloric acid, and bromine water and ammonia added to precipitate manganese. The solution is acidified with acetic acid, and the manganese is filtered off. The filtrate is then reprecipitated as before. Analyses of a large number of soils, both virgin and cultivated, by this method showed that cultivation led to a considerable loss of calcium. The most fertile soils contained the most calcium. The application of limestone and rock-phosphate to soils poor in calcium is beneficial on account of the calcium they supply as plant food, apart from any other benefit.

—J. H. J.

Calcium and nitrogen content of plants; Relation between the — and the function of calcium. F. W. Parker and E. Truog. *Soil Sci.*, 1920, 10, 49—56.

FROM the consideration of a large number of analyses of various plants with respect to their nitrogen, phosphorus, potassium, calcium, and magnesium content it was observed that calcium was the only element of which the content was closely related to the nitrogen content. The plants examined could be grouped in two classes, according to their calcium:nitrogen ratio. In one class the ratio was low, averaging 0.306, and this class consisted of the grass family, which have a low lime requirement and are tolerant to soil acidity. In the other class the ratio was high, 0.553, and this class contained the legumes and other plants which have a high lime requirement and are sensitive to soil acidity. From these results it may be concluded that plants which have a high protein or nitrogen content, and consequently produce a large amount of acidity from protein metabolism, require a large amount of calcium as carbonate for the neutralisation of plant acids.

—J. H. J.

Barley varieties and fertilisation. J. Ahr and C. Mayer. *Brochure*, Freising, 1919. Biedermann's Zentr., 1920, 49, 334—310.

A RECORD of pot experiments with six varieties of barley, fertilised with lime, potash, phosphoric acid, and nitrogen, singly and in various combinations. Copious supplies of lime, potash, and phosphoric acid were found beneficial in respect of brewing quality and yield of grain, whilst excess of nitrogenous fertiliser proved harmful in both respects, especially when other fertiliser constituents were deficient. Some of the varieties were more injured than others by excess of nitrogen.

—J. H. L.

Ammoniacal nitrogen; Preventing volatilisation of — by means of calcium chloride. A. Stutzer. *Fühling's Landw. Zeit.*, 1919, 68, 59—63. Biedermann's Zentr., 1920, 49, 321—324.

THE volatilisation of ammonia from solutions of

ammonium carbonate, and therefore from stable manure, can be in large measure prevented by addition of calcium chloride, owing to the formation of ammonium chloride and calcium carbonate. About 75% of the ammonia can be fixed in this way, by adding 6 pts. of calcium chloride per 1 pt. of ammoniacal nitrogen present.—J. H. L.

Insecticide tests; Miscellaneous soil —. J. J. Davis. *Soil Sci.*, 1920, 10, 61—72.

THE use of kerosene and coal tar preparations as applications to soils infested with the grubs of beetles, chiefly *Popillia japonica*, gave only moderately successful insecticidal results. Mercuric chloride, sulphuric acid, and acetaldehyde were quite ineffective. A large number of experiments were made with sodium cyanide, which proved to be more efficacious than the others. The granular cyanide was used dissolved in the proportion of 165 lb. per 12,000 galls., which was sufficient for an acre of land, and was applied as a spray from a perforated pipe attached to a tank of the liquid. The treatment had no permanent injurious effect on grass, but corn (maize) crops were appreciably injured. The cyanide disappeared from the soil in 7—10 days, and the addition of ammonium sulphate accelerated its disappearance.—J. H. J.

Seed treatment; Presoak method of —: A means of preventing seed injury due to chemical disinfectants and of increasing germicidal efficiency. H. Braun. *J. Agric. Res.*, 1920, 19, 363—392.

THE use of formalin and copper sulphate for the disinfection of seeds usually causes injury to germination. It was found that if the seeds were saturated with water before applying the disinfectant, then the latter in penetrating the seed was diluted beyond the danger point. It is best to soak the seeds in water for not more than 10 mins., and then to remove them and allow them to remain covered for 6 hrs. The seeds are then soaked in formalin of strength 1:400 or in copper sulphate of strength 1:80 for 10 mins., drained, covered for 6 hrs., and then air-dried. Stimulation of germination was observed in treated seeds. The seeds experimented with were the cereals, but other seeds are amenable to the treatment if the process be modified according to the rate of absorption of water by the seed, its susceptibility to the disinfectant, and the time necessary for the beginning of germination and of the vegetative activity of the infecting organisms.—J. H. J.

Carbon dioxide. Robinson. See VII.

PATENTS.

Sewage sludge; Utilisation of — for the production of manure and other products. G. Young and G. Watson. E.P. 150,375, 29.4.19.

SLUDGE, dried to 50—60% moisture, is placed in a closed furnace of the rotary kiln type and subjected to slow combustion by means of a gentle air draught which is so regulated, in accordance with the moisture in the sludge, as to maintain a low temperature. Such combustion of the non-volatile greases and other organic matter as takes place is complete, without soot formation, but a considerable proportion of organic matter is retained in the ash in a sterilised condition, and the process is conducted so as to produce a porous, easily powdered mass instead of clinker. The volatile oils distilled off during the combustion are led into an ammonia-recovery apparatus, and the ammonium sulphate obtained may be sold separately, or added to the furnace residue with or without other fertilising agents.—W. J. W.

Superphosphate; Process of producing —. C. C. Meigs. U.S.P. 1,351,672, 31.8.20. Appl., 28.10.19.

PHOSPHATE rock is treated with one-half of the

amount of acid used in producing ordinary superphosphate. The product is treated with excess of a solution of an alkali sulphate, and the mass is filtered, and free sulphate removed. The filtrate is crystallised, after which the crystals are heated to at least 100° F. (38° C.) and treated with dilute hydrochloric acid, the solution being then applied to the treatment of phosphate rock, and the resulting product dried.—W. J. W.

Salt mixtures suitable for fertilisers; Process of obtaining — from explosives etc. containing perchlorates. Dynamit-A.-G. G.P. 321,878, 20.9.18.

SALT mixtures free from perchlorates are obtained from explosives or mixtures of salts by extracting the material with solutions of potassium salts, and evaporating the extracts to dryness.—C. A. M.

XVII.—SUGARS ; STARCHES ; GUMS.

Sucrose and some other carbohydrates; Ethylene oxide structure of —. E. F. Armstrong and T. P. Hilditch. Chem. Soc. Trans., 1920, 117, 1086—1090.

FURTHER evidence of the presence of the ethylene oxide form of levulose in sucrose (cf. J., 1920, 127 A) is furnished by the rapid increase in the reducing power to permanganate when sucrose is hydrolysed by means of invertase. When the hydrolysis is carried out by means of hydrochloric acid isomeric change is promoted by the acid, and the figures give little indication of the presence of any more of the ethylene oxide form than is normally produced by acid in a mixture of dextrose and levulose, the ethylene oxide form first produced presumably undergoing rapid rearrangement into the butylene oxide form. Galactose, arabinose, and xylose showed little difference in their behaviour towards permanganate in alkaline solution, but in acid solution the difference was more marked, the pentoses being especially active as reducing agents.—G. F. M.

Dextrose; Optical rotation of — under the influence of hydrochloric or sulphuric acid. I. H. Murschhauser. Biochem. Zeits., 1920, 104, 214—236.

THE influence of acid on the mutarotation of dextrose consists in an acceleration of the reaction, this effect increasing with the concentration of the acid. (Cf. J.C.S., i., 661.)—T. H. P.

Honey; Antiscorbutic value of —. H. K. Faber. J. Biol. Chem., 1920, 43, 113—116.

HONEY appears to have no value as an antiscorbutic, since guinea pigs developed severe scurvy when fed on a diet of oats, water, and honey.—J. C. D.

Starch; Compound of — with phosphoric acid. J. Kerb. Biochem. Zeits., 1919, 100, 3—15.

STARCH was esterified with phosphoric acid by treating a soluble preparation with a solution of phosphorus oxychloride in chloroform in the presence of calcium carbonate. The calcium salt of the amylophosphoric acid was precipitated with alcohol from the concentrated solution and purified. By means of diastatic cleavage hexose monophosphoric acid was obtained from the compound in the form of a calcium salt. The latter substance was fermented by yeast.—S. S. Z.

Ultra-filtration. Schmitt. See 1.

PATENTS.

Starch and conversion products thereof; Method of manufacturing —, and improved products obtained therefrom. A. W. H. Lenders. E.P. 149,374, 5.3.19.

STARCH prepared by known methods is freed from residual proteins by the action of bacteria, such as *B. putrificus*, which render the proteins soluble without affecting the starch appreciably; the dissolved proteins are then removed by washing. Starch thus purified can be converted, without loss, into dry, finely divided dextrose, free from colour or bitter flavour, by hydrolysing with acid, concentrating to a syrup, heating the latter to the consistency of whipped cream, and allowing it to crystallise.—J. H. L.

Sugar boiling; Process of — [for production of toffee]. Ballochmyle Creamery Co., Ltd., and R. McCrone. E.P. 150,588, 18.12.19.

Saccharic and tartaric acids. E.P. 108,494. See XX.

XVIII.—FERMENTATION INDUSTRIES.

Amylase; Process of purifying pancreatic —. H. C. Sherman, I. D. Garard, and V. K. La Mer. J. Amer. Chem. Soc., 1920, 42, 1900—1907.

A FURTHER study of the process of purification of pancreatic amylase previously described (J., 1911, 973; 1920, 37 A), determinations of the total solids and enzyme activity being made at each stage. Alcohol up to 5% or an alcohol-ether mixture up to 8% of the volume of the substrate did not materially affect the activity of the amylase. In the process of purification used about 50% of the amylolytic activity was lost by the end of the dialysis, but in another series of experiments, using liquid air cooling in the later stages, it was possible considerably to increase the proportion of active amylase recovered in the final product. The precipitate ("sac precipitate") which formed in the inner solution during dialysis showed very high proteolytic and little or no amylolytic activity. (Cf. J.C.S., Nov.)—W. G.

Alcoholic fermentation; Relation of aldehydes to —. C. Neuberg and M. Ehrlich. Biochem. Zeits., 1920, 101, 236—276.

THE influence of 71 aldehydes on alcoholic fermentation was tried. It was found that the stimulating effect of the aldehyde group was general and independent of the radical associated with it. This activation takes place whether the fermentation is produced by living yeast or by the cell-free juice.—S. S. Z.

Alcoholic fermentation; Relation of phytochemically reducible substances to the process of — and the nature of the action of activators. C. Neuberg and M. Ehrlich. Biochem. Zeits., 1920, 101, 276—319.

NUMEROUS experiments are described which show that most ketones and their derivatives and phytochemically reducible substances are capable of activating alcoholic fermentation. The activating influence is not so marked—in fact, almost absent—with the lower ketones.—S. S. Z.

Enzymes; Chemical nature of —. T. Bokorny. Biochem. Zeits., 1919, 100, 100—114.

THE alkylamino-nitrogen was estimated in a number of well-known enzymes by means of nitrous acid. The amino-nitrogen was found to vary from 3.05% in rennet to 7.66% in papayotin. The author considers that the results obtained are in favour of the theory of the protein nature of enzymes.—S. S. Z.

Yeast cell; Influence of surface-active nonylic acid and of some surface active homologues of the alcohol series (amyl alcohol and octyl alcohol) on the — and on fermentation. W. Windisch, W. Henneberg, and W. Dietrich. *Biochem. Zeits.*, 1920, 107, 172—191.

A CONCENTRATION of 0.005–0.02 of nonylic acid has an inhibitive action on yeast fermentation; 0.017–0.04 of octyl alcohol has a similar effect. Higher concentrations of amyl alcohol are required to inhibit fermentation. Nonylic acid and octyl alcohol also produce a diseased condition of the yeast cell accompanied by alteration of its appearance and production of fat in the cell. The experiments point to the fact that the influence of these substances is not due to their chemical properties but to their surface action.—S. S. Z.

Isoamyl alcohol; Preparation of — by Pasteur's method. F. Martin. *J. Pharm. Chim.*, 1920, 22, 220—226.

Is the preparation of the amylsulphuric acids, which is the first step in the isolation of pure isoamyl alcohol from fusel oil by the fractional crystallisation of the barium amylsulphates, the best results are obtained with 90–91% sulphuric acid (sp. gr. 1.820–1.825), using an excess of 15–20% above the theoretical quantity. Under these conditions about 60% of the alcohol is esterified, whilst if a very large excess of acid is used, considerable difficulties are encountered later in the separation of the barium sulphate without any adequate compensation in the increased percentage of alcohol esterified. A small proportion of the alcohol is apparently converted into the neutral sulphate, the actual yield of the acid sulphates amounting to 50–55%.—G. F. M.

Distillation and rectification. L. Gay. *Chim. et Ind.*, 1920, 4, 178—188.

THE author gives equations for calculating the composition of the liquid and vapour phases in the distillation and rectification of a mixture of two constituents, and for determining the minimum amount of heat necessary. In the case of aqueous alcoholic solutions, if the strength exceeds 8.2% by weight of alcohol, the heat required varies little with the concentration of the solution, and ranges from 900,000 to 1,000,000 cal. per 1000 kg. of alcohol (95%) recovered. With these solutions the height of the rectifying column may be kept unaltered with a theoretical minimum of 25 sections. On the other hand, the height of the distillation column should be decreased as the strength of the solution increases up to 40%; above this concentration it should be increased slightly as the percentage of alcohol increases. Thus, compared with the rectifying column, the height of the distillation column is equal at 8.2% ; half at 10% ; three-tenths at 40% ; and three-sevenths at 83%. With concentrations below 8.2% , the requisite height of distillation column governs the minimum heat supply. As the alcohol content decreases from 8.2%, the rectification column should be correspondingly reduced. At 3.7% its length will be $\frac{1}{4}$ of that of the distillation column. In practice theoretical data are modified by plant imperfections, and correction tables should be drawn up, giving for each metre of the column the equivalent theoretical number of plates. Proposed schemes for confining the heat supply to the plates of the distillation column and the cooling to the rectifying column or, in another case, for causing the emergent liquid to recirculate through the upper sections, are considered to be unsound. A greater efficiency of the plates, however, should be aimed at.—W. J. W.

Barley. Ahr and Mayer. See XVI.

Starch and phosphoric acid. Korb. See XVII.

Cereals. Lucas. See XIX A.

PATENTS.

Acetone and butyl alcohols; Fermentation processes for the production of —. C. Weizmann. E.P. 149,355, 21.2.16.

UNHOPPED brewers' or distillers' wort, of low gravity, is sterilised by heating under pressure and fermented by the bacteria previously described (E.P. 4845/15; J., 1919, 301 A). The concentration of carbohydrates in the wort may vary from 4 to 9% , but in these cases the acidity must not exceed 0.4–0.6%. The ordinary brewery or distillery plant may be employed, with such modifications as are necessary to exclude infection during the cooling and fermentation of the wort.—J. H. L.

Yeast; Manufacture of — from fermented musts, and more particularly the fermented musts of molasses and sugar beet pulp. L.J.P.M.J. Dupire. E.P. 149,438, 13.5.19.

WHERE the production of yeast is the chief aim of fermentation the worts are diluted to such an extent (e.g., to sp. gr. 1.030 in the case of molasses or sugar beet worts) as to limit the amount of sugar present to that required for yeast growth. The yeast is subsequently separated by centrifuging (cf. E.P. 123,711; J., 1919, 334 A). Molasses worts of sp. gr. 1.030 give practically as much yeast as those of sp. gr. 1.090.—J. H. L.

Yeast; Process of treating waste brewers' — to render it suitable for baking purposes. H. W. Andersehou. E.P. 149,533, 5.9.19.

BREWERS' yeast is treated first with a solution of sodium bicarbonate and a small quantity of sodium salicylate, and afterwards with a solution of borax. It is next preferably treated with a cleansing agent (sodium chloride and potassium carbonate), then aerated in presence of nutrient salts, cane sugar, pepsin, and infusion of willow, and finally mixed with cream of tartar, potato-flour, and dried milk powder, and pressed.—J. H. L.

Alcohol; Absolute — and process of making the same. G. B. Frankforter. U.S.P. 1,350,254, 17.8.20. Appl., 20.12.18.

ORDINARY commercial alcohol is partially dehydrated by treatment with a mixture of potassium carbonate and potassium fluoride, then decanted from the mixture and heated with quicklime in an autoclave under a pressure of 55 lb. per sq. in., and finally the last traces of water are removed by distilling and passing the vapours over calcium carbide.—J. H. L.

Liquids; Process of de-alcoholising —. Apparatus for treating liquids. W. Koedding and W. J. Lemp. U.S.P. (A) 1,351,521 and (a) 1,351,522, 31.8.20. Appl., 21.2 and 14.4.17.

(A) BEER is heated in a pipe system and then passed in a continuous stream through a vacuum chamber at a temperature sufficient to evaporate the alcohol without impairing the other constituents. (a) Liquids are evaporated in a tank heated by means of a water-jacket and having upon the wall opposite to the jacket a helical V-shaped trough. Means for delivering liquid into the upper and removing it from the lower part of the tank are provided, and the vessel is connected with a suction system within which is a surface condenser.

—C. A. M.

XIXA.—FOODS.

Cereals; Ripening of —. H. Lüers. *Biochem. Zeits.*, 1920, 104, 30—81.

THE author has devised a new method of estimating acidity in organic extracts and an improvement in Sørensen's formol-titration method (*cf.* J., 1920, 669 A), and has applied both of these to follow the alterations in acidity, formol-titratable nitrogen, and enzymic relations of barley, wheat, oats, and rye during the final stages of ripening and subsequent storage for two months. (*Cf.* J.C.S., Nov.)
—T. H. P.

Colloids; Changes in the physical state of —. XXIII. *Acid-albumin*. M. Adolf and E. Spiegel. *Biochem. Zeits.*, 1920, 104, 175—189.

CERTAIN of the changes accompanying the conversion of serum-albumin into acid-albumin by the action of boiling hydrochloric acid have been investigated. (*Cf.* J.C.S., i., 683.)—T. H. P.

Cooloids; Changes in the physical state of —. XXIV. *Precipitation of protein by acids and alkalis*. R. Wagner. *Biochem. Zeits.*, 1920, 104, 190—199.

THE results of various physico-chemical measurements indicate that the state of a protein salt as regards hydration and ionisation conditions the precipitability of this salt in presence of excess of acid, and that there is no reason to suppose that this precipitability is not primarily to be considered as displacement of a salt from solution by excess of its acid. (*Cf.* J.C.S., i., 683.)—T. H. P.

Gelatin. Salkowski. *See* XV.

PATENTS.

Milk and the like; Desiccating —. R. W. G. Stutzke, Assr. to G. A. Buhl Co. U.S.P. 1,350,248, 17.8.20. Appl., 9.7.17.

MILK is injected into a circulating current of superheated steam, the dried solids are collected within the circulatory system, and withdrawn therefrom together with superheated steam, and after the latter has been mixed with air the solids are separated.—J. H. L.

Food compound and process of preparing the same. A. S. Wahl, Assr. to Arnold Wahl Institute. U.S.P. 1,350,756, 24.8.20. Appl., 26.2.19.

A MASH of starchy material and malt, saccharified at 60° C., is mixed with a mash containing wheat bran, lactic acid, and malt, which has been subjected to proteolytic conversion at 45° C.; the combined mash is held at a temperature of at least 60° C., and the liquor is drawn off to yield an aqueous product containing at least 70% of total solids, 1.3% of total acidity, and 0.8% of free lactic acid, with maltose constituting at least 80% of the total solids.—J. H. L.

Yeast. E. P. 149,533. *See* XVIII.

XIXB.—WATER PURIFICATION; SANITATION.

Water; Rate of solution of atmospheric nitrogen and oxygen in —. W. E. Adeney and H. G. Becker. *Sci. Proc. Roy. Dublin Soc.*, 1920, 16, 143.

A CONTINUATION of experiments previously reported (*see* J., 1919, 156 A; 1920, 311 A; also J.C.S., Nov.)
—J. R. P.

Antiseptic action of some chlorine derivatives of methane, ethane, and ethylene. E. Salkowski. *Biochem. Zeits.*, 1920, 107, 191—202.

THE suitability as preservatives of methyl chloride,

chloroform, methylene chloride, acetylene dichloride, trichloroethylene, and ethyl chloride was tested. Trichloroethylene on account of low cost, of the small quantities required to be employed, and of its volatile character was found to be the most suitable.—S. S. Z. ●

Silver; Oligodynamies of —. II. R. Doerr. *Biochem. Zeits.*, 1920, 107, 207—219.

THE bactericidal action of metallic silver disappears when the metal is ignited, boiled several times with distilled water, or is embedded in an agar jelly. The inactivated metal can be re-activated by being treated with very dilute solutions of a strongly dissociated acid. The oligodynamic principle of silver is dialysable and shows the same properties of diffusion in agar jellies as silver oxide and silver nitrate. Bacteria seeded on an agar plate containing metallic silver, silver oxide, and silver nitrate grow often in two concentric rings similar to the Liesegang phenomenon. Pieces of silver embedded in a blood-agar plate produce hæmolytic zones.—S. S. Z.

Ultra-filtration. Schmitt. *See* I.

Insecticide tests. Davis. *See* XVI.

Seed treatment. Braun. *See* XVI.

Selenium and tellurium. Joachimoglu. *See* XX.

PATENTS.

Sewage sludge or other wet or moisture-laden material; Method of and means for drying —. L. Linden. E.P. 150,368, 25.2.19.

IN the upper part of an enclosed structure are arranged a number of horizontal or inclined troughs provided with mechanical stirring devices; below each pair of troughs is a drying floor divided into separately-hinged sections. A second series of troughs and floors may be placed beneath the first. Below these may be arranged hoppers from which the material passes to grinding rollers and then on to a bottom floor, which may be heated, and from which the material may pass to a conveyor. At the base of the structure are lateral air inlets, supplemented if required by others near the top, and in the roof are air outlets, near which fans may be fixed to facilitate removal of moisture-laden air. The partly-dried sludge is introduced by means of distributors into the top troughs in which it is aerated and dried by a current of air, heated by radiators at the base. Gangways for workers, placed between the troughs, serve at the same time as baffles for the air current. When the material in the troughs has assumed a granular condition, a further quantity of partly-dried sludge is mixed with it, together with disinfectant if desired, and the excess material is then expelled by the stirrers on to the floor below, whence, after a suitable period, it is tilted on to the next series of troughs and floors, from which it finally passes to the hoppers and delivery floor.—W. J. W.

Sewage sludge. E.P. 150,375. *See* XVI.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Codeine; Determination of —. H. E. Annett and H. Sen. *Analyst*, 1920, 45, 321—328.

TEN g. of dry opium, or a suitable quantity of other substance (*e.g.*, alkaloids, plant material, etc.), is mixed with 4 g. of calcium hydroxide with the gradual addition of 100 c.c. of water; the mixture is stirred for 30 mins., filtered, 50 c.c. of the filtrate is treated with 40 c.c. of 2% acetic acid and 10 c.c. of basic lead acetate solution (*sp. gr.* 1.25), filtered, 75 c.c. of the filtrate is treated with 2.5 g. of calcium

hydroxide, the mixture shaken for 30 mins., filtered, and 50 c.c. of the filtrate extracted with three successive quantities (50 c.c. each) of toluene. The united extracts are filtered and dry gaseous hydrogen chloride is introduced in slight excess; this excess is removed by a current of air, the precipitated codeine hydrochloride is collected on a filter, dissolved in a small quantity of water, the solution evaporated, and the residue dried and weighed. If the treatment with basic lead acetate is omitted, the codeine hydrochloride obtained is highly coloured, but the error introduced is not more than 5–10%.—W. P. S.

Colchicum seeds; Alkaloidal content and fatty oil of —. C. Grimme. Pharm. Zentralh., 1920, 61, 521–524.

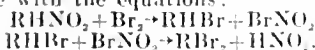
SPEAKING generally, the colchicine content (0.11–0.52%) in colchicum seeds is smaller the higher the relative weight of 1000 of the seeds. A sample of powdered seeds, extracted with ether, yielded 17.6% of a light brown odourless oil with the following characters: Sp. gr. (15° C.), 0.9176; solidif. p., –9° C.; n_D^{20} = 1.4642; acid value, 20.32; saponif. value, 184.3; iodine value, 128.5; unsaponif. matter, 0.71%. *Fatty acids*: m.p., 24.0° C.; solidif. p., 22.5° C.; neutralisation value, 187.6; iodine value, 131.0; and mean molec. equiv., 300.3.—C. A. M.

Digitalis; Some of the important constituents of —. W. J. McGill. J. Amer. Chem. Soc., 1920, 42, 1893–1900.

With careful manipulation and uniform methods of procedure, using various solvents, it is possible to obtain crude fractions from digitalis which will have a reasonably constant chemical composition, but in view of the difficulty of separating individual glucosides in a chemically pure state from these crude fractions, it does not appear that such an undertaking has at present any commercial value. The cold water extract of digitalis leaves contains, at the most, only a very minute quantity of digitoxin, its activity being entirely due to a chloroform-soluble and chloroform-insoluble fraction. Digitasaponin from the leaves is entirely inactive and non-haemolytic when purified. The chloroform-soluble fraction from the hot water infusion of the leaves is a mixture of gitalin and digitoxin.—W. G.

Halogenation; Experiments on —. Direct displacement of negative groups by halogen in the aromatic series. I. Displacement of the nitro-group by bromine. S. N. Dhar. Chem. Soc. Trans., 1920, 117, 993–1001.

THE interaction between bromine and aromatic nitro-compounds at 200°–250° C., under pressure, results as a rule in the displacement of the nitro-groups, and, sufficient bromine being present, in further halogenation, to an extent, in some cases, beyond what is possible by direct halogenation under any other known conditions, the best example of this being the formation of octabromoxanthone from tetrabromodinitroxanthone. It is assumed that in the reaction a more powerful halogenating agent than the bromine itself is formed, possibly in accordance with the equations:



In the case of nitrophenols an excess of bromine results in the displacement of the hydroxyl group as well, picric acid giving, for example, a mixture of tetra- and pentabromophenol and hexabromobenzene. 2,4,6-Trinitrotoluene gives pentabromobenzyl bromide, whilst various tetrabromonaphthalenes and hexabromonaphthalenes were obtained from 1,5- and 1,8-dinitronaphthalene. The xanthone and coumarin series supplied examples of particularly stable nitro-groups which could not be displaced by bromine under the experimental conditions studied.—G. F. M.

Gallie acid; Derivatives of —. I. Synthesis of 4-hydroxy-3,5-dimethoxyphthalic acid. R. L. Alimchandani and A. N. Meldrum. Chem. Soc. Trans., 1920, 117, 961–970.

GALLIC acid trimethyl ether was condensed with chloral in presence of sulphuric acid with the formation of a trichloromethylphthalide, indicating that the 4-methoxyl group of the ether was first hydrolysed by the acid, with formation of syringic acid. Somewhat better results were obtained by using syringic acid (4-hydroxy-3,5-dimethoxybenzoic acid) for the condensation, the latter being conveniently obtained from the above gallic acid trimethyl ether by preliminary treatment with 96–98% sulphuric acid. On hydrolysis the trichloromethylphthalide gave the corresponding carboxylic acid which on heating lost carbon dioxide with the formation of 4-hydroxy-3,5-dimethoxyphthalide. This on methylation and oxidation gave 3,4,5-trimethoxyphthalic acid. This acid readily gave an anhydride which lost a methoxyl group on treatment with sulphuric acid in the cold with the formation of 4-hydroxy-3,5-dimethoxyphthalic acid. Both these phthalic acids on boiling with hydrochloric acid are reconverted into the corresponding gallic acid derivative with loss of carbon dioxide.—G. F. M.

Dichloroethyl sulphide [“mustard gas”]; Properties of —. F. Martin. J. Pharm. Chim., 1920, 22, 161–165.

LIQUID dichloroethyl sulphide is heavier than water; its alcoholic solution when mixed with a large volume of water yields a white turbidity which disappears after some time. When it is boiled with alcoholic potassium hydroxide solution the vapours produced give a white precipitate on contact with Deniges' reagent (acid mercuric sulphate solution). If the vapours are passed into iodine solution an odour resembling that of mercaptan is noticed. The substance is hydrolysed fairly rapidly by water; its aqueous solution gives a white precipitate (di-iododiethyl sulphide) with Grignard and Rivat's reagent (sodium iodide, 8, copper sulphate, 0.8 g., gum arabic solution, 1, and water, 33 c.c.).—W. P. S.

Organic acids; Dissociation constants of — as means for their identification. I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 514–518.

THE hydrogen ion concentration of a solution of a weak organic acid, and hence the dissociation constant, may be conveniently determined colorimetrically by observing the colour change of a suitable indicator in the solution and in a solution of known hydrogen ion concentration. A selection of indicators suitable for various weak acids is given. Standard solutions of known p_H values for comparison may be prepared from dilute hydrochloric acid or from mixtures of potassium hydrogen phthalate with hydrochloric acid or sodium hydroxide.—W. S. M.

p-Cymene as a solvent. A. S. Wheeler. J. Amer. Chem. Soc., 1920, 42, 1842–1846.

*p-CYME*NE is the principal constituent of so-called spruce turpentine, a by-product of the manufacture of sulphite pulp, and may be obtained from it by the following process. Air is bubbled through the turpentine for 10 hrs. to remove sulphur dioxide. The liquid is then distilled with superheated steam in an apparatus so arranged that the vapours pass first through a 30% solution of sodium hydroxide and then into the condenser. The distillate is purified by shaking it with a 5% solution of sodium hydroxide and then distilling it over sodium. So prepared *p*-cymene has b.p. 176°–176.5° C., n_D^{20} = 1.4905, and only very slowly develops a yellow colour when a trace of *p*-anisidine is added and the

mixture exposed to light. Under similar conditions an impure sample develops a red colour. The solubilities of a considerable number of organic compounds in this solvent are tabulated.—W. G.

Terpin; Quantitative estimation of —. O. Fernández and N. Luengo. *Anal. Fis. Quim.*, 1920, 18, 158—165.

On treatment with acetic anhydride and anhydrous sodium acetate, only one of the two hydroxyl groups of terpin is acetylated; but when a solution of terpin in oil of turpentine is treated in like manner, complete acetylation is achieved. (*Cf. J.C.S.*, Nov.)—W. R. S.

Eucalyptol determination. C. T. Bennett and M. S. Salamon. *Perf. Essent. Oil Rec.*, 1920, 11, 302.

The results of numerous trials indicate that the cresol method (Cocking, J., 1920, 610 A) is no more reliable than the official phosphoric acid method for the estimation of eucalyptol, as the constituents of eucalyptus oil other than eucalyptol have a marked influence on the melting point of the cresol compound.—G. F. M.

Ether-air mixtures; Self-ignition of —. J. A. McClelland and H. V. Gill. *Sci. Proc. Roy. Dublin Soc.*, 1920, 16, 109—119.

IGNITION occurs when a mixture of ether vapour and air is allowed to rush into a partially evacuated tube. Measurements of the temperature in the tube showed that the rise of temperature produced reached the ignition point of the mixture. The rise of temperature is attributed to the conversion of the translational energy of the gas rushing into the tube into heat. The length of the explosion tube affects the results.—J. R. P.

Pharmacology of selenium and tellurium. I. Action of their acids on bacteria. G. Joachimoglu. *Biochem. Zeits.*, 1920, 107, 300—313.

THE IONS of tellurous and selenious acids are much more active than those of telluric and selenic acids. The growth of moulds is hardly affected by the action of tellurites and tellurates. Bacteria, especially of the typhus-coli group, are, on the other hand, very sensitive to the action of these substances. The author suggests the application of tellurium compounds in the chemotherapy of typhoid infection.—S. S. Z.

Isoamyl alcohol. Martin. *See* XVIII.

Weak acids and bases. Kolthoff. *See* XXIII.

PATENTS.

Saccharic and tartaric acids; Production of — from carbohydrates. Diamalt-A.-G. E.P. 108,494, 4.8.17. Conv., 4.8.16.

SACCHARIC and tartaric acids are readily obtained by oxidising carbohydrates at 100° C. in presence of oxidising catalysts, particularly molybdenum, mercury, or platinum, by means of sulphuric and nitric acids, or sulphuric acid and oxides of nitrogen, peroxidised by treatment with oxygen, ozone, or air. For example, 100 pts. of starch is mixed with 150 pts. of water and 3 pts. of sulphuric acid, and saccharified. Concentrated sulphuric acid (25 pts.), nitric acid of sp. gr. 1.4 (60 pts.), and an oxide of molybdenum (1 pt.) are then added, and the whole is maintained at 100° C., a further 100—140 pts. of nitric acid being added during the course of the reaction. At the end of the operation the nitric acid is evaporated, and after diluting the residual liquor with water, the molybdenum is precipitated by hydrogen sulphide, and the sulphuric and traces of oxalic acid by lime or baryta, and saccharic acid separated from the filtrate after

concentration as potassium hydrogen saccharate. The yield is 65%. If mercury is used as catalyst and twice as much nitric acid is added, a yield of 60% of potassium hydrogen tartrate, together with about 10% of saccharate, is obtained.—G. F. M.

Acetic acid from acetylene as primary material; Catalytic processes for preparing —. Soc. des Acieries et Forges de Firminy. E.P. 132,529, 18.2.19. Conv., 13.3.18.

THE vapours of acetaldehyde produced by the catalytic hydration of acetylene (E.P. 124,194; J., 1920, 582 A) are absorbed from the gaseous current by means of a 15—20% solution of sulphuric acid at 20° C., which polymerises the aldehyde to paraldehyde. The latter is separated from the acid and is depolymerised by boiling with 30—50% sulphuric acid and converted into acetic acid by the action of air or oxygen in presence of copper permanganate or a mixture of copper and manganese acetates. The object of the temporary polymerisation of the aldehyde is to attain a better recovery of the product from the reaction gases by conversion into the less volatile and more easily handled paraldehyde, and it also enables the material to be temporarily stored without risk in the event of a breakdown of the oxidising plant. The hydration, polymerisation, depolymerisation, and oxidation reactions are conveniently carried out in a series of five sandstone towers which are traversed from the bottom upwards by the reacting gases in counter-current with the liquid reagents. The first tower is used only for removing the excess aldehyde from the spent hydrating catalyst previous to its electrolytic regeneration, and each of the remaining towers serves for one of the four reactions above mentioned.—G. F. M.

2-Phenylquinoline-4-carboxylic acid; Manufacture of the allyl ester of —. O. Imray. *From Soc. of Chem. Ind. in Basle.* E.P. 150,401, 28.5.19.

THE allyl ester of 2-phenylquinoline-4-carboxylic acid is, like the alkyl esters, tasteless, and has the further advantage of dissolving uric acid more readily than either the alkyl esters or the acid itself. It may be prepared by any of the usual methods of esterification, such as the action of an allyl halide on a salt of the acid, or by the action of allyl alcohol on the acid chloride or on the acid itself in presence of hydrogen chloride or sulphuric acid. The ester boils at 260° C. under 15 mm. pressure, and melts at 30° C. Its hydrochloride crystallises from alcohol in small citron-yellow needles, m.p. 145°—147° C.—G. F. M.

Nitro-compounds; Reduction of aromatic —. J. Tcherniac and S. H. Davies. E.P. 150,412, 29.5.19.

AROMATIC nitro-compounds, particularly hydroxy-nitro-compounds, are reduced by treatment with a mixture of hydriodic and hydrochloric acids. The consumption of iodine may be reduced to a minimum by adding a substance, such as phosphorus, which in presence of water re-converts the liberated iodine into hydriodic acid. An example is given of the reduction of *p*-nitrophenol by boiling 139 pts. with 36 of amorphous phosphorus, 13.9 of iodine, and 180 of 20% hydrochloric acid under a reflux condenser. The reaction is completed when iodine vapour appears in the condenser.—G. F. M.

Thienylquinolinecarboxylic acids. M. Hartmann and E. Wybert. Assignors to Soc. of Chem. Ind. in Basle. U.S.P. 1,350,408, 24.8.20. Appl., 21.10.18.

2-THIENYLQUINOLINE-4-CARBOXYLIC acids, obtained by condensing isatin compounds with acetothienone, are crystalline compounds of definite melting point. They are soluble in most organic solvents and in strong mineral acids, and are of value for histological and therapeutical purposes.—G. F. M.

Desoxycholic acid; Process of obtaining —. H. Wieland. G.P. 321,726, 10.8.16.

Desoxycholic acid is precipitated as an alkaline salt from its solution in strong alcohol.—C. A. M.

Carbamides [ureas]; Method for producing asymmetric —. A. V. Blom. U.S.P. 1,351,888, 7.9.20. Appl., 3.4.19.

SEE E.P. 125,581 of 1919; J., 1920, 582 A.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic emulsions; Size and sensitiveness of the grains in —. T. Svedberg. Z. wiss. Phot., 1920, 20, 36—50.

IN considering the blackening-law (*Schwarzungsgesetz*) of a photographic plate and the behaviour of the individual grains contained in it, the view that the law is a consequence of the lack of uniformity of the photo-chemical light-field inside the film is negated by the work of Luppo-Cramer, Scheffer, and others, and by the author's own experiments with very thinly coated plates for which the usual type of characteristic curve was obtained. The possibility that each grain obeys the law of the plate appears to be inadmissible, since the size of grain resulting by development appears to be dependent on the amount of development and not on the exposure. In experimental examination of the two remaining possibilities, viz., that each class of grains of approximately equal sizes obeys its own law, or that only an emulsion with grains of varying sizes obeys the law, uniform thin coatings of very dilute emulsions were used; after exposure and development the plates were treated with a silver solvent (acid potassium permanganate solution or acid potassium bichromate solution) and photomicrographs of the resulting films taken with a linear magnification of 1000. Prints of the micrographs were made with a further magnification of 4 and the grains for a definite area of each print counted and assorted into 4 classes of size with the aid of a screen ruled in sq. mm. The grains counted in this way are of course those which are not developed, and the numbers and sizes of the developed grains have to be obtained by comparison with the same emulsion untreated. Tables and curves are given for one type of plate, showing the relationship between exposure and percentage of grains affected in the 4 size-classes, and between size of grain and the percentage number attacked; the curve for the former is similar to the usual characteristic curve and that for the latter to an exponential. Similar results are given for solarisation exposures, for hydrogen peroxide effect and for ordinary chemical fog, and the results are all in general agreement with the view that the larger grains are the more sensitive.—B. V. S.

Photographic printing process without silver bromide. K. Wilcke. Phot. Korr., 1920, 57, 173—175. Chem. Zentr., 1920, 91, IV., 380.

THE process depends on the increase of electrical conductivity of selenium on exposure to light. A glass plate, provided with an excessively thin coating of gold or platinum, is placed in intimate contact with a thin sheet of selenium. In contact with the other side of the selenium is placed a sheet of paper soaked in a solution of some substance, such as potassium ferrocyanide, which gives a coloured substance on passage of an electric current; the paper is backed up by a metal plate. When the glass side of the system is exposed in a camera, an electric potential difference being applied to the gold or platinum film on one side and the metal

plate on the other side, a reproduction is obtained on the paper.—B. V. S.

PATENT.

Celluloid films. U.S.P. 1,352,216. See V.

XXII.—EXPLOSIVES; MATCHES.

Nitroglycerin; Application of Devarda's method to the determination of nitrogen in —. Kochler, Marquoyrol, and Jovinet. Ann. Chim. Analyt., 1920, 2, 271—272.

ABOUT 0.5 g. of nitroglycerin is placed in a flask together with 40 c.c. of hydrogen peroxide (12 vol.) and 6 c.c. of potassium hydroxide solution (sp. gr. 1.4), the mixture is agitated by a mechanical stirrer (1500 revs. per min.) and heated gradually to 40° C. After 40 mins., the walls of the flask are rinsed down with a few c.c. of alcohol and the heating continued at 70° C. for a short time. The saponification is thus completed and the nitrate in the solution is reduced in the usual way with Devarda's alloy (J., 1893, 550), the resulting ammonia being determined by distillation.—W. P. S.

"Brisance" of explosives; Measurement of the —. H. Kast. Z. ges. Schiess- und Sprengstoffw., 1920, 15, 171—173, 181—184.

THE Trauzl lead block test, although it gives results having a definite relationship to the calculated specific pressure for the same classes of explosive, takes no account of density or of the time factor. Tables are given showing the figures for specific volume, explosion temperature, solid residue, specific pressure, and lead block tests, for various explosives, as well as the effect of density on the lead block expansion. The nature of the casting, and the temperature during the test also exert an influence on the results. The causes of the discrepancies between practical results and energy calculated from the formula $t = p_0 v_0 t / 273(1 - a)$ are discussed. The author gives equations representing the decomposition of a series of explosives, together with the calculated values for Q , Q_0 , t , i , and v_0 . In contrast with the Trauzl block, the lead crusher test (Kast, Z. ges. Schiess- u. Sprengstoffw., 1913, 8, 88) provides a more accurate means of determining the energy of explosives in that it takes into account the density and velocity of detonation. It has the disadvantage of being inapplicable to explosives which are not easily detonated. Comparative results obtained with the lead block and the lead crusher with trinitrotoluene and ammonium nitrate mixtures are tabulated, and a table showing lead-crusher tests in relation to density, velocity of detonation, specific pressure, and the calculated brisance is appended.—W. J. W.

Military explosives; Strength and velocity of detonation of various —. W. C. Cope. J. Ind. Eng. Chem., 1920, 12, 871—873.

TEN grms. of TNT is fired in a standard mortar, and then by means of trial shots the weight of the explosive under test which will deflect the mortar by an approximately equal number of ballistic degrees is determined. A table of the TNT values of the most common explosives is given. The velocities of detonation of various explosives under definite conditions of density and confinement are tabulated. Tetryl had a velocity of 5450 m. per sec., as compared with 4600 m. per sec. for TNT. Coarse ammonium nitrate has no deleterious effect on the velocity of amatol, especially in the presence of 55% of TNT. Fragmentation tests, in which the number and sizes of the fragments resulting from detonation in steel shells were measured, showed that in this respect TNT was superior to a mixture of TNT and ammonium nitrate or of TNT, tri-

nitroxyline, and ammonium nitrate, although inferior in strength.—C. A. M.

After-corrosion in the bores of firearms; Cause and prevention of —. W. J. Huff. *J. Ind. Eng. Chem.*, 1920, 12, 862—870.

THE present high-pressure smokeless cartridges leave no nitrocellulose or corrosive acid residue. After-corrosion is due to the deposition of water-soluble salts (e.g., potassium chloride from the primer), with subsequent exposure to excessive humidity in presence of oxygen. These salts are retained in pitholes in the boring, and cannot be mechanically removed. After-corrosion may be prevented by tightly closing both ends of the bore after firing. The present U.S.A. service ammunition may be rendered non-corrosive by eliminating potassium chlorate from the primer. A simple method of testing cleaning compositions is described. Corrosion from nitrocellulose powder probably only occurs when the confining pressure is very low.—C. A. M.

PATENTS.

Explosive powder; Perforated pellets of — and process of making same. F. I. and E. du Pont. U.S.P. 1,352,121, 7.9.20. Appl., 12.6.18.

EXPLOSIVE pellets are made from black powder dust formed into masses by means of collodion.—W. J. W.

Fertilisers from explosives. G.P. 321,878. See XVI.

XXIII.—ANALYSIS.

Indicator; A polychromatic —. J. Moir. *J. S. Afr. Assoc. Anal. Chem.*, 1920, 3, 6—8.

THE indicator is 3,3'-dihydroxybenzaurine-2"-sulphonic acid (catecholsulphonphthalein), and is prepared by condensing catechol with *o*-sulphobenzoic acid. It shows the following range of colours in acid and alkaline solutions:—Strong acid, purple-pink; N/10 acid, orange; N/40 acid, yellow; neutral, colourless or very faint green; just alkaline, violet; dilute alkaline, indigo-blue; strong alkaline, grass-green.—W. P. S.

Hydrogen-ion concentration of some standard solutions at various temperatures. L. E. Walbum. *Biochem. Zeits.*, 1920, 107, 219—229.

THE H-ion concentration of mixtures of glycine and sodium hydroxide, borate and hydrochloric acid, borate and sodium hydroxide increases with increased temperature between 10° and 70° C. The reverse is the case with a citrate and sodium hydroxide mixture. In the former three mixtures the alteration is more marked in the alkaline mixtures and diminishes with diminution of the alkalinity of the mixtures.—S. S. Z.

Analysis; Apparatus for evolution methods of —. [Determination of sulphur and carbon in steel.] E. R. Dovey. *Analyst*, 1920, 45, 330—332.

AN apparatus for the determination of sulphur in steel, etc., consists of a small reaction flask provided with a side tube connected with a tube extending to the bottom of a test-tube; the latter contains an absorbent for the gas evolved from the flask. The test-tube is connected, in turn, with a U-tube containing a small quantity of the absorbent solution. A second apparatus described is suitable for the absorption of carbon dioxide in the wet combustion method for the determination of carbon in steel. The reaction flask is attached to a short reflux condenser through the centre tube of which the stem of a tapped funnel extends nearly to the bottom of the flask; the evolved gases are conducted through a tube leading from the top of the condenser to the jet of a burette containing

a rod fitted with discs and partially filled with barium hydroxide solution. The top of the burette is connected with an aspirator so that a current of air free from carbon dioxide may be drawn through the apparatus. At the end of the operation the burette is disconnected, and the jet passed through a rubber stopper closing a filter-tube, so that the barium carbonate formed may be filtered off without contact with the atmosphere.—W. P. S.

Weak acids; Titration of — with weak bases. I. M. Kolthoff. *Pharm. Weekblad*, 1920, 57, 787—796.

THE exact titration of a weak acid with a weak base is possible if the indicator is suitably chosen. An expression for the hydrogen-ion concentration of a solution of a salt of a weak acid and a weak base is derived, involving only the dissociation constants of the base, the acid, and water. With the help of this equation the degree of hydrolysis of the neutral salt solution may be calculated. An indicator is then chosen for which the colour change takes place at or about the same p_H . Exact results were obtained in the titration of ammonia with acetic, oxalic, succinic, formic, and salicylic acids, using in the first three cases neutral red as indicator, and methyl red in the last two.

—W. S. M.

Gases; Application of the thermal conductivity method to the automatic analysis of complex mixtures of —. E. R. Weaver and P. E. Palmer. *J. Ind. Eng. Chem.*, 1920, 12, 894—899.

THE method previously described (J., 1920, 470 A) is applicable in numerous directions. By comparison with a standard gas of known composition (air or hydrogen) helium may be determined in admixture with nitrogen, hydrogen in air, and impurities in hydrogen for balloons. Comparison of the thermal conductivity before and after a chemical reaction affords a means of determining chlorine, sulphur dioxide, acetylene, water vapour, etc., in various gaseous mixtures. Further, a gas, such as hydrogen, may be added to a mixture (flue gas), the oxygen removed by combustion, and its amount determined from the difference in the conductivity before and after the reaction. In other cases successive combustion with oxygen and hydrogen may be used, as in the determination of carbon monoxide in presence of large amounts of hydrogen.

—C. A. M.

Potassium and sodium; Estimation of — when present together [as chlorides]. A. Quartaroli. *Gaz. Chim. Ital.*, 1920, 50, ii., 64—69.

THE method consists in converting the mixture of chlorides into nitrates and determining the solidifying point of the mixed nitrates.—T. H. P.

Calcium and magnesium in different saline solutions; Estimation of —. E. Canals. *Comptes rend.*, 1920, 171, 516—518.

THE whole of the calcium and magnesium may be separated from the iron and aluminium in a solution containing salts of the four metals, by acidifying the solution with sulphuric acid, adding sodium phosphate, making alkaline with ammonia, and then acidifying with acetic acid and shaking vigorously for several minutes. In this way the calcium and magnesium pass into solution, whilst the precipitated iron and aluminium remain undissolved.—W. G.

Diphenyl derivatives; Use of — in qualitative analysis. F. Feigl. *Chem.-Zeit.*, 1920, 44, 689—690.

TO detect minute traces of manganese, ceric, cobaltic, or thallic compounds, their solution is rendered alkaline with sodium hydroxide, filtered, and the filter then treated with a drop of benzidine

acetate solution; an intense blue coloration is obtained. The reaction will detect 1 part of manganese in 125 million parts of solution. Thallic salt solutions give the coloration without previous treatment with alkali; slightly acid iron solutions and those from which the iron has not been precipitated completely by alkali also give a blue coloration with the reagent. Halogen- and alkyl-substituted benzidines and tolidines may be used in place of benzidine.—W. P. S.

Iodine; Estimation of —. E. C. Kendall. J. Biol. Chem., 1920, 43, 149—159.

MODIFICATIONS of the method previously published (J., 1912, 748) are described which enable the determination of iodine in blood and tissues to be carried out satisfactorily.—J. C. D.

Iodine; Estimation of — in blood and animal tissues. E. C. Kendall and F. S. Richardson. J. Biol. Chem., 1920, 43, 161—170.

A METHOD for destroying the large amount of organic matter in blood and animal tissues without loss of iodine is described. (Cf. J.C.S., n., 631.) —J. C. D.

Phosphoric acid; Estimation of —. Parts 1, to F. H. Kleinmann and J. Feigl. Biochem.-Zeits., 1919, 99, 19—190.

Estimation as magnesium ammonium phosphate and effect of serum constituents on the precipitation.—Serum constituents, e.g., dihydrogen potassium phosphate, urea, glycine, and calcium carbonate do not influence the precipitation of phosphoric acid as magnesium-ammonium-phosphate. The presence of sodium chloride in certain concentrations, however, leads to high results. Reagents employed in the precipitation of proteins, e.g., picric acid and trichloroacetic acid, influence the precipitation, although they do not hinder it. Serum does not contain any substances which are capable of preventing the complete precipitation of phosphoric acid as magnesium-ammonium phosphate. On precipitating 1 mg. P_2O_5 as magnesium-ammonium phosphate in a volume of solution of 50—75 c.c., 0.006 mg. remains unprecipitated. With a smaller quantity, e.g., 0.1 mg. P_2O_5 , the unprecipitated fraction is even proportionately higher.

Estimation as phosphoric acid-molybdenum complex phosphate.—The uranium acetate method yielded good results with quantities as low as 10 mg. P_2O_5 . Quantities below 10 mg. gave unreliable results. With Liebermann's silver phosphate method high results were obtained, while the estimation of phosphoric acid as the vanadium-phosphoric acid-molybdenum complex was found satisfactory.

Estimation as phosphoric acid-molybdenum complex. Gravimetric and colorimetric methods.—The precipitation of molybdenum by Raper's method gave too high values. The most suitable results were obtained with potassium ferrocyanide. 1 mg. of Mo was estimated in 100 c.c. with an error of 0.5%. The procedure was therefore successfully applied for the colorimetric estimation of phosphoric acid. 0.1 mg. of P_2O_5 can be estimated by this method with a 0.5% error. *Estimation in the phosphoric acid-molybdenum complex. Volumetric and sedimentic methods.*—Low results were obtained with Neumann's method for the estimation of phosphorus when the theoretical factor was used. By washing the phosphorus-ammonium-molybdate precipitate with 50% alcohol instead of with water better results were obtained. In micro-Neumann estimations it is more satisfactory to wash the precipitate than centrifuge it, as a certain amount of loss is thus obviated. A "sedimentic" method is described by means of which the phosphorus is rapidly estimated by measuring the volume of the precipitate after centrifuging it in a graduated tube. The height of

the phosphorus-ammonium-molybdate precipitate is proportional to its P_2O_5 -content. *Estimation as a strychnine-phosphoric acid molybdenum compound. (Nephelometry.)*—The development of nephelometry and a new nephelometer and its manipulation are described. *Estimation as a strychnine-phosphoric acid molybdenum compound. Special phosphoric acid nephelometry and a new preparation of the strychnine-molybdenum-reagent.*—Strychnine and molybdenum form a compound suitable for use in estimation of phosphoric acid. 0.1—0.0005 mg. P_2O_5 can be estimated in this way nephelometrically with an error of 0.5%.—S. S. Z.

Phosphoric acid; Volumetric determination of — by Neumann's method. P. Iversen. Biochem. Zeits., 1920, 104, 15—21.

THIS method for determining phosphorus in organic substrata consists in destroying the organic matter by treatment with sulphuric and nitric acids, precipitating the phosphate thus formed as ammonium phosphomolybdate, and washing the precipitate with ice-cold water, which is passed through a filter. The latter, together with all the precipitate, is then treated with rather more $N/2$ sodium hydroxide solution than is necessary to dissolve the precipitate, the ammonia thus liberated being boiled off and determined by titration: (e.c. of $N/2 NaOH$) $\times 0.5539$ = amount of phosphorus in mg. Gregersen found it to be necessary to over-titrate with 0.5—1 c.c. of acid and to boil to expel carbon dioxide. The author's results show that this modification gives good results provided that the excess of $N/2$ alkali taken is at most 4 c.c., and is subject to no systematic error; the mean value of the factor is found, from 25 analyses, to be 0.5526.—T. H. P.

Selenium [in organic compounds]; Estimation of —. F. Wrede. Z. physiol. Chem., 1920, 109, 272—275.

THE substance is burned in a tube in oxygen in presence of platinum as catalyst and the selenious acid formed is titrated with $N/100$ sodium hydroxide solution, sodium hydrogen selenite being neutral to methyl orange. When both sulphur and selenium are present, the mixed sulphuric and selenious acids obtained are titrated and the sulphuric acid then estimated separately as barium sulphate. If the substance contains either nitrogen or a halogen titration is useless, and the selenious acid may then be reduced by means of sodium sulphite in presence of a large amount of hydrochloric acid and the selenium thus formed weighed. The combustion is carried out in a so-called bead-tube (Pregl, "Die quant. org. Mikroanal.," 1917, 107) of about 7 mm. bore, the beads, previously moistened with water, serving for catching the selenious acid. In the empty part of the tube are placed one or two 5 cm. "stars" of thin platinum foil, the boat containing the substance lying between the "stars" and the end of the tube. The flow of oxygen should be 8—10 c.c. per min. Over the capillary end of the tube an Erlenmeyer flask is inverted, and into this the selenious acid is subsequently washed. Before being used for the first time the combustion tube is treated with caustic alkali solution and then with acid and is finally rinsed out thoroughly with freshly boiled distilled water to which methyl orange and dilute acid or alkali have been added until it is just not pure yellow; the excess of water is blown out, but the tube is not dried. The ignited platinum stars and the boat are introduced, the tube at the stars being heated to bright redness and the substance burnt very slowly. The selenious acid separates in shining white crystals at the beginning of the beads, the appearance of red selenium or of carbon indicating too rapid combustion. The acid is rinsed out into the Erlenmeyer flask by means of the methyl orange water and the solution titrated

with $N/100$ sodium hydroxide solution. If sulphur as well as selenium is present, the bead tube is moistened, before combustion, with neutral 5% hydrogen peroxide solution, the combustion being carried out as usual. The platinum stars are ignited after each combustion or etched with *aqua regia*, as they often become coated with a thin layer which reduces their catalytic action. The method gives good results with small quantities (0.02—0.06 g.) of substance.—T. H. P.

See also pages (A) 683, *Specific heat of mineral oils* (Bailey and Edwards); 685, *Dyes* (Moir); 687, *Carbon dioxide* (Robinson); *Thiocyanates* (Meurice); 691, *Steel* (Whiteley); 693, *Lead-antimony alloys* (Bertiaux); 697, *Chinese wood oil* (Jameson); *Turpentine oil* (Allina and Salvaterra); 699, *Calcium in soils* (Shead); 702, *Cocaine* (Annett and Sen); 703, *Dichloroethyl sulphide* (Martin); *Organic acids* (Kolthoff); 704, *Terpin* (Fernández and Luengo); *Eucalyptol* (Bennett and Salamon); 705, *Nitroglycerine* (Koehler and others).

PATENTS.

Gas analysing and recording apparatus. A. B. Cunningham, Toledo, Ohio. U.S. Pat. 1,311,952, 5.8.19. Appl., 8.4.19.

THE gas is drawn into the apparatus by means of a water jet, and a measured volume is forced by the rising of the water level into an absorption tank wherein the soluble portion is absorbed; the residual gas passes to a measuring bell, where its volume is recorded. Arrangements are provided for automatically setting the apparatus after each test for a fresh operation.

Moisture in solid materials; Method and apparatus for determination of —. L. E. McAllister. U.S.P. 1,347,106, 20.7.20. Appl., 8.5.19.

THE apparatus comprises means for drying air, for determining the temperature of the dry air, for passing the air through a tube containing the sample, and for determining the change in temperature of the sample and the rate of flow of the air.—J. H. L.

Gases; Apparatus for automatic and continuous analysis and recording of —. W. D. R. Brown and W. T. Pickston. E.P. 149,752, 16.5 and 30.10.19.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Abbott. Non-conducting linings or coverings of heat retaining or excluding chambers. 28,621. Oct. 9.

Arcari. Combined mixing and crushing device. 28,255. Oct. 6.

Barbet et Fils et Cie. Distilling and rectifying columns. 27,689. Sep. 29. (Fr., 1.10.19.)

Baxter Asphalt Paving Co. 27,425. See IX.

Bedford, Fuller, and Roberts. Furnaces. 28,582. Oct. 8.

Bruhl. Grinding rollers or balls. 28,340. Oct. 6.
Douglas. Condensers, dephlegmators, and column stills. 27,965. Oct. 2.

Fraymouth, Reavell, and Kestner Evaporator and Engineering Co. Extraction of soluble matter from powdered material other than tanstuffs. 28,173. Oct. 5.

Keene. Filtering apparatus. 28,100. Oct. 4.

Kirkbride. Crushing mills. 27,816-7. Oct. 1.

Morison. Treatment of boiler feed etc. water. 28,590. Oct. 8.

Schmieding. Regenerative furnaces. 27,898. Oct. 1. (Ger., 30.6.19.)

Wade (Barrett Co.). 28,472. See XX.

COMPLETE SPECIFICATIONS ACCEPTED.

13,050 (1919). Bulgin, Hall, and Searle. Rotary machines for treating air or gases with liquids and *vice versa*. (151,314.) Oct. 6.

15,841 and 15,844 (1919). Smallwood. Furnaces. (151,366-7.) Oct. 6.

16,063 (1919). West and Hodgson. Kilns. (151,709.) Oct. 13.

16,247 (1919). Dressler Tunnel Ovens, Ltd., and Williamson. Continuous and intermittent ovens. (151,722.) Oct. 13.

16,665 (1919). Gilmour. Manufacture of heat-insulating material for steam and other pipes, boilers, etc. (151,738.) Oct. 13.

20,148 (1919). Davis. Pulverisers. (151,440.) Oct. 6.

25,673 (1919). Fleury and Robertson. Chambers for drying. (151,488.) Oct. 6.

29,687 (1919). Phillips and Rose. Comminution and mixing of materials. (151,508.) Oct. 6.

32,851 (1919). Imray (Kuntz). Rotary driers. (151,518.) Oct. 6.

10,565 (1920). Thaberg. Apparatus for extracting liquid from fresh peat and similar watery masses. (151,907.) Oct. 13.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Anderson and Macnochie. Gas-producer. 27,385. Sep. 27.

Burnell and Dawe. Motor spirit. 27,395. Sep. 27.
Chown. Carbonising, distilling, and briquetting carbonaceous material. 28,676. Oct. 9.

Goskar. Drying coal etc. 27,881. Oct. 1.

Goss (Blake). Manufacture of fuel for heat, light, and power. 27,811. Sep. 30.

Johnston. Liquid fuels. 27,771. Sep. 30.

Lewis. Distilling and gasifying solid carbonaceous materials. 27,910. Oct. 1.

Maxted and Thompson. Absorption of ethylenic hydrocarbons. 27,621. Sep. 29.

Morgan, and Thermal Industrial and Chemical Research Co. Manufacture of coal gas. 28,344. Oct. 6.

Neill and Russell. Gas-producers. 28,054. Oct. 4.

Rudder. Utilising waste products as fuel in steam-raising plant. 28,633. Oct. 9.

Sauer. 28,682. See XVII.

Tulloch. Fuel. 27,770. Sep. 30.

Wallace. Distillation of carbonaceous material. 27,798-9. Sep. 30.

COMPLETE SPECIFICATIONS ACCEPTED.

15,295 (1919). Franklin. Suction gas generators. (151,329.) Oct. 6.

20,501 (1919). Wells. Gas-cleaning apparatus. (151,443.) Oct. 6.

20,568 (1919). Yeo. Gas-producers. (151,445.) Oct. 6.

22,544 (1919). Marbais and Deguide. Complete extraction of naphthalene from coal gas. (151,463.) Oct. 6.

1263 (1920). Grocott. Gas-producer. (151,520.) Oct. 6.

5220 (1920). Kennedy. Retorts. (141,025.) Oct. 13.

10,565 (1920). Thaberg. See I.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

Glover, West, and West's Gas Improvement Co. Separation of liquor from tar and oils. 27,999. Oct. 2.

Wade (Barrett Co.). 28,472-3. *See* XX.

COMPLETE SPECIFICATION ACCEPTED.

22,544 (1919). Marbais and Deguide. *See* 11.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

Dawson. Manufacture of leuco-alizarin bordeaux and derivatives thereof. 28,376. Oct. 7.

Imray (Soc. Chem. Ind. in Basle). Manufacture of dyestuffs. 28,008. Oct. 2.

COMPLETE SPECIFICATIONS ACCEPTED.

7719 (1918). Clarke (Chem. Fabr. Worms). Colours soluble in fats and oils and process of preparing same. (151,657.) Oct. 13.

16,042 (1919). Perkin. Manufacture of anthranol. (151,707.) Oct. 13.

31,611 (1919). British Dyestuffs Corp., Turner, and Badier. Manufacture of diphenylamine derivatives. (151,868.) Oct. 13.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Bearder and Schwartz. Manufacture of cellulose acetates for producing artificial silk, films, celluloid, etc. 27,973. Oct. 2.

British Cellulose and Chemical Manuf. Co., and Richardson. Treatment of products made with cellulose derivatives. 27,531. Sep. 28.

Bustamante. Obtaining cellulose from vegetable matter. 27,904. Oct. 1.

Dreyfus. Manufacture of cellulose derivatives. 28,350. Oct. 6.

Foster (Mahy). Treating flax, hemp, etc. 27,777. Sep. 30.

Kawabe. Treating ramie, hemp, etc. 27,933. Oct. 1.

Radeliffe. Production of cellulose acetate and artificial silk therefrom. 28,413. Oct. 7.

Ringer. Manufacture of a hard substance from fibres. 27,443. Sep. 27. (Austria, 19.2.20.)

COMPLETE SPECIFICATION ACCEPTED.

16,816 (1919). Johnson and Phillips, and Tracey. Process for making paper, string, etc. (151,391.) Oct. 6.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Carpmael (Bayer u. Co.). Manufacture of mordants and process of dyeing basic dyestuffs on cotton. 27,466. Sep. 27.

Radeliffe. Dyeing artificial silk. 28,412. Oct. 7.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Blanc and Jourdan. Treatment of leucitic rocks to render the potash available. 28,295. Oct. 6. (Ital., 6.10.19.)

Collins. Purification of zinc chloride. 28,551. Oct. 8.

Kelly. Manufacture of acid sodium pyrophosphate. 27,660. Sep. 29.

Maclaure. Production of aluminium and zinc salts. 28,326. Oct. 6.

Norsk Hydro-Elektrisk Kvaestofaktieselskab. Catalyst for making ammonia. 27,491. Sep. 29. (Norway, 28.10.19.)

Oldbury Electro-Chemical Co. Manufacture of oxalates and oxalic acid. 28,356. Oct. 6. (U.S., 23.3.20.)

Pease. Extraction of ammonia from fluids and production of ammonia etc. compounds. 28,311. Oct. 6.

Soc. l'Azote Français. Recovering nitrous vapours in form of aqueous nitric acid. 28,347. Oct. 6. (Switz., 8.10.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

7869 (1919). General Chemical Co. Production of nitric acid. (124,759.) Oct. 13.

15,026 (1919). Thompson. *See* XVI.

15,406 (1919). Phillips and Rose. Manufacture of readily soluble alkali silicates. (151,339.) Oct. 6.

19,085 (1919). Chailaux. *See* XIII.

VIII.—GLASS; CERAMICS.

APPLICATION.

Duckham, and Woodall, Duckham, and Jones. Gas-fired pottery kilns. 28,248. Oct. 5.

COMPLETE SPECIFICATIONS ACCEPTED.

15,764 (1919). Marks (Universal Glass Co.). Glass bait. (151,361.) Oct. 6.

26,095 (1919). Johnson. Manufacture of china and earthenware. (151,835.) Oct. 13.

19,445 (1920). Schott u. Gen. Reduction in size of the bubbles in glass. (147,487.) Oct. 13.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Baxter Asphalt Paving Co. Bituminous lining for surfaces exposed to corrosive action. 27,425. Sep. 27. (U.S., 2.1.15.)

Ringer. Working and increasing stability of objects made of Sorel cement. 27,444. Sep. 27. (Austria, 19.2.20.)

Uzzell. Concrete for building. 27,595. Sep. 29.

Wade (Pfaff). Hydraulic cement. 27,768. Sep. 30.

COMPLETE SPECIFICATIONS ACCEPTED.

5208 (1919). Vecchis. Preserving and fire-proofing wood. (151,661.) Oct. 13.

16,665 (1919). Gilmour. *See* I.

20,174 (1919). Holzapfel and Walther. *See* X.

28,397 (1919). Honda. Road-making or paving material. (151,852.) Oct. 13.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Bourcoud. Reducing metallic oxides. 27,493. Sep. 27. (U.S., 17.1.18.)

Burden. Aluminium alloys. 28,087. Oct. 4.

Craven. Aluminium solder. 28,506. Oct. 8.

Goldschmidt A.-G. Aluminothermic production of carbon-free ferrochromium etc. for chrome-iron ore. 27,659. Sept. 29. (Ger., 14.10.19.)

Hamilton. Treatment of sulphide and oxidised ores. 28,539. Oct. 8. (Australia, 8.10.19.)

Lofts. 27,509. *See* XI.

Meagher. 27,964. *See* XI.

Metals Extraction Corp. Separation of metal from ores. 28,359. Oct. 6. (U.S., 6.10.19.)

Naef. Manufacture of metals from their sulphides. 28,139. Oct. 5.

Selas-Turner Co., and Turner. Crucible furnaces. 28,049. Oct. 4.

Vivian. Treatment of ores etc. 28,436. Oct. 7.

COMPLETE SPECIFICATIONS ACCEPTED.

15,174 (1919). August. Furnaces for heat treatment of iron, steel, etc. (151,323.) Oct. 6.

15,783 and 31,797 (1919). Elmore. Treatment of argenteiferous sulphide ores. (151,698.) Oct. 13.

15,916 (1919). Clegg. Treating tin scruff. (151,374.) Oct. 6.

- 16,401 (1919). Wild. Testing the hardness of ferrous metals. (151,383.) Oct. 6.
 17,320 (1919). Backer. Alloys. (151,400.) Oct. 6.
 20,174 (1919). Holzapfel and Walther. Composition for coating iron, steel, and wood. (151,793.) Oct. 13.
 21,603 (1919). Palmer, Seale, and Nevett. Flotation separation of mineral substances. (132,260.) Oct. 13.
 28,749 (1919). Wade (Lindsay Light Co.). Manufacture of thorium. (151,854.) Oct. 13.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

- Adam and Antonoff. Electrolysis. 27,573. Sep. 28.
 Heraeus Ges., Rohn, and Stahlwerke Lindenburg. Induction furnaces. 27,932. Oct. 1. (Ger., 19.5.20.)
 Lofts. Alloy for electric heating appliances. 27,510. Sep. 28.
 Meagher. Alloy for electrical make-and-break contacts. 27,964. Oct. 2.
 Thompson (Plauson). Electrodes. 27,727. Sep. 30.

COMPLETE SPECIFICATIONS ACCEPTED.

- 14,559 (1919). Houston. Electric batteries. (151,680.) Oct. 13.
 32,793 (1919). Levin. Electrodes of electrolytic gas generators. (151,873.) Oct. 13.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

- Ashby. Manufacture and use of washing compounds. 28,482. Oct. 8.
 Peetz. Treatment of oils. 28,449. Oct. 7.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

- Clarke, and Cookson and Co. Manufacture of paints and enamels. 28,661. Oct. 9.
 Fitzgerald. Lithographic inks. 28,124. Oct. 4.

COMPLETE SPECIFICATIONS ACCEPTED.

- 8290 (1919). Ivinston and Roberts. Paint etc. (151,666.) Oct. 13.
 14,008 (1919). Simonson and Blair. Producing indurated compounds from lac or its derivatives. (127,825.) Oct. 13.
 14,845 (1919). Koppers Co. Manufacture of paint or varnish. (129,264.) Oct. 6.
 15,373 (1919). Schneider. Manufacture of paints. (151,687.) Oct. 13.
 15,860 (1919). Koppers Co. Manufacture of resin. (132,229.) Oct. 13.
 19,085 (1919). Chaillaux. Manufacture of gold-coloured sulphurs and vermilion of antimony. (151,422.) Oct. 6.
 28,726 (1919). Richard and Dubois. Paint. (151,853.) Oct. 13.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

COMPLETE SPECIFICATION ACCEPTED.

- 15,530 (1919). Davidson. Treatment of raw rubber when freshly coagulated from latex. (151,344.) Oct. 6.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

- Carmichael and Ockleston. Treatment of hides, skins, etc. 27,837-8. Oct. 1.
 Clark (Chem. Fabr. Worms). Tanning hides. 28,247. Oct. 5.
 Fraymouth, Reavell, and Kestner Evaporator and Engineering Co. Extraction of tannin from tanstuffs. 28,174. Oct. 5.

- Fraymouth, Reavell, and Kestner Evaporator and Engineering Co. Treatment of powdered etc. tanstuffs. 28,175. Oct. 5.

COMPLETE SPECIFICATION ACCEPTED.

- 16,672 (1919). Boston and Sons, and Fairrie. Liming of hides. (151,387.) Oct. 6.

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

- Bitterling, Lyman, and Bitterlings, Ltd. Fertilisers. 28,158. Oct. 5.

- Dumont. Manufacture of combined phospho-organic manure. 28,001. Oct. 2.

- Soc. l'Azote Français. Granulating and coating calcium cyanamide. 28,059. Oct. 4. (Fr., 4.10.19.)

COMPLETE SPECIFICATION ACCEPTED.

- 15,026 (1919). Thompson (Comp. des Phosphates de Constantine). Mechanical treatment of tricalcic phosphates. (151,684.) Oct. 13.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATION.

- Sauer. Manufacture of decolorising carbon. 28,682. Oct. 9.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

- Bedford, and Burmah Oil Co. Manufacture of alcohol. 28,352. Oct. 6.

- Holzverkohlungs Ind. A.-G. Production of vinegar. 28,227. Oct. 5. (Ger., 23.10.19.)

- Scott. Producing condition and head of draught beer. 27,738. Sep. 30.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

- Atmosterol, Ltd., and Wallace. Sterilisation, disinfection, etc. 28,102. Oct. 4.

- Carmichael and Co., and Guillaume. Treatment of offal, sewage, etc., and extraction of by-products. 28,258. Oct. 6.

- Coopman. Food product. 28,289. Oct. 6.

- Leffler. Manufacture of cocoa substitute. 28,126. Oct. 4.

- Leffler. Bettering smell and taste of foodstuffs. 28,127. Oct. 4.

- MacLachlan. Treatment of waste organic substances. 27,780. Sep. 30.

- Morison. 28,590. See I.

- Remington. Treatment of wheat. 28,235. Oct. 5.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

- Bedford, and Burmah Oil Co. 28,352. See XVIII.

- Blagden, and Howards and Sons. Manufacture of calcium, magnesium, and lithium acetylsalicylates. 28,343. Oct. 6.

- Dreyfus. Manufacture of alkyl compounds. 27,646. Sep. 29.

- Johnson (Badische Anilin u. Soda Fabr.). Manufacture of alcohols, ketones, etc. 28,657. Oct. 9.

- Wade (Barrett Co.). Purifying chemical compounds. 28,472. Oct. 7.

- Wade (Barrett Co.). Manufacture of oxidation products of naphthalene. 28,473. Oct. 7.

COMPLETE SPECIFICATIONS ACCEPTED.

- 15,776-7 (1919). U.S. Industrial Alcohol Co. Apparatus for making esters. (130,969 and 130,970.) Oct. 6.

I.—GENERAL; PLANT; MACHINERY.

Evaporator design; Studies in —, W. L. Badger and E. M. Baker. Amer. Inst. Chem. Eng., 29.6.20. Chem. and Met. Eng., 1920, 23, 569-574. (See this J., 1920, 617A, 681A.)

THE influence of the elevation of boiling point, resulting from increase in salt concentration, upon the effective temperature drop between the heating steam and the liquid being evaporated is dealt with. If the boiling point rises and the temperature of the heating medium remains constant there results a loss of evaporator capacity but not of thermal efficiency. The published figures relating to increase of boiling point with increase of salt (NaCl) concentration differ considerably, and fresh determinations have been made. The results are given in a number of curves and tables, together with a

Evaporating liquors; Method of — and apparatus therefor. Metallbank und Metallurgische Ges. A.-G. E.P. 110,059, 9.2.20. Conv., 13.3.19.

Is an evaporator where the vapour from the liquor is compressed and used as the heating medium, the vapour which remains uncondensed after such use is returned to the liquor space of the evaporator or to some other point on the inlet side of the compressor, the object being to keep in circulation a larger quantity of vapour than is theoretically necessary, so that the partial pressure of any entrained air is reduced.—B. M. V.

Evaporation of liquids or recovery of vapours or gases developed therefrom; Apparatus for —. F. Merz. E.P. 150,785, 4.6.19.

A gas is kept in circulation between a cooling coil, a heating coil (or other means of cooling and heat-

Vapour Pressure of Sodium Chloride Solutions at Various Temperatures.

Vapour pressure of solution at temp. T.	G=0	G=4	G=8	G=12	G=16	G=20	G=24	G=28	G=32	G=36	Saturated solution.
760	100.0	100.6	101.2	102.0	102.8	103.7	104.6	105.6	106.6	107.6	108.7
740	99.3	99.9	100.5	101.3	102.1	103.0	103.9	104.9	105.8	106.8	108.0
700	97.8	98.4	99.0	99.8	100.6	101.5	102.4	103.4	104.3	105.3	106.5
660	96.1	96.8	97.4	98.2	98.9	99.8	100.7	101.8	102.7	103.7	104.8
620	94.4	95.0	95.7	96.5	97.2	98.0	99.0	99.9	100.8	101.9	103.0
580	92.6	93.3	93.9	94.6	95.4	96.2	97.1	98.0	99.0	100.0	101.1
540	90.7	91.4	92.0	92.7	93.5	94.2	95.2	96.1	97.1	98.1	99.0
500	88.7	89.4	90.0	90.7	91.4	92.1	93.1	94.1	95.0	96.0	96.7
460	86.5	87.3	87.9	88.5	89.2	90.0	90.9	91.9	92.9	93.9	94.7
420	84.2	84.8	85.5	86.2	86.9	87.6	88.5	89.5	90.5	91.6	92.3
380	81.7	82.3	83.0	83.6	84.3	85.1	86.0	86.9	87.9	89.0	89.8
340	78.9	79.6	80.2	80.8	81.5	82.3	83.2	84.1	85.1	86.1	86.9
320	77.5	78.1	78.7	79.3	80.0	80.8	81.7	82.6	83.5	84.6	85.3
300	75.9	76.5	77.1	77.7	78.4	79.2	80.0	80.9	81.9	82.9	83.6
280	74.3	74.9	75.5	76.1	76.7	77.5	78.3	79.2	80.2	81.2	81.8
260	72.5	73.1	73.7	74.2	74.9	75.8	76.6	77.4	78.4	79.4	79.9
240	70.7	71.2	71.8	72.4	73.0	73.8	74.6	75.5	76.4	77.5	77.9
220	68.7	69.2	69.8	70.3	70.9	71.7	72.5	73.5	74.3	75.4	75.9
200	66.6	67.0	67.6	68.1	68.8	69.6	70.4	71.2	72.2	73.1	73.7
180	64.1	64.7	65.3	65.8	66.4	67.2	67.9	68.8	69.8	70.8	71.2
160	61.6	62.1	62.7	63.2	63.8	64.6	65.3	66.1	67.1	68.1	68.6
140	58.6	59.2	59.8	60.3	60.8	61.5	62.2	63.1	64.1	65.2	65.6
120	55.4	55.8	56.3	56.9	57.5	58.1	58.9	59.8	60.7	61.8	62.2
110	53.6	54.0	54.5	55.1	55.7	56.2	57.0	57.9	58.8	60.0	60.3
100	51.7	52.0	52.5	53.0	53.6	54.3	55.0	55.9	56.8	57.9	58.2

bibliography of the subject. The more important results, taken from smoothed curves, are summarised in the above table.—W. H. C.

PATENTS.

Centrifugal pumps for acids and corrosive liquids. W. P. Thompson. From Union Espagnole de Fabriques d'Engrais, de Prod. Chim., et de Superphosphates. E.P. 127,831, 6.6.19.

To minimise vibration the shaft of the pump is supported at both ends on bearings well outside the pump, and the glands have double packings with means for forcing a lubricant to the space between the two rings of packing. The liquid to be pumped is admitted to both sides of the rotor, and a valve is provided on the inlet pipe which can be adjusted to produce a slight vacuum round the shaft in order to prevent the liquid from penetrating the packing.—B. M. V.

Mixing of gases with liquids or solids (e.g., hydro-generation of oils); Apparatus for intensifying the mutual action one on the other during —. La Soc. Anon. l'Oxyhydrique Française. E.P. 129,978, 11.7.19. Conv., 19.7.18.

AN apparatus, e.g., for use in the catalytic hydro-generation of oils, consists of a horizontal cylindrical vessel provided with a rotating eccentrically disposed agitator, the periphery of the blades of which is almost in contact with the lowest point of the vessel. When a finely-divided catalyst is present, the ends of the blades are scoop-shaped to scrape up the paste which settles at the bottom of the vessel, and project it into the gas above the liquid.—L. A. C.

ing), and a vessel containing the liquid to be evaporated. The vapour absorbed by the warm gas is condensed on cooling, and means are provided for heat interchange between the hot moist gas and the cooled dry gas. The surface of the liquid exposed to the gas is increased by such means as rotating discs, or nests of sheets intermittently dipped into the liquid, or fixed sheets in separate liquor compartments which are alternately filled and emptied.—B. M. V.

Electrical precipitation of particles from gases. D. K. Morris and F. H. Haviland. E.P. 148,659, 15.5.19.

THE gas or fume passages are of such a form that the fume is caused to flow rapidly around an edge or edges at which there is a strong electric field. The momentum of the heavier dust or particles causes them to pass beyond the edges, into a region where the field is weaker, whence they descend by gravity.—B. N.

Electrical deposition of particles from gases. L. Lodge, Assr. to The Lodge Fume Co., Ltd. U.S.P. 1,345,790, 6.7.20. Appl., 10.5.20.

CHAMBERS for use in the electrical deposition of particles from gases, are provided with plates forming channels for the gases, discharge electrodes situated transversely and longitudinally between the plates, and deflectors for deflecting the gases toward the sides of the channels when passing longitudinally by the discharge electrodes.—B. N.

Tunnel oven or kilns. C. Whitfield. E.P. 150,482, 2.7.19.

A SEPARATE burner or combustion chamber is provided to which air under pressure and gas are admitted through separate jets and ports, so that a short intense flame is produced which afterwards passes to the heating chamber proper. The jet of pressure air may also induce a current of hot air from the interior of the oven to the burner chamber.—B. M. V.

Furnaces or kilns. R. J. Anderson. E.P. 150,762, 4.4.19.

A VERTICAL furnace for the smelting of ores or conversion of chemical compounds is surrounded at the bottom by combustion chambers into which fuel and air are injected in numerous fine streams which intersect at a large number of points. The fuel is completely burnt and the products pass under pressure into the charge in the furnace. Additional combustion chambers are mounted on the main combustion chambers, and in these fuel is burned to carbon monoxide, which is also injected into the charge to act as a reducing agent.—W. F. F.

Furnace walls. E. Bernitz. E.P. 151,031, 12.3.19. Addn. to 119,599.

FURNACE walls provided with air ducts as described in the original patent (J., 1918, 723 A) are constructed of special bricks, formed with their corners cut off, or with circular depressions in their ends or sides or with holes formed through them.—B. M. V.

Furnace. J. G. Aarts. U.S.P. 1,352,338, 7.9.20. Appl., 11.8.16.

A SHAFT furnace is provided with hollow walls composed of highly refractory bricks provided with a large number of narrow heating flues with transverse baffles. The flues of the several bricks are in communication to form continuous flues from the bottom to the top of the furnace.—W. F. F.

Extracting water from any material containing it. F. Merz. E.P. 150,786, 4.6.19.

A CURRENT of air or other gas is circulated over the material to be dried or concentrated and over a hygroscopic material. The hygroscopic material is regenerated by passing over it dried air which may also be heated if desired.—B. M. V.

Drying processes. T. Boberg, and Techno-Chemical Laboratories, Ltd. E.P. 150,807, 6.6.19.

IN the method of drying described in E.P. 150,068 (J., 1920, 682 A) the material from the first stage of drying is conveyed along, or dropped down, a conduit in the presence of a moving heated gas.—B. M. V.

Dryer. C. Slattery. U.S.P. 1,354,223, 28.9.20. Appl., 6.11.19.

A FRAME having vertical standards is provided with a series of supports one above the other on which trays may be supported. A conveyor is adapted to move up and down beside the supports and place the trays at any desired height.—W. F. F.

Dehydration; Process of —. A. W. Browne. U.S.P. 1,351,279, 28.9.20. Appl., 31.5.17.

THE material containing water is treated with a solution of a dehydrating agent in a non-aqueous solvent.—B. M. V.

Still. A. C. Jewell. E.P. 150,940, 3.2.20.

THE upper or main part of the still extends downwards and fits closely into a detachable bottom to below the liquor level; the top rim of the bottom is bent outwards and at the same height a groove is formed round the main part of the still, so that

packing can be caulked in. By this arrangement the bottom may be rapidly detached and replaced by another adapted for a different method of heating.—B. M. V.

Subdivision and treatment of fluids for various purposes. E. L. Pease. E.P. 151,038, 14.4.19.

STREAMS of fluids issue through holes or jets in the walls of a number of parallel pipes, and are guided in thin, flat streams by bars or sheets of metal or refractory material preferably arranged as a sort of lattice work between the pipes. The process is applicable to mixing coal dust or the like or combustible gases with air, to washing or cooling gases, or to effecting heat interchange between gases and/or liquids.—B. M. V.

Rectifier and condenser. H. E. Beyer and W. G. Hall, Assrs. to Ingersoll-Rand Co. U.S.P. 1,352,648, 14.9.20. Appl., 25.1.19.

THE apparatus consists of a vacuum pan over which a rectifier is supported by means of a conical sheet, while a crown sheet placed above the opening of the vacuum pan acts as a baffle to intercept cooling liquid. The casing of the rectifier supports a condenser, through which cooling liquid is circulated and in which baffles are fixed so as to form chambers for collecting the distillate. Means are provided for returning the liquid in the rectifier to the vacuum pan and for draining the upper baffle chamber and the casing of the condenser.—A. R. P.

Condensing apparatus. J. C. Morton. U.S.P. 1,354,122, 28.9.20. Appl., 4.4.18. Renewed 23.8.20.

A TUBULAR condenser is made with concentric tubes, the inner being circular in cross section and the outer oval.—W. F. F.

Centrifugal machine and process of disintegrating [atomising] material. R. P. Perry, Assr. to The Barrett Co. U.S.P. 1,352,623, 14.9.20. Appl., 1.11.17.

MOLTEN material is introduced axially into the top of a closed, rotating vessel in the form of an inverted frustum of a cone. The side walls are perforated so that the molten material is projected along different horizontal planes, the velocity in each plane being higher than that in the plane below.—W. F. F.

Crushing machine. W. Ellk, Assr. to J. Sinkovitz. U.S.P. 1,352,797, 14.9.20. Appl., 26.3.19.

A CRUSHING machine comprises a pair of superposed annular surfaces which are rotated in opposite directions about their common vertical axis. The crushed material is discharged into a casing having a hood above it through which the material is exhausted. Any particles falling back from the hood pass through a central opening in the upper grinding member on to the central part of the lower grinding member, where they are crushed by grinding balls rolling on a circular track.—W. F. F.

Extraction; Process of —. T. J. Fairley, Assr. to Southern Pine Products Co., Inc. U.S.P. 1,353,035, 14.9.20. Appl., 14.7.17.

THE material to be extracted is placed in a closed vessel completely filled with water at 180° F. (82° C.), and the pressure in the vessel is increased by the injection of steam to about 50–70 lb. per sq. in.—W. F. F.

Cooling and crystallising hot solutions; Apparatus for —. Maschinenbau A.-G. Balcke. G.P. 321,773, 10.9.18. Addn. to 319,968 (J., 1920, 563 A).

THE lower part of each conveyor band in the apparatus described in the chief patent is parallel to the inclined bottom of the vessel, and the bands are provided with scrapers, which remove the crystals from the bottom and convey them away from the vessel.—L. A. C.

Evaporating, cooling, concentrating, washing, and other purposes; Method of and means for effecting intimate contact between liquids and gases for —. H. E. La Bour. E.P. 125,380, 3.1.19. Conv., 8.1.18.

SEE U.S.P. 1,318,774 of 1919; J., 1919, 937 A.

Drying of solid moist substances; Apparatus for —. A/S. Myrens Verksted. E.P. 110,173, 20.3.20. Conv., 7.2.18. Addn. to 105,072 (J., 1918, 280 A).

SEE U.S.P. 1,317,501 of 1920; J., 1920, 619 A.

Separating mixed gases; Process of and apparatus for —. F. E. Norton, Assr. to Jefferies-Norton Corp. U.S.P. 1,351,056 9, 28.9.20. Appl., 31.8.15, 20.10.15, 26.1.18, 28.1.18. Renewed 29.7.12, 29.7.19, 14.7.20, 14.7.20.

SEE E.P. 106,676-7 of 1916; J., 1917, 859.

Furnace-front. C. Zulver and L. E. Smith. U.S.P. 1,351,429, 28.9.20. Appl., 11.8.19.

SEE E.P. 132,977 of 1919; J., 1919, 853 A.

IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

Coals; Determination of sulphur forms in —. A. R. Powell. J. Ind. Eng. Chem., 1920, 12, 887—890.

THE method described by Powell and Parr (J., 1920, 145 A) is trustworthy for the determination of the different forms of sulphur in coal, but for rapid routine work the following modifications are suggested. Sulphates may be extracted by boiling the coal for a few mins. with hydrochloric acid (sp. gr. 1.12) and the pyrites then extracted by shaking for 2 hrs. with dilute nitric acid. The difference between the total sulphur and the sum of the sulphate and pyrites sulphur gives the amount of organic sulphur. Since dilute nitric acid dissolves small quantities of the unstable phenol-soluble form of organic sulphur, it is preferable to calculate the pyrites sulphur from the pyrites iron content. In the case of semi-bituminous coals and the harder varieties of bituminous coals, the sample must be digested for a long time with concentrated nitric acid before the residue from the extraction becomes completely soluble in ammonia. The sodium peroxide used for the determination of sulphur in coal should have a low and constant sulphur content.—W. P. S.

Coal and its products; Determination of the specific gravity of —. H. J. Hailstone. Gas J., 1920, 131, 566.

THE specific gravity of small coal, pitch, etc., can be determined by flotation, using a solution of sodium thiosulphate of sp. gr. 1.350. For materials (e.g., pitch) of sp. gr. 1.2—1.35, a small pellet of the substance is dropped into 10 c.c. of the solution, and water is then added from a burette until the pellet is just suspended in the liquid, neither floating nor sinking, and the volume of water added is noted. From a curve, the sp. gr. is obtained. For materials of less than 1.2 sp. gr. the thiosulphate solution is run into 10 c.c. of water until the pellet just begins to rise.—A. G.

Fuels; Valuation of — on the basis of their flame temperature. W. Schwier. Stahl u. Eisen, 1920, 40, 1033—1037, 1108—1111, 1170—1177, 1236—1240.

CALORIFIC value alone is not a safe criterion of the value of fuel for metallurgical purposes. In high temperature operations, only that portion of the heat which is given up when the products of com-

bustion are cooled to the working temperature of the furnace is effectively used. Therefore the flame temperature is of equal importance. The calorific value is no criterion of flame temperature, and this is particularly shown in the case of gaseous fuels. Loss of heat by radiation increases with rise of temperature, but the possible efficiency of heating operations increases more rapidly. A safer criterion for the comparison of fuels, it is suggested, is a "figure of value" $H_{ut} \times 10^{-6}$, where H_u is the net calorific value and t the theoretical flame temperature. The method of calculating t is described, and data for specific heats and heat capacities are tabulated. The utilisation of the "figure of value" in the study of a number of metallurgical heating operations is illustrated. In a fuel gas, the usefulness of a component depends on the nature of the other components which accompany it; thus the utility of a given quantity of carbon monoxide differs in water gas and producer gas, a difference which is reflected by the $H_{ut} \times 10^{-6}$ figures. The relative efficiencies of the different modes of consuming 1 kg. of carbon can be compared by this criterion. In the comparison of fuel gases, it is necessary to take account of dust, water vapour, tar, and sensible heat. The tar has an $H_{ut} \times 10^{-6}$ figure greater than that of coal. Moisture, owing to high specific heat in addition to the latent heat, has a markedly depressing effect on flame temperature, and yet it usually does not appear in analyses.

—H. J. H.

Silica bricks in coke-oven construction. J. Enzenauer. Glückauf, 1920, 56, 785—793.

AN account is given of two batteries of ovens built, the one in 1915 of silica bricks (SiO_2 , 95%, CaO 2%) and the other one year later of ordinary coke-oven silicious bricks (SiO_2 , 80%, Al_2O_3 , 16%). The higher thermal conductivity of the silica brick was recorded in a shorter coking period, 21 hrs. as against 29 hrs., in the latter battery. In spite of initially unpromising texture and brittleness of the silica brick walls, they are now in the better condition of the two. The silicious bricks are more extensively corroded by salt and more worn by erosion. The walls of silica brick are less susceptible to fusion or deformation due to unskilled attention. The yield of by-products may possibly be adversely influenced in the silica brick construction, but this does not outweigh the advantages of increased output of good coke and of gas. The fact that higher temperatures may be maintained in flues of silica brick without damage to the walls may be a temptation to shorten the coking period at the expense of by-products, but the rise in heat consumption finally renders such practice uneconomical. Material and thermal balance sheets of the two batteries are given.

—H. J. H.

Coal; Swelling of — during coking. F. Korten. Stahl und Eisen, 1920, 40, 1105—1108.

THE tendency to swell during carbonisation possibly depends on the variation in temperature and mode of decomposition of the bituminous cementing material (cf. Fischer and Glud, J., 1920, 258 A). The author proposes a test for the swelling on coking:—100 g. of the coal is coked under standardised conditions in a cylindrical crucible of iron closed by a perforated piston. The latter supports a weighted lever fixed at one end so that the free end moves in accordance with the rise or fall of the piston. The free end is made to trace a magnified record of the movement of the piston, i.e., of the change of volume of the coal, upon a revolving drum covered with paper. The results agree with large-scale practice. A coal with 27% of volatile matter which showed a contraction of 4% was known to be easily coked and pushed from the oven. Another coal with 14% of volatile matter expanded nearly 6% during carbon-

isation and 3.5% at the end. In practice the coke from this coal was difficult to push from the ovens, and then only with damage to the walls.—H. J. H.

Oil-shale; Treatment of — and description of a specially high-grade shale. M. Dolch. Oesterr. Chem.-Zeit., 1920, 23, 122–125, 130–131.

AN Austrian shale of specially high quality is described. It has sp. gr. 0.8, is bright yellow in colour, and resembles earth saturated with natural petroleum. In laboratory trials it yielded 31.2% of shale oil on dry distillation, and 42.4% on distillation with superheated steam. This contrasts with 12–14% from Scottish shales or the torbanite of South Africa, 20% from Norfolk shale, and 35% reported from New South Wales. In the redistillation of the oil a loss of 18–19% occurred, due to decomposition with evolution of gas from 250° C. onwards. 2 g. of the crude oil heated slowly to 300° C. in an atmosphere of nitrogen yielded more than 3 c.c. of carbon dioxide. The improved quality and quantity of the distillate with the use of steam is explained by the prevention of local overheating and the partial decomposition described above. The crude oil yields no pitch, a result approached in the case of tar from certain Bohemian lignites. The nitrogen content of the shale varies from 0.5 to 2.0%. On dry distillation 100 kg. of shale yields 25 cub. m. of gas of 6260 cal. per cub. m. A quantity of light paraffins equal to 6% on the shale could be removed from the gas and still leave it with a calorific value of 4000 cal. per cub. m.

—C. I.

Hydrocarbons; Oxidation of — by oxygen. Oxidation of paraffin wax. C. Kelber. Ber., 1920, 53, 1567–1577.

THE residue obtained by the oxidation of paraffin wax (J., 1920, 220 A), amounting to 90–100% of the original substance, is a yellowish mass, iodine value, 1–2; saponification value, 250–300; acid value, about 200. When purified by washing with water and subsequently hydrolysed, it yields a highly complex mixture of fatty acids. The following have been definitely isolated: capric, myristic, palmitic, heptadecanoic, stearic, and arachidic acids, and an acid, $C_{16}H_{32}O_2$, which is isomeric with palmitic acid and possibly identical with Bergmann's isopalmitic acid. (Cf. J.C.S., Nov.)—H. W.

PATENTS.

Coal and other minerals; Apparatus for washing —. P. Habets and A. France. E.P. 138,905, 10.2.20. Conv., 12.8.13. Addn. to 22,655/12.

THE washing apparatus is of the type in which the coal or mineral is carried in a stream of water through a launder having a number of slots in its bottom through which streams of water are directed upwards, and through which the heavier material falls. The horizontal length of the slots may be modified by baffles moved horizontally by means of a screwed rod, and a vertical baffle may also be provided at the edge of the slot to intercept part of the heavier material and provide an artificial slope in the bottom of the launder. The upper part of each separating pocket, below the slot, may be trapezoidal, and the lower part rectangular in vertical cross-section.—W. F. F.

Coal; Treatment of — especially in the manufacture of fuel briquettes. C. H. Smith. E.P. 125,379, 3.4.19. Conv., 3.4.18.

COAL is supplied from a storage bin to a crusher, and thence to travelling trucks by which it is conveyed to carbonising retorts, preferably constructed as described in E.P. 123,739 (J., 1920, 650A). Bituminous coal containing 36% volatile matter and 55% fixed carbon is used, and is carbonised at 850°–1200° F. (455°–650° C.), leaving a spongy residue containing 5–15% of volatile matter, which

is conveyed to a cooler to which water is supplied. The material is then mixed with pitch, crushed, and treated with steam to convert it into a plastic mass. The mass is mixed with water if necessary, moulded into briquettes, and transferred to an inclined furnace heated by combustion flues in the walls, the hot gases afterwards passing downwards through a regenerator to pre-heat the air supply. The furnace is heated to 1400°–1850° F. (760°–1010° C.), and the briquettes are finally cooled by a spray of water.—W. F. F.

Briquette dryer. R. S. Plummer, Assr. to American Briquet Co. U.S.P. 1,352,377, 7.9.20. Appl., 11.9.19.

THE briquettes are carried by a travelling conveyor which divides the drying tunnel into upper and lower compartments. Air is forced into the lower compartment at one end by a fan, and discharged from the upper compartment at the other end. A series of intermediate fans draw air from points along the length of the upper compartment and deliver it to the lower compartment, and means are provided for heating the air prior to its introduction into the first of the series of fans.—W. F. F.

Fuel; Manufacture of a solid —. Elektrizitätswerk Lonza. E.P. 144,589, 9.3.20. Conv., 16.9.19.

METALDEHYDE compressed into a coherent mass, with or without the addition of colophony, chloroform, or the like as binding agent, is used as a fuel.

Peat mass; Treatment of — and production of turf or peat briquettes. E. P. T. R. Ulmann. E.P. 149,869, 6.11.19.

THE peat mass is thoroughly disintegrated and mixed with water to form a homogeneous peat-sludge, in a rotary or turbine pump, which also produces the pressure necessary to force the mass through a pipe line directly to the drying place, where it is spread out in a thin layer and allowed to dry, being afterwards cut up into briquettes. The pipe line is composed of detachable sections, which can be removed from the line as the discharge advances over one area and can be rejoined as another area is spread with the peat mass.—A. G.

Burning pulverulent or liquid fuel in furnaces; Method of and means for —. J. E. Muhlfield. E.P. 150,230, 24.2.19. (See also E.P. 112,155; J., 1918, 83A.)

A MIXTURE of fuel and air is introduced in one or more streams into a preliminary combustion chamber or chambers leading in a direct line to the main combustion chamber but located outside the furnace and constituting a zone of smaller capacity than the main combustion chamber.—A. G.

Fuel; Process of and apparatus for burning —. E. C. R. Marks. From Schutz Hawley Co. E.P. 150,395, 27.5.19.

FUEL, e.g., coarsely crushed or pulverised coal, mixed with air, is fed tangentially into one end of a cylindrical furnace having refractory walls, and passes in a helical path to the other end of the furnace. The stream of fuel then returns axially through the furnace within the annular current. Incombustible solid matter may be withdrawn during the helical movement of the fuel. The two ends of the annular space may be interconnected to equalise the pressures.—W. F. F.

Fuel; Apparatus for the combustion of —. R. J. Anderson. E.P. 150,761, 4.4.19.

FUEL, which may be solid, liquid, or gaseous, is projected tangentially into an annular combustion chamber surrounding a heating chamber, and the air for combustion is supplied to a number of perforated pipes of refractory material which project radially into the combustion chamber. The air is thus projected transversely across the fuel jet and

an effective mixing is obtained. The air nozzles may be arranged in rows in stepped formation.

—W. F. F.

Combustible material contained in the ashes from industrial furnaces and steam boilers; Process for the recovery of —. A. Andre. E.P. 151,192, 30.12.19.

THE ashes are fed into an inclined conduit having a gap in the lower wall midway of its length. A current of air is delivered by a blower horizontally through the gap into the conduit and separates the lighter combustible material from the heavier slag. The former passes into the lower part of the inclined conduit and the latter falls through the gap into a vertical discharge conduit.—W. F. F.

[Gas] *retorts or ovens; Introduction of steam into vertical* —. Woodall, Duckham, and Jones, Ltd., and A. McD. Duckham. E.P. 150,589, 18.12.19.

STEAM is superheated above 700° F. (370° C.) by passing it through tubes built in or about the waste gas flues of a vertical externally-heated retort, and then discharged through nozzles into the lower part of the retort.—W. F. F.

Coking coal. A. Roberts, Assr. to American Coke and Chemical Co. U.S.P. 1,352,696, 11.9.20. Appl., 1.12.19.

METALLURGICAL coke is obtained from coals in which the disposable hydrogen does not exceed 5% of the disposable oxygen, by heating the retort very rapidly when first charged. The temperature of decomposition of the resinous constituents is reached before excessive volatilisation has taken place and a sufficient resinous residue is left to bind the mass.—C. I.

Gas producers. A. L. Galusha. E.P. 129,971, 24.6.19. Conv., 15.7.18.

A PRODUCER is combined with a gas engine, and the steam requisite for the producer is generated by introducing water into the exhaust pipe of the engine, the pressure being regulated by an adjustable deflector extending partially across the exhaust pipe. The mixture of steam and combustion products is deflected into a dome, and forced by its own pressure into the steam-supply pipe of the producer. This extends into the larger air inlet pipe, and draws in air by injector action. Excess of water in the exhaust pipe may be used to keep the pipe free from salts, sludge, etc.—A. G.

Gas producers. G. H. Bentley and E. G. Appleby. E.P. 119,759, 16.5.19.

THE shell of the producer is mounted eccentrically and rotates around the grate, which is mounted upon a frame, together with a trough forming a water-seal. The frame is supported by columns, and the shell rotates upon rollers on the frame. The ashes are thereby discharged without the use of a tray and scrapers.—A. G.

Gas producers. H. M. N. Milton. E.P. 149,790, 26.5.19.

A CHAMBER, divided by a water-seal into compartments, is interposed between the producer proper and the gas holder. Each of the compartments receives gas primarily from a portion of the producer, and each can be separated from the general gas circulation. The grate of the producer is divided by a diaphragm dipping into a water-seal. A portion of the producer may thus be isolated for cleaning etc. without cutting off the gas supply.—A. G.

Producer gas; Generation and utilisation of —. A. H. Lynn and N. E. Rambush. E.P. 150,787, 4.6.19.

THE waste heat of a gas-fired furnace is employed

to raise steam for the producer blast by the use of vessels resembling gas washers (E.P. 11,452/12 and 741/13; J., 1912, 1167; 1913, 1117). In one such vessel a shower of water is heated by the hot burnt gas, and in another the heated water saturates and warms the air blast to the producer. Between these two vessels a third vessel may be interposed, in which the hot producer gas neutralises the acidity of the heated water and further heats it.—C. I.

Producer gas plants and internal combustion engines supplied therefrom. D. J. Smith. E.P. 151,087, 11.6.19.

IS a gas producer plant, combined with an internal combustion engine, the water for use in the producer is preheated by passing it successively through the engine jacket, through a tubular water heater heated by the engine exhaust gases, and through a heater heated by the producer gas on its way to the engine, and then passes into a boiler mounted on the producer. The waste heat of the plant is thereby fully utilised.—W. F. F.

Gas generator; Pulverised-fuel —. N. F. X. Beaugard. U.S.P. 1,351,460, 31.8.20. Appl., 21.5.17.

POWDERED fuel mixed with air is subjected to intense heat, then mixed with an additional supply of air and supplied to a furnace chamber for combustion. Air is supplied to the inlet end of a conduit, together with the powdered fuel, and a supplementary supply of air is added at the outlet end of the conduit, the intermediate part of which is heated.—A. G.

Carbon and gaseous fuel; (a) Process and apparatus and (b) process for producing —. J. R. Rose. U.S.P. (a) 1,352,085, and (b) 1,352,086, 7.9.20. Appl., (a) 8.11.18, (b) 26.12.18.

(a) AN "acriform hydrocarbon fluid" is introduced into a generator in which it is decomposed into solid carbon and a gas containing a preponderating amount of hydrogen, by the heat of an electric arc. (b) A mixture of hydrocarbon fluid with insufficient oxygen for its complete combustion is subjected to "discontinuous resistance material in an electric circuit," e.g., coarse material packed between electrodes, whereby carbon and a gas rich in hydrogen are produced.—J. H. L.

Gas; Method of generating —. H. Batchelor. U.S.P. 1,352,241, 7.9.20. Appl., 8.2.17. Renewed 13.1.20.

TWO separate masses of porous refractory material are alternately heated and sprayed with a homogeneous mixture of hydrocarbon and water. The gas generated is passed through a third mass of refractory material.—W. F. F.

Cyanogen [compounds]; Recovery of — from coal-gas. J. J. Hood. E.P. 141,398, 14.3.19.

AFTER removing hydrogen sulphide and carbonic acid from the gas in a lime purifier, the gas is treated, in the same purifier, with slaked lime impregnated with a solution of an iron salt, such as ferrous sulphate. Ferrous sulphate may be added, for example, to the lime in a tier of the purifier near the exit. The cyanogen compounds in the gas are fixed as calcium ferrocyanide, and by leaching or agitating the spent purifying material with dilute alkali, a solution of alkali ferrocyanide is obtained.

Fuel; Aeroplane motor —. U.S. Industrial Alcohol Co., Assces. of A. A. Backhaus. E.P. 128,917, 16.6.19. Conv., 25.6.18.

THE fuel is composed of 40 to 60% of alcohol (98%), 25% to 35% of benzol, and 30% to 50% of gasoline or naphtha.—A. G.

Hydrocarbon liquids; Production of — from carbonaceous material and apparatus therefor. F. P. A. Rousseau. E.P. 132,490, 4.6.19. Conv., 9.9.18.

CARBONACEOUS material such as coal, peat, bitumen, resins, pitch, oils, wood, or the like, in a finely divided condition, is distilled under pressure in an autoclave heated by external coils. The material is fed into the autoclave through a long shaft fitted with two valves between which the charge is isolated to prevent the entry of air, and, in the case of solid material, falls on to a hearth situated near the top of the autoclave. Several of the autoclaves are arranged around a common vessel in the form of an inverted cone heated at the top by a coil and connected with each autoclave by a slide-valve. The distillation products collect in this vessel and pass through a tube at the lower end into a condenser. In treating material with a relatively high carbon content, hydrogen may be admitted into the autoclaves.—L. A. C.

Petroleum; Process of cracking —. L. P. Lowe and F. C. Ruff, Assrs. to By-Products Manufacturing Co. U.S.P. 1,351,859, 7.9.20. Appl., 15.1.17.

A CRACKING chamber filled with openwork material is heated by means of products of combustion to a temperature above that at which petroleum is cracked to produce carbon and condensable hydrocarbons but below that at which petroleum is converted into permanent gas. Petroleum is then passed through the chamber, and subsequently, after stopping the supply of petroleum, the carbon deposited on the surface of the openwork material is burnt to assist in again attaining the requisite temperature to repeat the process.—L. A. C.

Electrochemical treatment of [hydrocarbon] vapours; Electrical apparatus for the —. L. B. Cherry. U.S.P. 1,345,431, 6.7.20. Appl., 9.10.17.

LIQUID hydrocarbons are vaporised in a still, and the vapours on their way to a condenser pass through a conduit consisting of a series of elongated, metallic, tubular units, united by cross connexions to form a continuous treating chamber. The wall of the chamber is maintained approximately at a red heat by means of solenoids, which are arranged longitudinally outside the units but insulated therefrom, the units being heated by induction. The solenoids are composed of a good electrical conductor, through which an alternating current is passed, and means are arranged for controlling the current to vary the temperature of the units. The vaporised products are simultaneously subjected, whilst passing through the heated chamber, to a high-frequency electric current discharge.—B. N.

Hydrocarbons; Apparatus for treatment of —. W. F. Rittman. U.S.P. (A) 1,352,916 and (B) 1,352,917, 11.9.20. Appl., (A) 3.8. and (B) 7.11.17.

(A) A TUBE passes upwards through the lower end of a vertical retort, and the portion within the retort is provided with openings for the passage of liquids. Hydrocarbons are passed into the lower end of the annular space between the tube and the retort, which is maintained at a suitable temperature for vaporising and cracking the hydrocarbons, and the products pass into the central tube, or the hydrocarbons are passed into the central tube and the products pass into the annular space. (B) Oil is fed into an auxiliary retort, so arranged that it receives heat from a main vertical retort arranged in a furnace. A constant quantity of the oil is maintained in the auxiliary retort, and the overflow passes into the main retort.—L. A. C.

Mineral oils; Process for treating —. W. Johnson. U.S.P. 1,354,257, 28.9.20. Appl., 25.4.16.

HEAVY mineral oil vapour is superheated and cracked by passage through an electric arc.

—L. A. C.

Paraffin; Process and apparatus for purifying crude — crystallising in scales, by sweating. E. Bartusch. G.P. 307,625, 29.8.17.

THE adhering oils, impurities, etc., are removed along the shortest possible horizontal path, through perforated surfaces which surround the material laterally, by increasing the pressure in a direction at right angles to that of the expressed oil and by raising the temperature at the same time.—J. H. L.

Hydrocarbon mixtures; Separation of solidified constituents, e.g., paraffin, from the sulphurous acid solution containing unsaturated hydrocarbons, in the treatment of — with liquid sulphur dioxide. Allg. Ges. für Chem. Ind. G.P. 322,754, 17.3.17.

AFTER the treatment of the hydrocarbon mixture with liquid sulphur dioxide the main portion of the sulphur dioxide extract, containing unsaturated hydrocarbons, is drawn off, and the residue, consisting of saturated hydrocarbons, with solidified hydrocarbons and some included residual extract, is heated in a special vessel until this residual extract has formed a distinct layer, whereupon it is separated and united with the main portion of extract.—J. H. L.

Subdivision and treatment of fluids. E.P. 151,038. See I.

Separating hydrogen sulphide from gases. G.P. 322,938. See VII.

Paint vehicles. G.P. 321,828. See XIII.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Acetyl content of wood. Schwalbe and Becker. See V.

PATENT.

Glass surfaces [electric lamp bulbs]; Substances for coating —. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 150,598, 8.1.20.

THE glass surface is sand-blasted and coated with a mixture of, e.g., 5 pts. of hydrous magnesium silicate (French chalk), 5 pts. of zinc oxide, 15 pts. of sodium silicate (sp. gr. 1.375), and 8 pts. of water. When the coating is dry a thin layer of varnish is applied. The zinc oxide may be replaced by a coloured material, such as burnt sienna.—L. A. C.

III.—TAR AND TAR PRODUCTS.

Thionaphthene in coal tar. R. Weissgerber and O. Kruber. Ber., 1920, 53, 1551—1565.

CRUDE naphthalene is sulphonated with a greatly deficient amount of sulphuric acid at 90°—100° C., and the sulphonated product is decomposed by steam in the presence of a little sulphuric acid at 145° C.; the distillate has m.p. 73° C. and contains 2.3% S. Repetition of the process gives a material containing about 5—6% S, which can be increased to 12—14% by freezing out the naphthalene. The product obtained in this manner is dissolved in glacial acetic acid and treated with hydrogen peroxide when thio-

naphthene-S-dioxide, m.p. 142° C., is obtained, identical with the synthetic product, thus giving the first proof of the presence of thionaphthene in coal tar. Thionaphthene can be isolated from enriched naphthalene by Friedländer's mercuric acetate method, but it is essential to guard against undue rise in temperature during the formation of the additive compound; this can be easily accomplished by operating with methyl alcoholic solutions. By a similar process of partial sulphonation, followed by the use of mercuric acetate, thionaphthene has been isolated from technical "pure" naphthalene (containing 0.3–0.4 S), about 2 g. of the substance being obtained from 80 kg. naphthalene. For the industrial preparation of thionaphthene from crude naphthalene better results are obtained by the use of sodamide or preferably sodium and ammonia than with sodium; reaction is complete at 120° C.; practically the whole of the thionaphthene is regenerated when the sodium compound is treated with water; the process is applicable to "enriched" naphthalene. Sodium-thionaphthene appears to consist of about two-thirds of the monosodium and one-third of the disodium derivative. It is converted by carbon dioxide in the presence of an inert solvent into a mixture of thionaphthene-2-monocarboxylic and thionaphthene-2,3-dicarboxylic acids, which can be separated by taking advantage of their widely differing acidities or by fractional distillation of their esters. The dicarboxylic acid is readily transformed into the corresponding imide, m.p. 236° – 237° C., which when degraded by Hofmann's method yields exclusively 3-hydroxythionaphthene, thus affording a ready method of converting thionaphthene into Thioindigo Red. (Cf. J.C.S., Nov.)—H. W.

Diphenylene sulphide in coal tar. O. Kruber. Ber., 1920, 53, 1566–1567.

The presence of diphenylene sulphide in crude phenanthrene (containing about 1% S) has been established by oxidising the latter in hot glacial acetic acid with hydrogen peroxide, whereby diphenylene-sulphone, m.p. 229° – 230° C., is obtained.—H. W.

Aniline; Physical constants of — C. J. Knowles. J. Ind. Eng. Chem., 1920, 12, 881–883.

Pure aniline has the following physical characters:—Freezing p., -6.21° C.; b. p., 181.32° – 181.39° at 760 mm.; sp. gr. at $15^{\circ}/15^{\circ}$ C., 1.0268; $n_D^{20}=1.5850$. The freezing point appears to be the best criterion of the purity of aniline; the percentage (X) of aniline in a sample may be obtained from the formula $X=108.79+1.41t$, where t is the observed freezing point.—W. P. S.

Phenols; Conductometric titration of — I. M. Kolthoff. Z. anorg. Chem., 1920, 112, 187–195.

PHENOL and the cresols can be accurately titrated in $N/10$ — $N/100$ solution with alkali by the conductometric method. The hydroxy group in vanillin, sodium phenolsulphonic acid, salol, and methyl salicylate can be similarly titrated. Salicylic acid behaves as a monobasic acid, the hydroxy group having no acidic character, but *p*-hydroxybenzoic acid behaves as a dibasic acid. The hydroxy group in thymol and β -naphthol can also be titrated conductometrically. Catechol behaves as a monobasic acid, the second hydroxy group having no acidic properties, whilst resorcinol and quinol are dibasic. Resorcinol is best titrated with barium hydroxide; when sodium hydroxide is used the breaks in the conductivity curve are not distinct. Pyrogallol and phloroglucinol function as dibasic acids and gallic acid as a tribasic acid.—E. H. R.

Humic acids; Synthesis of — [from phenols]. W. Eller and K. Koch. Ber., 1920, 53, 1469–1476.

THE oxidation of phenols in alkaline solution leads to the formation of substances which have all the properties of natural humic acids. Thus solutions of catechol, quinol, or *p*-benzoquinone when exposed to air or treated with solid potassium persulphate give a product of the composition, $C_{11}H_6O_5$, whilst phenol gives a similar humic acid which is distinguished, however, by its greater solubility in alcohol. In general, only those phenols give humic acids which are capable of yielding quinonoid intermediate products. It would thus appear that there are a number of natural humic acids and that these substances when pure are free from nitrogen, and their acidic properties are probably due to the presence of an unchanged phenolic hydroxy-group. (Cf. J.C.S., Nov.)—H. W.

Sp. gr. of coal etc. Hailstone. See IIA.

PATENTS.

Benzene; Hydrogenation of — The Dayton Metal Products Co., Assocs. of T. Midgley, jun. E.P. 133,666, 7.5.19. Conv., 5.10.18.

A MIXTURE of benzene vapour and hydrogen is passed at a pressure of about 300 lb. per sq. in. over a catalyst, e.g., nickel oxide, maintained at about 300° C. By adjusting the rate of feeding the benzene into the apparatus, hydrogenation may be effected to yield either cyclohexane or a mixture of cyclohexane and benzene in any desired proportion, such as the eutectic mixture claimed in E.P. 133,667.—L. A. C.

Hydrogen for catalytic reactions. G.P. 301,361. See VII.

IV.—COLOURING MATTERS AND DYES.

Azo dyestuffs; Obtaining — by coupling diazo compounds with certain oxazine and chrome mordant dyes. E. Justin-Mueller. Rev. Gén. Mat. Col., 1920, 24, 130.

DIAZOTISED α -naphthylamine, ethoxybenzidine, or paranitraniline may be coupled with such oxazine dyes as Gallocyanine, Modern Violet, and phenocyanine and with azo chrome mordant dyes (diazotised benzidine sulphonatedisulphonate may also be used) to produce dyestuffs sparingly soluble in water but soluble in sodium bisulphite, and suitable for printing. A suitable printing paste is made by allowing 3 pts. of dyestuff with 2 pts. of sodium bisulphite of 38° B. (sp. gr. 1.36) to stand overnight, adding 2 pts. of glycerin and 24 pts. of gum solution, warming, cooling, and adding 6 pts. of chromium acetate of 20° B. (sp. gr. 1.16). The printed cloth is steamed for 1 hr., soaped at 80° C., and dried. The moss green to marine blue effects so obtained are fast to washing, soaping, air, and light.—A. J. H.

Yellow AB and Yellow OB in mixtures; Detection and determination of — W. E. Mathewson. J. Ind. Eng. Chem., 1920, 12, 883–887.

THE oil-soluble dyes Yellow AB (benzenazo- β -naphthylamine) and Yellow OB (*o*-toluenazo- β -naphthylamine) may be separated from Butter Yellow and Sudan I by means of petroleum spirit and sulphuric acid of different concentrations. When a solution of 0.011 g. of Yellow AB in 40 c.c. of petroleum spirit is shaken with 13N sulphuric acid and the acid extract shaken in turn with fresh portions of petroleum spirit, 92% of the dye will be found in the acid solution. Under similar conditions only 10% of Yellow OB can be recovered from

the acid solution. With 16N sulphuric acid 99% of Yellow OB passes into the acid solution, whilst all the Sudan I which may be present remains in the petroleum spirit. Butter Yellow is removed entirely from its solution in petroleum spirit by 10N sulphuric acid, but Yellow AB remains in the petroleum spirit layer. Spectrophotometric data are given which have been applied to the quantitative determination of the four dyes mentioned.

—W. P. S.

Thionaphthene. Weissgerber and Kruber. See III.

PATENT.

Dyestuffs; Manufacture of indigoid —. A. Jolles. G.P. 305,559, 17.10.15. Conv., 7.10.14. Addn. to 305,558 (J., 1919, 894A).

In the process described in the chief patent the aromatic hydroxy compound is replaced by 3-hydroxy-(1)-thionaphthene or its 2-carboxylic acid, or derivatives of these. An example is given of the preparation of 2-thionaphthene-2-indole-indigo from indoxyl acid and 3-hydroxy-(1)-thionaphthene.—L. A. C.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cellulose; Preparation and properties of a highly-oxidised —. E. Knecht and L. Thompson. J. Soc. Dyers and Col., 1920, 36, 251—255.

PREVIOUS methods for preparing oxycellulose are open to criticism, since they take no account of its decomposition by alkalis into non-reducing substances and its hydrolysis by acids. An oxycellulose is obtained by treating filter paper with an acid solution of potassium permanganate (either 1 or 2 atoms of oxygen to 1 mol. of cellulose, $C_6H_{10}O_5$), washing the brown powder so formed, decolorising with hydrogen peroxide, and drying at a temperature not exceeding 40° C. This oxycellulose has strong reducing power, especially in the presence of alkalis, and readily reduces Methylene Blue, Safranin, Rosinduline, Indigo, and Indanthrene in the presence of caustic soda. Thus the formation of oxycellulose may account for the well-known bleeding of Indanthrene and vat colours during kier boiling. It reduces solutions of silver, cupric, and ferric salts. With ammonia and sodium bisulphite it forms unstable compounds. A strong yellow colour develops with caustic soda, and on neutralising the solution an acid cellulose is precipitated which has only slight reducing power. It is not possible to estimate oxycellulose by means of its reaction with *p*-nitrophenylhydrazine, since only adsorption compounds are formed. The new oxycellulose had a copper number of 14.2 as compared with 7.6 for Nastjokoff's bleaching powder oxycellulose. (Cf. J.C.S., Nov.)—A. J. H.

Cellulosic substances; Accelerated method of estimating the reducing values of —. E. Knecht and L. Thompson. J. Soc. Dyers and Col., 1920, 36, 255—257.

THE Schwalbe method for determining the copper number of oxycelluloses is modified by dissolving the precipitated cuprous oxide in a ferric salt solution and estimating the ferrous salt so produced with permanganate. A confirmatory value is obtained by estimating the unreduced copper in the filtrate with titanous chloride, and the difference between the two values is equal to the copper hydrate number (unreduced copper absorbed by all celluloses). The two methods give nearly identical results, which are slightly lower than that obtained by the original Schwalbe method. The possible effect which the alkalinity of Fehling's solution may have on oxy-

cellulose can be avoided by heating the oxycellulose with a solution of cupric thiocyanate containing sodium carbonate, treating the precipitate of cuprous thiocyanate with warm caustic soda, and estimating the cuprous oxide so formed with a solution of a ferric salt and potassium permanganate. The method is less liable to error, but the copper number so obtained is slightly high. The copper number was shown to be a reliable criterion of the reducing power of cellulose, by heating oxycellulose with caustic soda and Rosinduline and then titrating with titanous chloride.—A. J. H.

Cellulose in woods; Determination of —. S. A. Mahood. J. Ind. Eng. Chem., 1920, 12, 873—875.

SAMPLES composed of particles of a uniform size are essential if comparable results are to be obtained in the determination of cellulose in woods, and it is recommended that the material (sawdust) used for the determination should pass an 80-mesh sieve and be retained by a 100-mesh sieve. The most satisfactory material for analysis is obtained by sawing the wood and then grinding the sawdust. Sieber and Walter's modification of the chlorination process, in which the material is allowed to remain in the crucible throughout the treatment with chlorine and sodium sulphite (J., 1913, 974), gives a lower yield of cellulose than does the original method of Cross and Bevan, probably because of the higher temperature during chlorination. The modification proposed by Johnsen and Hovey, comprising preliminary hydrolysis with acetic acid in glycerin at 136°—140° C. (J., 1918, 132T) appears to be untrustworthy, since the cellulose is attacked as well as the hemicelluloses and furfural-yielding substances.—W. P. S.

Wood and lignin; Acetyl content of —. C. G. Schwalbe and E. Becker. Z. angew. Chem., 1920, 33, 225—226.

THE statements by Pringsheim and Magnus (cf. J., 1919, 714A; 1920, 263A) that the greater quantity of the acetic acid obtained from wood products is derived from the lignin are controverted. Probably only one-third of the acetic acid has its source in this substance.—W. P. S.

Lignin. III. Preparation of a tannic acid from lignosulphonic acids. M. Hönig and W. Fuchs. Monatsh. Chem., 1920, 41, 215—222. (Cf. J., 1920, 330A.)

THE barium salts of the three fractions of the lignosulphonic acids are converted by boiling aqueous barium hydroxide solution into different insoluble compounds, which have not been identified, and a single soluble salt, $C_{12}H_{10}O_{10}S_2Ba$, the acid corresponding to which has the character of a tannic acid of the catechu group with properties which are somewhat modified by the sulphur content; when fused with potassium hydroxide, it gives protocatechuic acid in good yield. (Cf. J.C.S., Nov.)—H. W.

PATENTS.

Retting flax; Process for —. L. A. Johnson. E. P. 151,143, 20.8.19.

WHITE and silky fibres are produced when flax, previous to retting, is steeped for successive periods in a solution containing 2% (on the weight of flax) of natural Magadi soda, which contains 43.55% of sodium carbonate and 40.41% of sodium bicarbonate, whereby gum and resin compounds unfavourable to the retting bacteria are removed. It is advantageous to add 0.5% (on the weight of flax) of Magadi soda to the retting liquors, which should be maintained at 80°—95° F. (25°—35° C.), and periodically aerated.—A. J. H.

Cellulose solutions; Production of artificial fibres from —. K. Schreiner. G. P. 322,538, 25.12.17.

The method of producing fine threads by drawing out thick threads spun from wide orifices, is applied to cuprammonium cellulose solutions with the aid of slowly acting gaseous precipitants. After use the gases are passed into a well-cooled tubular vessel in which the moisture is deposited and the greater part of the ammonia is absorbed; after being moistened and reheated the gases are used again to extract ammonia from the threads. —A. J. H.

Nitrocellulose film of low inflammability. J. G. Jarvis. U.S.P. 1,351,401, 28.9.20. Appl. 13.3.19

FILMS of low inflammability are made from a solution of nitrocellulose and acetylcellulose, to which are added an acetone-collodion and chloral hydrate together with cinchonine.—A. J. H.

Cellulose ethers; Process for increasing the softness and flexibility of articles made from —. Farbenfabr. vorm. F. Bayer und Co. G.P. 322,619, 20.3.17.

DIETHYL resorcinoldicarbonate, $C_6H_4(O.CO_2.C_2H_5)_2$, b.p. 298°—302° C., is used. Ethylcellulose can be worked with 50% of its weight of this substance without crystallisation occurring.—A. J. H.

Adhesive for joining articles made of cellulose derivatives to the same or other materials. Farbenfabr. vorm. F. Bayer und Co. G.P. 322,613, 21.1.17.

ESTERS or ethers of phenols or naphthols are employed for the purpose, e.g., β -naphthol anyl ether may be used as an adhesive for ethylcellulose.

—L. A. C.

Digesters and the like used in paper making and the like; Stationary —. S. Milne. E. P. 150,782, 4.6.19.

A VERTICAL cylindrical digester has a conical lower portion within which are perforated plates for supporting the grass or like material under treatment. A valve at the lowest part of the digester has a hollow spindle connected at the upper end with a perforated pipe passing into the lower part of the digester. During digestion the liquor drains through the perforated plates, passes out from the space between the plates and the outer walls, and is returned to the top of the apparatus by a steam injector or pump; when the process is complete the valve at the bottom is opened and the contents are washed out by water, spent liquor, steam, or air, and forced under pressure into the digester through the valve spindle and perforated pipe.—L. A. C.

Cellulose waste liquor; Manufacture of a material resembling dextrin from —. M. Clauss. G.P. 322,688, 15.2.18.

CELLULOSE waste liquor is improved both in adhesive properties and in colour by treatment below 100° C. with acids, sulphates, and a sulphonylate. —L. A. C.

Waterproof paper or felted sheets; Manufacture of —. L. Kirschbraun. E.P. 151,029, 6.3.19.

SEE U.S.P. 1,302,810 of 1919; J., 1919, 491A.

Artificial silk; Apparatus employed in manufacture of —. J. Clayton. E.P. 150,889, 12.8.19.

Paper making. Great Northern Paper Co., Assees. of C. E. Pope. E.P. 137,845, 13.1.20. Conv., 25.10.18.

Paper-stock; Separating wax-like material from —. W. H. Smith. U.S.P. 1,352,553, 14.9.20. Appl. 21.11.16. Renewed 23.1.20.

Camphor recovery. E.P. 150,654. See XX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Vat dyes; Method for printing colour reserves under sulphur colours with —. L. Diserens. Rev. Gen. Mat. Col., 1920, 24, 113—115.

CLOTH is printed with a paste containing per 1000 pts., 150 pts. of Ciba Blue 2B 20% (or other vat dye), 250 pts. of hydrosulphite-sulphoxylate 35%, 250 pts. of British gum, 250 pts. of zinc chloride, and 100 pts. of caustic soda of 38° B. (sp. gr. 1.36), dried, twice steamed in a Mather-Platt for 4 mins., dyed in a bath containing per 1000 pts., 40 pts. of Immedial Carbon BN, 40 pts. of sodium sulphide, 10 pts. of caustic soda of 38° B. (sp. gr. 1.36), and 40 pts. of castor oil soap, then immediately washed, soured, washed, and soaped. Heavy ground shades may thus be obtained. It is important that the printing paste contain the dyestuff in the un-reduced form, and in suitable cases zinc sulphonylate may be used in place of the two separate components; the alkali employed may then be caustic soda, borax, or sodium acetate. Sulphur dyes may be used in the reserve paste if they be only slightly sensitive to zinc chloride. Less satisfactory results are obtained by the use of iron sulphate instead of zinc chloride, but the dyestuff in its reduced form may then be present in the printing paste.

—A. J. H.

Printing resists under formaldehyde-sulphoxylate discharges; Method for —. E. Justin-Mueller. Rev. Gen. Mat. Col., 1920, 24, 129.

PROVIDED that no caustic soda be present, sodium, potassium, or ammonium ferrieyanide is a satisfactory resist. Dyed cloth is printed with a paste containing 200—300 g. of potassium ferrieyanide, 600 g. of water, and 400 g. of soluble starch, dried, printed with a discharge paste containing hyal-dite C.W. extra, dried, steamed in the Mather-Platt, and washed.—A. J. H.

Basic colours; Production of lakes of — with zinc ferrieyanide. E. Justin-Mueller. Rev. Gen. Mat. Col., 1920, 24, 129—130.

COLOUR lakes suitable for printing on paper and textiles are obtained by mixing solutions of a basic dye (1 pt.) and zinc sulphate (2 pts.), and then adding a solution of sodium or potassium ferrieyanide (2 pts.).—A. J. H.

Azo dyestuffs. Justin-Mueller. See IV.

Ammonia in Turkey-red oil etc. Welwart. See XII.

PATENTS.

Printing [half discharge effects on] cotton fabrics. The Calico Printers' Assoc., Ltd., and W. Rouse. E.P. 151,056, 5.6.19.

HALF discharge effects are produced on unprepared fabric or on fabric prepared with Turkey-red oil, β -naphthol, or chlorate mixtures, by printing with a paste containing alkali sulphites with or without the addition of metallic mordants, slop padding with a metallic mordant, drying, and ageing or steaming to fix the mordant. The fabric is then either dunged or otherwise prepared for dyeing and dyed with alizarin or other mordant dyestuff, or tanned and dyed with basic colours. A suitable half discharge paste is made by adding 8 galls. of gum water, 1 galls. of aluminium acetate of 14° Tw. (sp. gr. 1.07), and $\frac{1}{2}$ gall. of turpentine to 4 galls. of a solution containing $\frac{1}{2}$ gall. of caustic soda of 70° Tw. (sp. gr. 1.35) and $\frac{1}{2}$ gall. of sodium bisulphite of 70° Tw. per 2 $\frac{1}{2}$ galls. The aluminium acetate may be replaced by chromium acetate of 40° Tw. (sp. gr. 1.20). If alizarin be used, red and pink effects are obtained.—A. J. H.

Cotton goods; Process for bowking — in one operation. W. Mathesius and M. Freiberger. G.P. 322,992, 15.7.16.

THE goods, which may have previously been steamed, are steeped in a solution of purified used liquor, or a mixture of the same with fresh liquor, containing about 80% of alkali hydroxide, and then bowked in a fresh solution (3 l. per 1 kg. of cotton) containing sodium hydroxide equal to 2.25–3% of the weight of the goods; the liquor is subsequently displaced by a weak alkaline solution, and the goods finally washed with hot and cold water.—L. A. C.

Scouring, dyeing, or otherwise treating with liquor, wool, yarn, or other fibrous material; Apparatus for —. H. Walker. U.S.P. 1,354,081, 28.9.20. Appl., S.12.17.

SEE E.P. 116,129 of 1917; J., 1918, 462 A.

Jiggers for use in dyeing and other operations [; Brake device for —]. S. Rawlinson, and Mather and Platt, Ltd. E.P. 150,750, 30.6.17.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Electrical precipitation of [sulphuric] acid fog and of dust. A. Delasalle. Chim. et Ind., 1920, 4, 291–316.

AFTER preliminary laboratory research a plant was erected for the Service des Poudres for precipitating the sulphuric acid fog in the fumes from two Gaillard towers. The precipitator was placed between the Gaillard plant and the fan, so that it was always under suction, and the connexions were so arranged that it could be worked in conjunction with either or both of the towers, with or without the coke box, or cut out altogether. A voltage of about 40,000 was employed. The plant consisted of 60 lead pipes, of 3 mm. thickness, 20 cm. diam., and 3 m. length, supported at their upper ends by lead-covered iron collars resting on the floor of the upper chamber. In order to prevent drops of liquid from entering the lower chamber, the pipes were made in two sections, of which the smaller was enclosed by the larger for part of its length, thus forming an annular space which was expanded into a lute, into which the acid fell and which served as a liquid seal to prevent the entry of air. From this seal the acid flowed to a collecting vessel of lead. The electrodes consisted of wire formed like a star and attached to lead-covered iron rods in the upper chamber, terminating in leaden plates in the lower chamber. The fume was made to traverse the precipitator from top to bottom, and its distribution was effected by special devices. In order to obtain acid of a strength of 40° B. (sp. gr. 1.384) maximum, the temperature at which the gases enter the precipitator must not be below 100° C., and they were therefore conducted to the apparatus direct from the coke-box inlet. The average yield from two Gaillard towers, producing 1950 kg. H₂SO₄, excluding the coke-box acid, was 11.5 g. per cub. m. of fume, the latter having a residual acidity after treatment of 1 g., of which 0.18–0.2 g. represented SO₂. Including the amount recovered from the coke-box, which averaged 5 g. per cub. m., the total yield was 575 kg. H₂SO₄ per Gaillard per 24 hrs. The use of the plant with nine Gaillard towers produced an economy of 95,000 francs per annum, and when it was also applied to the recovery of acid from seven 15-ton Kessler systems, the total gain was 200,000 francs per annum. The efficiency may be increased by raising the temperature, but is reduced if the fume velocity is too high, or by presence of nitric acid. Modifications

of the plant were effected as regards increasing the pipe lengths to 3.25 m. and the diameter to 100 mm., and the direction of the fume through the plant was reversed. In connexion with the application of the process to the recovery of dust from pyrites burning, only preliminary investigations have been carried out. A sample of recovered dust gave on analysis: FeSO₄, 24.5; FeO, 25.1; PbSO₄, 39.27; As₂O₃, 8.60; S(sol. in CS₂), 0.24; insoluble matter (Al₂O₃, SiO₂), 2.95%. The dust particles agglomerate and adhere to the collecting electrodes when the temperature does not exceed 250° C. and it is desirable to have as high a temperature as possible, and also to remove the dust mechanically if necessary. Loss of sulphur and dust must be guarded against.—W. J. W.

Persulphuric acid; Decomposition of —. H. Palme. Z. anorg. Chem., 1920, 112, 97–130.

PERSULPHURIC acid, when warmed with sulphuric acid, decomposes with formation of Caro's acid and hydrogen peroxide: H₂S₂O₈ + H₂O = H₂SO₅ + H₂SO₄; H₂SO₅ + H₂O = H₂O₂ + H₂SO₄. Both reactions are unimolecular, and their velocity is increased with increasing concentration of sulphuric acid. The velocity of the first reaction is about 39 times that of the second at 50° C. When persulphuric acid reacts with hydrogen peroxide: H₂S₂O₈ + H₂O₂ = 2H₂SO₄ + O₂, it is probable that Caro's acid is formed as an intermediate product, which then reacts with hydrogen peroxide: H₂SO₅ + H₂O₂ = H₂SO₄ + H₂O + O₂. A method of determining persulphuric acid, Caro's acid, and hydrogen peroxide when all are present in solution depends on the fact that Caro's acid reacts immediately with potassium iodide, hydrogen peroxide much more slowly, and persulphuric acid slowest of all. The solution to be analysed is diluted so as to contain the equivalent of 2.0 g. of potassium persulphate or its decomposition products per l. A 200 c.c. sample is treated with potassium iodide, and the iodine liberated is titrated rapidly with sodium thiosulphate with starch as indicator. The time taken for the titration is observed, and after the end of titration the rate of re-appearance of iodine, due to the slower action of hydrogen peroxide, is noted. A correction can then be applied to the first titration reading, which gives the quantity of Caro's acid present. The sum of Caro's acid and hydrogen peroxide is next estimated on a second sample by treating with potassium iodide and titrating with sodium sulphite. Finally in a third sample, Caro's acid and hydrogen peroxide are reduced by addition of the calculated quantity of sodium sulphite, and the persulphuric acid is estimated by the ferrous sulphate-permanganate method. (Cf. J.C.S., Nov.)—E. H. R.

Electrolysis of solutions of sodium nitrite using a copper anode. F. H. Jeffery. Faraday Soc., June, 1920. [Advance proof.]

THE reactions at the anode are stated to be: the formation of the complex anion Cu(NO₂)₂ from NO₂ ions and the copper; the decomposition of this Cu(NO₂)₂ by the water present giving the basic nitrite, Cu(NO₂)₂·CuO, and nitrous acid; the slow decomposition of the undissociated portion of the nitrous acid formed, giving nitric acid and nitric oxide. Although the presence of nitric acid in contact with the copper anode must give rise to cupric ions if no current were passing, yet, for a current of 0.16 amp., the concentration of such cupric ions as were formed was never sufficient for the deposition of copper on the cathode during electrolyses lasting several hours. The copper reacted with the NO₂ ions to form the complex anion, the anode potential remaining thereby at a low value, rather than with the NO₃ ions, which would have had the effect of raising the anode potential. The result

described is analogous to that taking place during electrolysis with a silver anode in a solution containing NO_2 and NO ions.—B. N.

Ammonium silicate. IV. *Ageing and transformations of silicic acid gels and the process of solution of silicic acid in ammonia.* R. Schwarz and O. Liede. *Ber.*, 1920, 53, 1509—1518.

THE ageing of silica gels when dried at the ordinary temperature consists in the condensation of $(\text{SiO}_2)_x$ to $(\text{SiO}_2)_y$, and this occurs without any change in the appearance of the gel; further changes occur subsequently which lead to the formation of $(\text{SiO}_2)_z$, this point being denoted by the appearance of cloudy particles in the colourless transparent mass. The phenomena at the ordinary and at higher temperature are thus very similar, but much greater dehydration is necessary in the latter case before the end-point is reached. When silicic acid dissolves in ammonia, a colloidal solution of silicic acid is primarily formed which subsequently passes into a molecular solution. (Cf. J.C.S., Nov.)—H. W.

Rubidium (caesium)-silver-gold compound and its application to the microchemical detection of gold, silver, rubidium, and caesium. E. Bayer. *Monatsh. Chem.*, 1920, 41, 223—241.

CHARACTERISTIC crystalline precipitates are formed when solutions of rubidium or caesium chloride are mixed with gold and silver solutions; rubidium-silver-gold chloride, $\text{Ag}_x\text{Au}_{2-x/3}\text{Cl}_3\cdot 3\text{RbCl}$, forms blood-red prisms and platelets, whilst the caesium compound, $\text{Ag}_x\text{Au}_{2-x/3}\text{Cl}_3\cdot 3\text{CsCl}$, crystallises in opaque cubes and stars; the silver and gold are mutually replaceable in these compounds. The crystals are adapted to the microchemical identification of gold, silver, rubidium, and caesium, the smallest quantities recognisable in this manner being 0.1, 0.01, 0.1 and 0.1 microgram, respectively. (Cf. J.C.S., Nov.)—H. W.

Rubidium (caesium)-silver-gold compound and its application to the microchemical detection of gold, silver, rubidium, and caesium. F. Emich. *Monatsh. Chem.*, 1920, 41, 243—252. (See preceding abstract.)

CONTRARY to previous observations (Emich, *Monatsh. Chem.*, 1918, 39, 775), it is now found that the place of rubidium cannot be taken by potassium in this compound, and the value of the method for the detection of caesium and rubidium is thereby greatly enhanced. (Cf. J.C.S., Nov.)—H. W.

Aluminium with sodium peroxide; Ignition of a mixture of — by means of water. O. Olmann. *Ber.*, 1920, 53, 1427—1429.

A MIXTURE of finely-divided aluminium with sodium peroxide, preferably in the proportion of 1:4.3, is readily ignited with feeble explosion by the addition of a few drops of water.—H. W.

Antimony sulphide; Golden —. F. Kirchhof. *Z. anorg. Chem.*, 1920, 112, 67—80.

THE ordinary golden antimony sulphide is described in the literature as consisting of a mixture of antimony pentasulphide and trisulphide with a varying amount of sulphur, according to the method of preparation. It is shown that, in its purest form, golden antimony sulphide contains about 8% of free (extractable) sulphur, and that the residue has the composition $\text{Sb}_2\text{S}_3 + 3\text{ZnCl}_2 + \text{H}_2\text{S}_2 + 2\text{H}_2\text{S}$. The orange-red compound thus obtained contains no free sulphur. It is concluded that antimony pentasulphide does not exist. The

principal constituent of golden antimony sulphide is the tetrasulphide Sb_2S_4 , with a variable quantity, from 0 to 50%, of the trisulphide, Sb_2S_3 , and a variable amount of free sulphur. Sb_2S_4 is to be regarded as antimony thioantimonate, i.e., $\text{Sb}(\text{SbS}_2)$. (Cf. J.C.S., Nov.)—E. H. R.

Stannic and plumbic acids; Salts of —. H. Zocher. *Z. anorg. Chem.*, 1920, 112, 1—66.

SODIUM stannate, prepared by dissolving freshly-prepared stannic hydroxide in hot, concentrated sodium hydroxide, forms a crystalline precipitate, $\text{Na}_2\text{Sn}(\text{OH})_6$, almost insoluble in 6—7 *N* caustic soda solution. A higher hydrate, $\text{Na}_2\text{Sn}(\text{OH})_6\cdot\text{H}_2\text{O}$, was also obtained by precipitation from an aqueous sodium hydroxide solution with alcohol. Three hydrates of potassium stannate were isolated: $\text{K}_2\text{Sn}(\text{OH})_6$, $\text{K}_2\text{Sn}(\text{OH})_6\cdot\text{H}_2\text{O}$, and $\text{K}_2\text{Sn}(\text{OH})_6\cdot 2\text{H}_2\text{O}$. The alkali stannates are unstable and readily decomposed by carbonic acid. Sodium plumbate was prepared by dissolving freshly-precipitated lead peroxide, in small quantities at a time, in strong boiling sodium hydroxide solution. It forms a white, crystalline precipitate, probably $\text{Na}_2\text{Pb}(\text{OH})_6$; analyses always show a slight excess of sodium, $\text{PbO}_2:\text{Na}_2\text{O}=1:1.14$, but this may be due to the presence of carbonate or to adsorption of NaOH . The properties of stannic and plumbic acid are discussed at great length from the colloidal standpoint. (Cf. J.C.S., Nov.)—E. H. R.

Air; Separation of — into oxygen and nitrogen. F. E. Norton. *Chem. and Met. Eng.*, 1920, 23, 511—518.

ANY considerable extension of the use of oxygen for industrial purposes is prevented by the high cost of separating the gas, and this is ascribed to the neglect of sound thermodynamical principles in the processes employed up to the present. The chief defects are the expansion of the compressed air at a very low temperature, which results in the loss of all the work of the primary compression, and the introduction of external heat into the fractionating still. To obtain favourable results the heat of the high-pressure gas should be transferred to the return current of high-pressure gas, and all heat leakage absorbed into high-pressure gas at as high a temperature as possible. All heat carried out of the system should be carried by the smallest stream of low-pressure gas and at the highest temperature possible. Temperature differences between streams of gas should be as small as possible, especially at the cold end of the apparatus. The latent heat of condensation of entering fluids should be exchanged as completely as possible for the latent heat of evaporation of escaping fluids, and the production of liquid air by the cooling effect of nozzles or expansion engines should be avoided. Compression should be effected at the temperature of separation, and all heat rejected to the high-pressure return circuit with subsequent high-pressure expansion. (Cf. Cottrell, J., 1919, 124 *et seq.*)—W. H. C.

Carbonic acid and its salts. Kolthoff. *See XXIII.*

Phosphoric acid and its salts. Kolthoff. *See XXIII.*

Ammonia. Hahn and Kootz. *See XXIII.*

PATENTS.

Nitric acid; Process of making —. British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 150,836, 17.6.19.

OXIDES of nitrogen from the catalytic oxidation of ammonia are absorbed in nitric acid at -40°C . in a tower or absorption vessel fitted with a revolving shaft and discs. This absorber is cooled by the evaporation of liquid ammonia under reduced pressure, the ammonia gas being returned to the

oxidation apparatus. The nitric acid saturated with oxides of nitrogen passes on through a heat interchanger to the anode chamber of an electrolytic cell to oxidise the nitrogen oxides to nitric acid, and the hydrogen liberated at the cathode (which is immersed in dilute sulphuric acid) and the residual nitrogen from the absorber are utilised in the production of ammonia.—C. I.

Alkali sulphates and sulphuric acid; Manufacture of — from [alkali] bisulphates. Farbenfabr. vorm. F. Bayer und Co. G.P. 297,931, 24.3.16.

An alkali bisulphate is heated in a revolving tube furnace, yielding a light, porous sulphate.

—L. A. C.

Hydrocyanic acid; Method of producing —. G. H. Buchanan, Assr. to American Cyanamid Co. U.S.P. 1,352,655, 14.9.20. Appl., 12.11.19.

CRUDE cyanide containing substantial quantities of lime and calcium and sodium chlorides is dissolved in a refrigerated dilute solution of a mineral acid, more acid being present than is necessary to react with all the impurities. The solution is then passed in counter-current against a source of heat whereby the hydrocyanic acid is distilled.—A. R. P.

Mercuric oxide; Manufacture of — by decomposition of mercurous nitrate. G. Brusa, and V. Borelli & Co. E.P. 150,917, 30.10.19.

Two connected revolving drums are used, one heated and the other cooled. In the former mercurous nitrate is decomposed, and in the latter mercury is acted upon by oxygen mixed with nitrogen peroxide from the first drum. The contents of the second drum are then used as the charge for the first drum, which yields in turn the desired mercuric oxide.—C. I.

Silicate of soda and the like; Method of producing —. W. H. Stanton. U.S.P. 1,352,700, 14.9.20. Appl., 26.1.17.

In the manufacture of sodium silicate a furnace with two hearths is used, which are heated successively by gases of combustion. When the treatment of the charge on the hotter hearth is complete, a new charge is introduced and the direction of the heating gases reversed.—C. I.

Alkaline melts; [Electrolytic] manufacture of reducing —. E. Bergve, Assr. to Norsk Hydro-Elektrisk Kvaelfstofaktieselskab. U.S.P. 1,354,451, 28.9.20. Appl., 10.3.19.

An alkaline melt containing dissolved alkali metal for chemical reductions and syntheses is produced by electrolysing a melt containing an alkali hydroxide, with an anode current density of 20–50 amps. per sq. cm., and a cathode current density of less than 1 amp. per sq. cm.—J. S. G. T.

Alumina and silica; Treating material [e.g., clay] containing —. H. Fleissner. G.P. 322,844, 9.5.19. Conv., 14.4.16.

A MIXTURE of the material (containing 1 mol. of alumina) and calcium sulphate (2 mols.) is heated to a temperature not high enough to liberate sulphur dioxide, i.e., a dull red-heat, with the production of calcium metasilicate (2 mols.) and basic aluminium sulphate (1 mol.). The product is leached with dilute sulphuric acid to dissolve the aluminium sulphate.—L. A. C.

Hydrogen sulphide; Separation of — from gases. A. Krieger. G.P. 322,938, 10.12.18.

THE gases are washed with ammoniacal liquor, fresh water, or a dilute solution of ammonia, at 30°–45° C. Above 45° C. little hydrogen sulphide is absorbed, whilst below 30° C. the absorption of carbon dioxide increases.—J. H. L.

Ammonium chloride; Process for the direct production of — from ammonium carbonate. Wagenmann, Seybel, and Co., A.-G. G. P. 323,038, 20.11.17. Conv., 10.11.17.

DRY or nearly dry hydrogen chloride is led over dry neutral and/or acid ammonium carbonate, or into solutions containing the same in suspension, with or without ammonium chloride in addition. The mother liquor, after separation of the solid product, is treated with ammonia and the carbon dioxide liberated by the action of hydrochloric acid on a previous quantity of the material to produce a fresh suspension of ammonium carbonate.

—L. A. C.

Liquid oxygen; Apparatus for producing —. C. F. and O. J. Crommett, Assrs. to G. L. Cabot. U.S.P. 1,354,380, 28.9.20. Appl., 7.1.14. Renewed 1.7.16.

AIR is forced under high pressure through a coil surrounded by a cooler containing nitrogenous gas at a temperature below freezing point and through a separator. A portion of the cooler extends through the separator, wherein water is frozen out of the air.—L. A. C.

Hydrogen for catalytic reactions; Increasing the activity of —. Berlin-Anhaltische Maschinenbau A.-G. G.P. 301,364, 18.3.16.

INERT hydrogen is activated by the addition of a small percentage of air, oxygen, or other material capable of entering into reaction during its subsequent passage over a catalyst; e.g., water-gas or hydrogen prepared by the action of steam on iron is rendered suitable for the manufacture of tetrahydronaphthalene from naphthalene at 180°–190° C. and 10 atm. pressure by addition of 2% of air or oxygen and passage over hot, finely-divided nickel.—L. A. C.

Zinc chloride; Manufacture of —. C. F. Burgess Laboratories, Asses. of O. W. Storey. E.P. 133,319, 2.10.19. Conv., 26.9.18.

SEE U.S.P. 1,314,715 of 1919; J., 1919, 818 A.

Cyanides; Production of —. C. T. Thorssell and H. L. R. Lundén. E.P. 151,098, 20.6.19.

SEE U.S.P. 1,342,834 of 1920; J., 1920, 544 A.

Molybdenum compounds. E.P. 131,898. See X.

VIII.—GLASS; CERAMICS.

Sedimentation [of kaolin]. P. Rena and P. György. Biochem. Zeits., 1920, 105, 133–140.

THE authors find that nearly all of a number of non-electrolytes examined increase the velocity of sedimentation of kaolin; only in the case of compounds virtually insoluble in water could no effect be detected. The magnitudes of the accelerations produced by homolegous compounds increase with the length of the chain. (Cf. J.C.S., Nov.)

—T. H. P.

Lead problem in the ceramic industry. L. Petrik. Sprechsaal, 1920, 53, 405–406.

GLAZES with a lead solubility (Petrik, J., 1914, 80) of less than 1% may be obtained by preparing a lead frit containing a minimum of alkali and no boric acid and a leadless frit containing the requisite quantities of alkali and boric acid and mixing these frits for use. A series of recipes is given, including leadless frits in which pandermite (calcium borate) is used. The solubility of lead glazes containing about 15% of tin oxide is exceptionally low, but they must be completely fused and not merely fritted. It is suggested that standard frits could be prepared more cheaply on a large scale

at lead works than by the potters themselves. Leadless frits might also be made on a large scale, and by the use of mixtures of such frits, together with felspar, quartz, kaolin, pandermite, etc., glazes containing less than 1% of soluble lead could be prepared to meet any required conditions.

—A. B. S.

Silica bricks for coke-ovens. Enzenauer. See Hb.

PATENTS.

Glass-melting furnaces. L. le B. Mount. E.P. 151,181, 28.11.19.

THE outlets for feeding molten glass to the glass-ware machines are located at the end of the tank adjacent to the recuperator, and consist of holes formed through a raised portion of the bottom of the tank near its sides or end. The outflowing glass is sufficiently fluid without providing any means for heating the glass as it flows out. The products of combustion flow to and fro through the horizontal tubes of the recuperator, and are then led by passages above the crown of the furnace to the chimney. Air passes along a flue below the tank, flows upwards around the tubes of the recuperator, and is then led through passages on the furnace crown placed below the waste gas passages and admitted to the furnace by ports in the neighbourhood of the incoming fuel.—H. S. H.

Coating glass. E. P. 150,598. See Hb.

IX.—BUILDING MATERIALS.

PATENTS.

Concrete; Preparing pumice-stone for use in —. E. C. Bayer. U.S.P. 1,354,233, 28.9.20. Appl., 15.11.19.

PUMICE-STONE is rendered suitable for making light concrete by crushing and heating to a temperature ranging from 1000° C. to the melting point. The fragments contract and become glass-hard, and the outer pores are completely closed through superficial melting, while each separate piece still remains porous inside.—H. S. H.

Cement; Waterproofing —. B. von Leski. G.P. 323,031, 22.11.13.

AMMONIUM resinate is added to cement to make it more impervious to water after setting.—L. A. C.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Cast iron; Influence of nickel and cobalt on the physical and chemical properties of —. O. Bauer and E. Piwowarsky. Stahl u. Eisen, 1920, 40, 1300—1302.

AN addition of up to 1.0% of nickel to cast iron gives favourable results. The resistance to bending is increased by about 30%, the crushing strength increases by 30%, the tensile strength by 25%, and the hardness by 18%. The rate of solution in acid is lowered, although not to the extent expected, the catalytic influence of the graphite being apparently too strong. Nothing is gained by addition of over 1.5% of nickel, as the influence of nickel on the separation of graphite outweighs the refining effect on the ferrite grains. The structure of nickel iron is very similar to that of ordinary grey iron. Cast irons with a nickel content of 1.2% are recommended for machine parts and toothed wheels. With additions of cobalt up to 2% the resistance to bending, crushing strength, and tensile strength decrease, while the hardness

increases to a slight extent. Cobalt hinders the separation of graphite and favours the formation of carbide. The rate of solution in acid is also lowered continuously as the cobalt increases.

—J. W. D.

Tin; Electrolytic refining of —. E. F. Kern. Trans. Amer. Electrochem. Soc., 1920, 38, 187—206. [Advance copy.]

EXPERIMENTS were made with the following electrolytes, each containing 6% Sn: Stannous sodium chloride containing 2.5 g. sodium chloride per 100 c.c.; stannous fluoborate with 5% of free acid; stannous naphthalenesulphonate with 10% of free acid; stannous sulphate with 2.5, 5, and 7% of free acid. The last-named solution gave the best anode deposit when no addition agent was added, but in no case were smooth adherent deposits obtained. When 0.1—0.2% of peptone or gelatin was added to the electrolyte adherent smooth deposits were obtained with the fluoborate and sulphate electrolytes, especially if the solutions were warm (40° C.).—A. R. P.

Brass; Electro-deposition of — from cyanide solutions. A. L. Ferguson and E. G. Sturdevant. Trans. Amer. Electrochem. Soc., 1920, 87—122. [Advance copy.]

EXPERIMENTS on the effect of temperature, current density, and constitution of the anode on the electro-deposition of brass from cyanide solutions led to the following conclusions: The percentage of copper in the deposit is increased by increasing the ratio of copper to zinc in the solution (a Cu:Zn ratio of 4:2 gives a deposit containing 65% Cu), by increasing the temperature, and by the addition of slightly acid substances or of weak acids to the electrolyte, whereas it is decreased by an increase of current density or by the addition of slightly alkaline substances to the electrolyte. Neutral substances have no influence on the electrolysis, but excess of free cyanide decreases the cathode efficiency without increasing the anode efficiency. The most satisfactory deposits of brass are obtained by using a solution containing 35 g. of metals per l. in the above ratio and a small quantity of ammonia, which ensures a bright yellow deposit, together with a current density not greater than 0.3 amp. per sq. dm. Brasses containing 62.3—85% Cu dissolve as such anodically, the efficiency of corrosion being about equal to that of copper. Electrolytically deposited brasses containing 37.6—82% Cu have, in a plating solution, approximately the same potential, which is nearer to that of copper than to that of zinc.—A. R. P.

Lead-bismuth; The binary system —. W. Herold. Z. anorg. Chem., 1920, 112, 131—151.

THE electrical conductivity at 0° and 123° C., the hardness, and the microscopic structure of lead-bismuth alloys were studied with the object of determining the limits of the eutectic range. The conductivity experiments indicated for the limit of solubility of bismuth in lead, 17.5—18.5% Bi, and for the solubility of lead in bismuth, 1.6% Pb. The hardness-composition curve also had a maximum at about 17.5—18.5% Bi, but the interpretation of the hardness results was uncertain owing to the influence on the hardness of previous treatment. The curve had a wave-form between 18.5% and 100% Bi, with an inflexion close to the eutectic point, indicating that the hardness of the eutectic alloys is not derived additively from that of the two series of mixed crystals. The microscopical examination showed that equilibrium is attained in these alloys only after prolonged annealing. The true eutectic limits appear to lie at about 40% Bi on the lead side and 0.2 to 0.5% Pb on the bismuth side.—E. H. R.

Binary systems [alloys]; Relation between chemical resistance and constitution of —. K. Wagenmann. *Metall u. Erz*, 1920, 17, 377—381, 402—408.

FROM consideration of the available experimental data on the resistance of binary alloys to solvents, with or without the presence of atmospheric oxygen, it is concluded that if chemical compounds are possible between the constituents of the alloys a maximum resistance to solution is often found at concentrations corresponding to these compounds. In alloys, the constituents of which form mixed crystals, a maximum occurs at the saturation concentration of these solid solutions, or at least an increased resistance to attack occurs within the range of concentration corresponding to a solid solution of the pure components. Resistance to chemical action is influenced by grain size of the alloy. A minimum in the resistance to attack is obtained with a heterogeneous structure, specially with an eutectic, in the lamellar structure of which electrolytic action probably takes place. In an appendix particulars are given of the methods and apparatus used in the experiments.—T. H. Bu.

Electrical conductivity in metals and alloys at high temperature; Measurement of —. J. L. Haughton. *Faraday Soc.*, June, 1920. [Advance proof.]

AN apparatus for the measurement of electrical conductivity at high temperatures, up to 1000° C., was designed primarily to determine the conductivity of a series of alloys at varying temperatures, with a view to obtaining some information on their constitution. The principle on which the apparatus works is the measurement of the voltage drop along a fixed length of the specimen, through which a constant current is passing. This voltage drop is proportional to the resistance between the points of contact so long as the current is kept constant. To ensure this, the same current is passed through a constantan strip, the resistance of which can be arranged to be of the same order as that of the specimen, and is accurately known, and the voltage drop along this resistance is measured with the same instrument and almost at the same time as that along the specimen. In this way, not only is the constancy of current checked (this being adjusted by means of a rheostat when necessary), but also the instrument which is reading the voltage drop is calibrated directly in some multiple of ohms.—B. N.

Valuation of fuels. Schwier. See IIa.

Aluminium and sodium peroxide. Ohmann. See VII.

PATENTS.

Electrical [steel hardening] furnaces. L. W. Wild and E. P. Barfield. E.P. 150,793, 4.6.19.

IN furnaces employed for hardening steel by means of a bath of molten salt contained in a silica crucible or the like (E.P. 112,731; J., 1918, 154A), the silica crucible is replaced by a silica furnace lining within which is a loosely fitting metal (e.g., iron) pot for the reception of the molten salt having a coefficient of expansion approximating to that of the salt.—J. S. G. T.

Iron silicide; Process for producing articles of —. N. Petinot. U.S.P. 1,346,333, 13.7.20. Appl., 18.8.19.

ARTICLES of iron silicide, resistant to corrosion, are made by casting the alloy in graphite moulds.—B. N.

Alloy of iron, chromium, and titanium, and [electrical] process for the production thereof. Method for making chrome-steel. H. C. Sicard, Assr. to United States Ferro-Alloys Corp. U.S.P. (a) 1,346,343 and (b) 1,346,344, 13.7.20. Appl., 18.8.19.

(A) CHROMIUM ore, rutile, and scrap iron are mixed

with carbon sufficient to reduce the chromium and titanium oxides, and also to form by combination with the chromium, titanium, and iron a complex carbide in the finished alloy. The mixture is then smelted in an electric furnace. (b) Chromium steel is made by adding to molten steel an alloy of iron, chromium, titanium, and carbon.—B. N.

Steel; Manufacture of —. G. B. Waterhouse. U.S.P. 1,352,710, 14.9.20. Appl., 31.7.19.

THE physical structure of steels with a maximum content of 0.25% C is improved and the distribution of sulphur rendered more uniform by treating the molten steel with such a quantity of silicon that the silicon content of the resulting metal is not materially increased.—A. R. P.

Electrolytic soft iron; Production of —. M. Schlötter. G.P. 308,543, 7.2.18.

FERRIC salts formed during electrolysis are reduced to ferrous salts by the addition of hydroxylamine, hydrazine, compounds of the same, or carbon-free electrolytic iron. Iron with a Brinell hardness of 50—60 is thus obtained.—L. A. C.

Ingot iron and steel and other metals; Removing oxygen, sulphur, and phosphorus from molten —. Deutsch-Luxemburgische Bergwerks- und Hütten-A.-G., and S. Hilpert. G.P. 322,988, 22.9.16.

ALUMINIUM carbide is added to the molten metal, and, if necessary, owing to the absence of slag, e.g., in ingot moulds, other carbides, such as calcium carbide, are also added so that a fusible slag is produced.—L. A. C.

Molybdenum compounds; Extraction of — from yellow lead ores [wulfenite]. Deutsche Molybdaen-Werke, G.m.b.H. E.P. 131,898, 27.8.19. Conv., 2.10.16. Addn. to 131,897.

FINELY powdered wulfenite is digested hot or cold with a solution of ammonium or alkali sulphide or fused with alkali sulphide or with alkali sulphate and carbon, the amount of the sulphur compound used in any case being less than that required to transform the molybdenum into alkali sulphomolybdate, so that any of the latter compound first produced is changed into molybdate by the excess of ore present.—A. R. P.

Electrodes for electric welding. A. Le Chatelier. E.P. 147,270, 14.4.19.

A COATING for electrodes for electric welding consists of a mixture of substances, preferably anhydrous and only containing a small proportion of oxides decomposable by heat, refractory enough to prevent too rapid fusion of the coating and giving a fusible and acid slag. It contains from 33 to 67% of silica (or equivalent quantities of native silica or silicates), 23 to 62% of iron oxide (or equivalent quantities of suitably ground and dried iron ores), 5 to 22% of lime as carbonate (or equivalent amounts of carbonates of other alkaline earths, including magnesia and alumina) in a finely powdered state with addition of carbon. A small quantity of another metallic oxide, such as manganese or nickel oxide, may be added to the iron oxide. The mixture is made into a paste by means of a binding material such as a solution of dextrin or alkali silicates, or by a binding medium dissolved in a liquid which does not contain water.—B. N.

Electrodes for use in electric welding. A. C. Hyde. E.P. 148,645, 1.5.19.

AN electrode, for the electric welding of iron or steel, is composed of iron or steel, and a deoxidising material, such as silicon or aluminium, which has been introduced into the iron or steel before it is formed into an electrode.—B. N.

Welding-rod. R. F. Russell, Assr. to Air Reduction Co. U.S.P. 1,352,534, 14.9.20. Appl., 24.6.19.

A RARE-EARTH metal is incorporated in a welding-rod composed mainly of iron, to prevent the formation of deleterious oxides and nitrides when the rod is melted to form a weld.—J. S. G. T.

Welding steel; Electrically —. J. W. Plant. U.S.P. 1,354,266, 28.9.20. Appl., 29.1.19.

AN arc is struck between the part to be welded and an electrode of corresponding material, within a welding chamber, in which a non-oxidising atmosphere is maintained.—J. S. G. T.

Alloy for electric welding. E. J. Kingsbury. U.S.P. 1,350,317, 24.8.20. Appl., 20.11.19.

AN electric welding alloy contains iron, carbon, manganese, and vanadium.—B. N.

Electro-deposition of metals. J. H. B. Jacobs. E.P. 147,338, 23.12.19.

THE apparatus comprises a revolving drum, and an anode formed in two parts, each of which is supported on a carrier of wood, or like non-conducting member, inserted into the drum through openings in each end. A distributor is fitted on the drum for supplying current to the cathode. The electroplated articles are removed from the drum through openings in the ends.—B. N.

Copper plates for process printing; [Electrolytic] manufacture of —. S. O. Cowper-Coles. E.P. 151,027, 5.3.19.

COPPER is electrolytically deposited upon cathode plates suspended by connecting rods attached to eccentrics, so that an oscillating or reciprocating motion is imparted to the plates. A vertical paddle at one or both ends of the depositing vat causes the electrolyte to pass with a velocity of 500 to 600 ft. per min. over the surface of the cathode plates, the circulating electrolyte being also passed continuously through an external filter.—J. S. G. T.

Zinc; Recovery of — by electrolysis. R. H. Stevens, Assr. to Electrolytic Zinc Co. of Australasia Proprietary, Ltd. U.S.P. 1,347,189, 20.7.20. Appl., 11.11.19.

THE cathode is provided with a protective coating above the normal solution level, by first carrying out a preliminary electrolysis with the cathode immersed to substantially a line immediately below the supporting bars, the cathodes having wooden strips fixed at or about the normal solution level which prevent the deposition of zinc under them. The strips are then removed and the electrolysis continued in the ordinary way in cells with the solution at normal level, whereby the zinc deposited subsequently can be stripped along the line of weakness at or about the solution level.—B. N.

Metals; Method of and apparatus for reduction of — to powder. J. P. Mellor. E.P. 150,490, 4.7.19.

THE metal is heated to a temperature approaching its volatilisation point (in the case of zinc, 700°–800° C.), and poured in a thin stream upon a flat jet of high-pressure steam.—B. M. V.

Furnace; Rotary electric — for continuously melting zinc dust. C. E. Cornelius. G.P. 322,808, 21.10.19. Conv., 20.8.19.

THE zinc dust is introduced into the middle portion of the furnace.—L. A. C.

Electrolytic pickling process. J. Coulson, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. 1,347,897, 27.7.20. Appl., 3.11.17.

COPPER objects are pickled electrolytically by subjecting them as cathodes to the action of an elec-

tric current, so as to dissociate the surface oxide, and simultaneously recover any copper which is set free.—B. N.

Reverberatory furnace [; Method of charging a —]. H. L. Charles. U.S.P. 1,351,835, 7.9.20. Appl., 30.8.17. Renewed 23.1.20.

THE ore charge is dropped through the roof on to the hearth so as to form mounds at frequent intervals.—B. M. V.

Copper and other metals; Process and apparatus for reclaiming and refining —. A. R. Partidge. U.S.P. 1,352,912, 14.9.20. Appl., 15.9.19.

MATERIAL from which copper or other metals are to be reclaimed is heated to the melting temperature, and the temperature is then lowered to cause the materials to "sweat" and trickle down an inclined surface, the trickling material being purified by treatment with oxygen.—L. A. C.

Molten metal; Process of treating —. O. Thiel, Assr. to The Chemical Foundation, Inc. U.S.P. 1,353,126, 14.9.20. Appl., 28.12.16.

SLAG is removed by overflow in any desired quantity from the surface of a bath of molten metal by adding a corresponding amount of unmelted metal of the same kind to the melt.—A. R. P.

Zinc-lead alloy. H. Falkenberg. G.P. 322,960, 25.4.19. Addn. to 300,111 (*cf.* J., 1919, 292A, 374A, 869A).

THE alloy contains up to 5% Fe, less than 2% Al, and about 1.5% Cu.—L. A. C.

Steel; Process of making —. G. L. Fisk. E.P. 151,092, 17.6.19.

SEE U.S.P. 1,312,474 of 1919; J., 1919, 727A.

Steel; Manufacture of —. G. Cinille. U.S.P. 1,352,580, 14.9.20. Appl., 10.8.10. Renewed 30.1.19.

SEE F.P. 416,771 of 1909; J., 1910, 1458.

Ores of certain metals; Treatment of —. R. D. Lance. E.P. 14,267, 13.6.14. Conv., 15.4.14.

SEE F.P. 476,304 of 1914; J., 1916, 54.

Zinc; Recovery of — by electrolysis. Electrolytic Zinc Co. of Australasia Proprietary, Ltd. E.P. 136,151, 26.11.19. Conv., 3.12.18.

SEE U.S.P. 1,347,189 of 1920; preceding.

Furnace [; Metallurgical tilting —]. A. M. Aubert. U.S.P. 1,353,818, 28.9.20. Appl., 26.4.18.

SEE E.P. 115,840 of 1918; J., 1919, 1A.

Electric welding; Methods of and apparatus for —. The British Thomson-Houston Co. From General Electric Co. E.P. 150,756, 15.10.18.

Furnace; Open-hearth —. J. O. Griggs. U.S.P. 1,353,830, 28.9.20. Appl., 7.7.19.

Oxidising steel billets. U.S.P. 1,350,877. See XI.

XI.—ELECTRO-CHEMISTRY.

Electrical precipitation. Delasalle. See VII.

Electrolysis of sodium nitrite. Jeffery. See VII.

Refining tin. Kern. See X.

Electrical conductivity at high temperatures. Haughton. See X.

Electro-chemistry of tannins. Grassier. See XV.

PATENTS.

Electric furnace. R. Steiger, Assee. of H. Blumer. E.P. 125,391, 7.4.19. Conv., 8.4.18.

ELECTRIC furnaces, using polyphase current, are operated with combined arc and resistance heating, and are provided with an electrode for each phase of the system above the surface of the bath and oppositely arranged electrodes in the hearth. The hearth electrodes and one of the upper electrodes are connected with the same point in the supply system. The upper electrodes are switched in during those stages of the working in which the quietest possible behaviour of the material under treatment is required, with a more or less energetic surface heating, whilst in those stages of the working in which an energetic permeation of the whole of the material in the direction of its depth is necessary, one of the upper electrodes is switched out and the hearth electrodes are switched in.

—B. N.

Resistance for electric furnaces. Electric furnace. E. Piquerez. E.P. (A) 139,448, and (B) 132,232, 18.7.19. Conv., (A) 26.2.19, (B) 2.9.18.

(A) ELECTRIC resistance elements, in the form of rods with greater cross-section at the parts where the current leads are attached, are provided with a deposit of metal of low electrical resistance at the enlarged portions, the deposit being formed on the outside of the rod or in a hole bored in the rod. The deposit may be covered with a coating of enamel of high melting point. (B) The heads of the resistance elements are situated in spaces provided between the outside of the furnace walls and an outer casing, through which spaces air currents can flow for cooling the heads.—B. N.

Electric furnaces; Transformation of polyphase currents, particularly for use in —. J. Bibby. E.P. 149,049, 2.5.19.

POLYPHASE currents are transformed electrically by supplying three-phase current to five static transformers having transformer ratios of 0.886, 1.22, 1.67, 1.22, and 1.0 respectively, the primary windings being so connected with the supply mains and the secondary windings with load terminals, that eight-phase currents having eight voltages progressively 45° apart are obtained. The eight-phase current is supplied to an electric furnace having eight octagonally-spaced electrodes. By using the larger number of electrodes the heat can be generated more effectively and more uniformly than hitherto.—B. N.

Electric furnace. E. L. Smalley. U.S.P. 1,346,854, 20.7.20. Appl., 21.10.19.

THE furnace is provided with a series of heating units, supported by elements which may be shifted horizontally, whereby the units are rendered both adjustable and renewable.—B. N.

Furnace; Electric — for heating ribbons and bars. Double hot-chamber [electric] furnace. O. A. Colby, Assr. to Westinghouse Electric and Manufacturing Co. U.S.P. (A) 1,347,895 and (B) 1,347,896, 27.7.20. Appl., (A) 16.9.18 and (B) 14.12.18.

(A) A HEATING chamber is provided with a roof forming a resistor, and a supporting means associated with the resistor to provide the heating chamber with a rectangular cross-section, the resistor being adapted to be made hotter at one portion than at another. (B) The furnace is provided with two associated heating chambers, a pre-heating chamber, and resistors for supplying heat to all the chambers.—B. N.

Furnace; Electric —. Method of oxidising materials [steel billets] in electric furnaces. W. S. Mayer and W. C. Kennedy. U.S.P. (A) 1,350,876 and (B) 1,350,877, 24.8.20. Appl., (A) 15.12.16 and (B) 17.3.17.

(A) AN electric resistance furnace is provided with a wall including a number of resistance units, each comprising a block of refractory material and a resistance element. (B) Metals, e.g., steel billets, are oxidised on the surface to remove surface imperfections by heating in an electric furnace to a temperature sufficient to form a scale, a regulated predetermined amount of oxygen being supplied to the heated material.—B. N.

Electric-arc furnace. F. von Schlegell. U.S.P. 1,352,541, 14.9.20. Appl., 28.1.20.

THE electrodes are supported in contact with each other in such a manner that they can move relatively to one another under the influence of the magnetic field set up when a current flows through the electrodes, so that the electrodes are in contact when no heating current is flowing, and an arc is struck automatically when current is supplied.

—J. S. G. T.

Electrodes; Manufacture of carbon —. C. H. Thompson. E.P. 147,247, 8.3.19.

THE firing or burning of the electrodes is effected in a muffle or the like without the packing materials hitherto employed, and the material is subjected during the firing to the action of producer gas. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 9835 of 1888, 18,263 and 23,830 of 1899, and 371 of 1900; J., 1889, 697; 1899, 1112; 1900, 355, 911.—B. N.)

Electric [dynamo] brushes; Material for —. A. E. White. From Becker Bros. E.P. 150,765, 29.4.19.

CEROUS oxide, produced electrolytically, either alone or mixed with other oxides, by electrolysis of sodium chloride solution, using an anode of copper or a copper alloy, is mixed with an excess of graphitic carbon and reduced in a closed chamber in a reducing atmosphere, and the resulting mixture of graphite and copper or copper alloy is employed for making electric dynamo brushes and the like.—J. S. G. T.

Negative plate for (A) electric batteries or (B) electric accumulators, and process for making it. A. Pouchain. E.P. (A) 150,810 and (B) 150,811, 6.6.19.

(A) An amalgamated plate of zinc is employed as cathode in an electrolytic bath containing water slightly acidified with sulphuric acid to which are added about 5% of magnesium sulphate, 5% of mercuric sulphate, and preferably a small amount of ammonium sulphate. The current density is adjusted to about 2 ampères per sq. dcm. The plate is preferably perforated, and on removal from the bath is dried at a gentle heat and wrapped in a sheet of greased paper. (B) A supporting plate of a non-metallic electrically conducting material, preferably ribbed and perforated, is employed as cathode in a bath containing a salt of magnesium, a salt of mercury, and a salt of zinc, until a deposit of zinc about 1 mm. thick is obtained. If desired the support may first be covered with a very thin layer of copper electrolytically deposited.

—J. S. G. T.

Electrolytic cell. R. M. Shaw. U.S.P. 1,346,849, 20.7.20. Appl., 29.7.18.

THE apparatus comprises a receptacle forming one electrode, and a diaphragm carried by a removable member which is supported upon the receptacle. A second removable member, also supported upon

the receptacle, carries an electrode which is insulated from the receptacle and extends within the diaphragm. Means are provided for separately conducting the gases from the receptacle.—B. N.

Electrolytic apparatus. Apparatus for [electrically] treating liquids with gases. W. E. Greenawalt. U.S.P. (a) 1,317,088 and (n) 1,317,089, 20.7.20. Appl., (a) 1.2.17 and (n) 25.3.15. (n) Renewed 27.2.20.

(A) A chamber provided with a gas inlet and outlet is adapted to contain a liquid and to confine a gas over the liquid. Means are provided for causing the gas to flow from the inlet to the outlet, and for taking the gas from above the liquid and forcing it through the liquid, whilst passing an electric current through the liquid. (n) A stationary chamber is provided with gas and liquid inlets and outlets, and with means operated within the chamber for submerging some of the gas in the liquid. Electrical means are used for causing a chemical reaction between the liquid and the gas.—B. N.

Electrolytic apparatus. W. E. Greenawalt. U.S.P. 1,353,995, 28.9.19. Appl., 7.1.19.

An electrolytic vessel comprises an electrolyser and a chamber in which a relatively large volume of electrolyte is kept under continuous treatment with a reducing gas which is confined above the surface of the electrolyte. This chamber is provided with inlets and outlets for the gas and electrolyte. The reduced electrolyte flows from the chamber to the electrolyser, oxidised electrolyte flowing in the reverse direction, and the rate of flow and time of treatment in the electrolyser are so regulated that the oxidation and reduction processes are maintained within predetermined limits.—J. S. G. T.

Electrolytic cell. H. I. Allen and K. R. Fox, Assrs. to Electron Chemical Co. U.S.P. 1,349,597, 17.8.20. Appl., 2.4.19.

An electrolytic cell comprises an upper closed free-board section, suspended from which, by a liquid-tight joint, is a metallic basket-like cathode, provided with perforated side, bottom, and end walls furnishing active faces. A porous diaphragm covers the inner faces of the cathode. A tubular anode, occupying the space within the cathode, is spaced from the inner faces of the end, bottom, and side walls, the upper edges of the anode being free so as to permit the electrolyte to flow over them. The free-board section rests on a tank, with the cathode suspended in it, but spaced from the walls, and means are provided for withdrawing the effluent from the tank so as to prevent the submergence of the cathode, the parts being so arranged that the level of the electrolyte in the free-board section may be above the top of the tank so as to increase the hydrostatic pressure.—B. N.

Electric battery. O. E. Ruhoff, Assr. to French Battery and Carbon Co. U.S.P. 1,352,987, 7.9.20. Appl., 21.11.18.

The battery contains a depolarising mixture consisting of 50 pts. of manganese dioxide, 49 pts. of carbonaceous material, 10 pts. of ammonium chloride, and 12 pts. of a zinc chloride solution of 45° B. (sp. gr. 1.45), the manganese dioxide being of such a character as to yield no trace of poisonous metal when leached with a 30° zinc chloride solution saturated with ammonium chloride.—J. H. L.

Electric battery; Oil-protected — and method of producing the same. O. E. Ruhoff, Assr. to French Battery and Carbon Co. U.S.P. 1,352,831, 11.9.20. Appl., 28.1.19.

Is an electric battery constituted of carbon, zinc, electrolyte, and depolarising agent, a thin coating

of oil is employed to protect the zinc from chemical action on open circuit.—J. S. G. T.

Electrolytic cells. W. J. Mellersh-Jackson. From Allen Electrolytic Cell Corp. E.P. 150,595, 5.1.20.

SEE U.S.P. 1,319,597 of 1920; preceding.

Electrodes for electric primary cells and storage batteries [; Tubular —], and process of making such electrodes. Svenska Akkumulator Aktiebolaget Jungner. E.P. 128,197, 23.5.19. Conv., 13.6.18.

Diaphragm for electrochemical and electro-osmotic purposes. B. Schwerin, Assr. to Chemical Foundation, Inc. U.S.P. 1,352,763, 11.9.20. Appl., 16.5.11.

SEE G.P. 271,001 of 1913; J., 1914, 427.

See also pages (A) 711, *Precipitating particles from gases* (E.P. 118,659 and U.S.P. 1,315,790); 715, *Carbon and gaseous fuel* (U.S.P. 1,352,085-6); 716, *Electrochemical treatment of hydrocarbons* (U.S.P. 1,315,131), *Treating mineral oils* (U.S.P. 1,354,257); 722, *Alkali melts* (U.S.P. 1,354,451); 721, *Electrodes for welding* (E.P. 117,270 and 118,645).

XII.—FATS; OILS; WAXES.

Oil and fat preparations [Turkey-red oil etc.]; Determination of ammonia in —. N. Welwart. Chem.-Zeit., 1920, 44, 719.

To determine ammonia in Turkey-red oil and similar products in which free acidity has been neutralised with ammonia, the sample is mixed with water and an excess of calcium chloride solution, a few pieces of pumice-stone and a sufficient quantity of sodium hydroxide are added, and the mixture is distilled, the ammonia being collected in standardised acid. The calcium soaps formed do not froth during the distillation. Light mineral oils, if present, may pass over into the distillate, but do not interfere with the determination. Methods which depend on the extraction of the ammonia by shaking the oil with dilute acid and subsequent distillation of the acid extracts with sodium hydroxide are not always trustworthy, since certain sulphonated oils contain the ammonia in such combination that it is not extracted even by boiling with acidified water.—W. P. S.

Oxidation of paraffin wax. Kelber. See IIa.

PATENTS.

Oils; Process and apparatus for hydrogenating —. J. S. Withers. From National Electro-Products, Ltd. E.P. 150,802, 5.6.19.

A tube enclosing a shorter concentric tube passes through the top and bottom of a cylindrical tank, and extends for some distance below the bottom of the tank. Two sets of openings are provided in the outer tube, near the top and bottom of the tank, the openings near the top being adjustable. A mixture of oil and catalyst is fed into the tank near the bottom, and hydrogen is pumped through a pipe passing in at the lower end of the outer tube and extending into the inner tube. The temperature is maintained at 200°–250° C. by an electric coil situated just above the hydrogen inlet, and the pressure at about 80 lb. per sq. in. The stream of hydrogen forces the oil upwards through the inner tube; residual hydrogen is drawn off at the top of the tube and is returned to the hydrogen inlet, and the oil flows through the upper set of openings into the tank, fresh oil flowing into the annular space through the lower set. The hydrogenated oil passes out at the top of the tank and flows around the inlet tube to preheat the fresh oil supply.—L. A. C.

Lanolin; Method of making —. W. C. Broadgate, Assr. to F. W. Harmon. U.S.P. 1,354,376, 28.9.20. Appl., 26.3.19.

AN alkali peroxide is added to wool grease which is heated to produce a thin, smooth product, and the mixture, after agitation, is allowed to stand until the grease is bleached. The lanolin is then separated by centrifuging.—L. A. C.

Oils and fats; Deodorisation of —. W. Alexander. G.P. 322,347, 4.4.19.

THE oil or fat is allowed to flow downwards through a tower packed with inert material, whilst steam is passed upwards through the tower.—J. H. L.

Fat and oil; Countercurrent process for extracting — from raw materials. H. Bollmann. G.P. 322,446, 30.5.18. Addn. to 303,846 (J., 1920, 459 A).

THE material, e.g., cereal embryos, in containers, is moved first downwards and then upwards through the chamber, and the solvent is led through the material in the opposite direction to the latter during its upward travel and in the same direction as the material during its downward travel.

—J. H. L.

Mixing gases with liquids or solids. E.P. 129,978. See I.

Fat from bones. G.P. 322,459. See XIXA.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Colour classification; Practical application of Ostwald's scheme of —. A. Wirth. Chem.-Zeit., 1920, 44, 737—738.

AN apparatus is described for determining the black, white, and coloured constituents in pigments in accordance with Ostwald's scheme of classification into 680 colours (J., 1919, 914 A). As a rule the colours of oil pigments can be readily matched, but the proportion of white is generally less, and that of the black higher, than the calculated amounts. Difficulties are only experienced with pigments containing less than 2% of colour. By the use of the five colour tones in Ostwald's diagrammatic circle, and the addition of suitable proportions of black and white, any of the other colour tones may be obtained in a state of sufficient purity.—C. A. M.

PATENTS.

Potentially-reactive [resinous] composition and process of making same. L. H. Baekeland, Assr. to General Bakelite Co. U.S.P. 1,354,154, 28.9.20. Appl., 31.10.19.

THE composition contains a phenol resin and a methyleneamine-phenol.—L. A. C.

Paint vehicles; Manufacture of — from mineral oils. J. Soukup. G.P. 321,828, 1.11.18. Conv., 15.5.18.

VASELINE, purified and dehydrated by heating with slaked lime, is heated with an oxidising agent such as manganese dioxide or lead peroxide, boiled with resin, and, after standing, is diluted and separated from any sediment.—L. A. C.

Varnish; Manufacture of — from unsaponifiable coumarone resins without use of benzol as solvent. W. Schwarz. G.P. 322,802, 14.6.18.

COUMARONE resin is purified by treatment with sulphuric acid, and the hot mixture is treated with air or oxygen under pressure. The resinous product is emulsified with water at 70°—100° C., and

a solution of glue and potassium bichromate is added to prevent separation of the emulsion.

—L. A. C.

Coating material. H. H. Robertson Co., Assees. of J. H. Young. E.P. 132,522, 11.9.19. Conv., 11.9.18.

SEE U.S.P. 1,327,933 of 1920; J., 1920, 227 A.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rubber; Methods of preparation of plantation —. Causes of the inferiority of plantation scrap rubber. Third report of the Ceylon Rubber Research Scheme. Indiarubber J., 1920, 60, 673—674.

SCRAP rubbers generally give low values for ultimate tensile strength and elongation, the inferiority being due to the presence of foreign matter not removed by washing; the mineral constituents, particularly the particles of sand, are mainly responsible. Investigations of the effect of smoking the rubber, of different methods of drying, and of various modes of preparation confirm those of earlier workers.—D. F. T.

Rubber; Determination of true free sulphur and the true coefficient of vulcanisation in vulcanised —. W. J. Kelly. J. Ind. Eng. Chem., 1920, 12, 875—878.

ONE g. of the ground rubber is extracted continuously with acetone for 16 hrs., the acetone solution is evaporated, the residue dried at 65° C. for 30 mins., and then treated in a flask with 50 c.c. of 95% alcohol saturated previously with sulphur. The flask and its contents are weighed, heated to 60° C., cooled, and any loss in weight adjusted by the addition of alcohol. After 24 hrs. the solution is decanted, the insoluble residue of sulphur washed with sulphur-saturated alcohol, dried, and weighed. Sulphur which is combined with resins, proteins, etc., and which is soluble together with the free sulphur in acetone, remains soluble in the alcohol, whilst the true free sulphur is insoluble in the latter solvent. The sulphur which is not extracted by acetone is not wholly combined with rubber, a part being also present in combination with resins and proteins; the sulphur thus combined should not be included in the value for the coefficient of vulcanisation. If the sample (1 g.) after extraction with acetone for 16 hrs. is dried, boiled with 5% alcoholic potassium hydroxide solution for 8 hrs., and then extracted with alcohol for 16 hrs., the alcoholic (alkaline and neutral) extracts will contain the sulphur which is combined other than with rubber. The quantity of this form of sulphur is ascertained by evaporating the united alcoholic extracts, oxidising the residue, and precipitating the sulphuric acid as barium sulphate.—W. P. S.

PATENTS.

Vulcanised product; Production of a soft, elastic, and ductile —. Production of a ductile, tenacious, and elastic vulcanised product. Farbenfabr. vorm. F. Bayer und Co. G.P. (A) 303,984, and (B) 305,667, 20.2.17.

(A) AMMONIUM compounds, aliphatic or aromatic amines, or their derivatives or salts, such as diphenylamine, diethylaniline, formamide, dimethylaniline, dimethyltoluidine, to the extent of 5% or more, and/or (B) aniline sulphate, which in small quantities acts as an accelerator, are added to the materials during vulcanisation.—L. A. C.

Vulcanised india-rubber goods embodying a foundation fabric; Treatment of —. W. E. Muntz. U.S.P. 1,354,123, 28.9.20. Appl., 21.1.15.

SEE E.P. 3158 of 1914; J., 1915, 672.

XV.—LEATHER; BONE; HORN; GLUE.

Tannins. Y. *Phloroglucinol-tannins and catechins. Constitution of gambier catechin.* K. Freudenberg. Ber., 1920, 53, 1416—1427.

GAMBIER catechin has been considered by Kostanecki to be a derivative of ethyldiphenylmethane, mainly on account of its reaction with diazonium salts. By the action of sodium and alcohol on its tetramethyl ether and subsequent methylation, a crystalline pentamethyl ether is produced which Kostanecki regarded as 3-ethyl-2,4,6-trimethoxyphenyl-3',4'-dimethoxyphenylmethane, but which is now shown to be 2,4,6,3',4'-pentamethoxy- α -diphenylpropane. Hence, like the nearly related flavone dyes, anthocyanidins and phenyl styryl ketones, gambier catechin must have an α -diphenylpropane skeleton. (Cf. J.C.S., Nov.)—H. W.

Tannins; Electro-chemistry of the —. G. Grasser. Collegium, 1920, 17—29, 49—69, 137—152, 200—207, 277—290, 332—341.

ELECTRO-OSMOTIC experiments showed that most tannins have a negative charge, as shown by anodic migration. In some cases the migration was sluggish or indefinite, and normal cataphoresis was only obtained after adding acids, preferably acetic acid. The only exception was quebracho, which migrated to the cathode. The presence of cations prevents tannin migration. The whole solute migrates in the case of galls, pine bark, maletto, and tannic acid in acetic acid, quebracho in sodium hydroxide, quebracho-calcium compound, quebracho-methylene blue compound, and mixtures of quebracho with sulphite-cellulose extract and neradol D. In several instances there is simultaneous decomposition of the tannins, especially in presence of electrolytes. Mangrove and catechu were found to be sodium salts of the fundamental tannins or tannin dyestuffs, hence their feeble tanning action, dark colour, and good solubility. From both there was obtained at the anode a brown base-free precipitate which was redissolved on addition of alkali. Electro-osmosis of soluble tannins generally results in decomposition, with the formation of soluble products which separate at the anode or cathode, or accumulate in the residue of the treated solution. Decomposition is frequently indicated by the qualitative chemical properties which differ from those of the original tannin: in some cases products are formed which do not precipitate gelatin. In most cases the electric current produces insoluble compounds which are sometimes soluble, but usually insoluble, in alkali, and which are sometimes formed so quickly—e.g., with oak bark—that it is impossible to obtain a dialysate containing any dissolved matter. Electro-osmosis can be employed to separate two qualitatively different tannin substances from a tanning material; this was effected in the case of valonia and oak bark. From knopperrn, on the other hand, two tannin substances were separated, of which one possessed no astringent properties. The separation of substances which no longer gave the tannin reaction but functioned as dyestuffs similar to indicators was effected with catechu, mangrove, and particularly with maletto. The purification and isolation of tannins from their extracts or adsorption compounds does not appear possible by the aid of electro-osmosis owing to the secondary changes. The detection of mangrove and quebracho in other extracts, and of sulphite-cellulose, neradol D, sulphite, bisulphite, and sugar by electro-osmosis was found possible. The experimental results show that the passage of an electric current through tan liquors should facilitate tanning, and there would seem to be special advantages in using weak tan liquors.—D. W.

Synthetic tannins. G. Grasser. Collegium, 1920, 234—255.

THE precipitation of gelatin and the absorption of a substance by hide powder are not necessarily accompanied by tanning properties. Thus, hydroxynaphthylmethanesulphonic acid precipitates gelatin, but it has no tanning action on pelt. Sodium diresylmethanedisulphonate does not precipitate gelatin nor tan pelt, but it is absorbed like tannin by hide powder. *o*-Nitrophenol does not precipitate gelatin, but it has a tanning action on both pelt and hide powder. All mono- and polyhydric phenols, after conversion into soluble sulphonic acids, can be converted into true tanning substances by condensation with substances capable of abstracting the elements of water. The condensation can be effected first and sulphonation afterwards, or *vice versa*. Alkaline solutions of phenol can be similarly condensed with materials capable of abstracting the elements of water, with formation of soluble products. Among the substituted phenols thio-, bromo-, chloro-, nitro-, and amino-derivatives give very similar tannins. Quinones are tanning materials. Aromatic di-alcohols condense very readily and give good tannins. All the aromatic acids appear to give soluble tannins of technical value after condensation and sulphonation; in some cases the soluble alkali salt must be condensed with formaldehyde to obtain a tanning material. Diphenyl derivatives and compounds containing condensed nuclei usually possess tanning properties if they contain the above-mentioned groups. The condensation can always be effected by means of formaldehyde; in a few cases with acetaldehyde and benzaldehyde. Other dehydrating agents, such as sulphur chloride, and phosphorus compounds generally effect condensation very readily, but secondary reactions impart a bad colour to the products, rendering them unsuitable for tanning purposes.—D. W.

Artificial tannins; Detection of —. R. Lauffmann. Collegium, 1920, 169—171.

THE indophenol reaction (Seel and Sander, J., 1916, 1028) is not a sure guide to the detection of synthetic tannins, but negative results with it prove their absence. The best method of carrying out the test is as follows:—10 g. of finely divided leather is treated with 100 c.c. of 2% caustic soda solution for 1 hr. with occasional shaking, the liquor filtered, and the leather residue treated with a further 100 c.c. of 2% alkali for 1 hr., filtered off, and the solution added to the previous filtrate. The liquid is neutralised with sulphuric acid, evaporated to dryness, and the dry residue heated with 20 c.c. of caustic potash solution (1:1). The product is treated with dilute sulphuric acid, nearly neutralised with sodium hydroxide, 100 c.c. of 10% ammonia solution added, and the whole allowed to cool. The liquid is extracted with ether, and after evaporating the ether the extract is dissolved in alcohol and treated with a few drops of concentrated ammonia, 2 drops of 0.6—0.7% aqueous solution of *p*-phenylenediamine hydrochloride, and 5% potassium ferricyanide solution. A positive indophenol reaction is shown by a blue or greenish-blue colour.—D. W.

Quebracho tannin. W. Moeller. Collegium, 1920, 106—109.

THE peculiar properties of quebracho extracts are due to the presence of predominating quantities of peptonised matter, or phlobaphenes. The extracts are derived from the heart wood. The sapwood differs from this in colour and composition, containing tans 6.4%, non-tans 2.2%, insoluble matter 76.4%, water 15.0%. The ratio of tans to non-tans is only 3:1, whereas in the heart wood it is 10:1, with a tannin content of 20%. The tannins in the

sapwood partake more of the nature of pyrogallotans. There are certain brighter parts in the heart wood which have evidently been protected from the oxidising and condensing influences of sap. These portions contain tans 7.8%, non-tans 1.7%, insoluble matter 77.1%, and water 13.4%, and give a dirty-blue colour with iron alum, a precipitate with bromine water, and a distinct precipitate with lead acetate and acetic acid; the ethyl acetate number is 54.24. Almost all these reactions and analytical constants differ from those usually associated with quebracho wood.—D. W.

Tannin analysis; Proteolytic factor in —. W. Moeller. *Collegium*, 1920, 307—319, 374—381.

Some substances which are not absorbed by hide powder alone are absorbed by hide powder from mixtures. Hide powder or pelt is considered to consist of an intact portion of micells and an easily decomposed portion of interfibrillary tissue which may be present in all stages of decomposition down to amino-acids. Hence a portion of the hide powder will be dissolved by water, and the author has determined this by weighing and by nitrogen estimations. The nitrogen content of the aqueous extract is an index of the amount of solubilised hide substance. Determinations with a large number of tanning materials have shown that large amounts of hydrolysed hide powder pass into the non-tan solutions in both the shake and filter bell methods of tannin analysis and adversely affect the tannin estimation. An adsorbent is required which will be resistant to the hydrolytic effects of the different tannins. Light chroming of the powder does not reduce the amount of hydrolysis, and either a stronger preliminary tannage with vegetable or mineral tanning agents should be given or some mode of preservation resorted to. Parker has suggested the use of formaldehyde, but this is no better than other tanning solutions. This hydrolysis is part of the tanning process and must be taken into consideration in the valuation, analysis, and purchase of tanning materials. The hydrolytic effects of different tannins at various concentrations over long periods of time on the pelt are not comparable with the effect of very dilute solutions on hide powder. In leather manufacture the tanning liquors are always more saturated than the pelt, whereas in tannin analyses the hide powder is always in great excess and completely detannises the solution. The amounts of hide substance found in the non-tans in both the shake and filter bell methods are on the average between those found in the blank determinations with unchromed and chromed hide powder.—D. W.

Pelt analysis. [Estimation of lime.] G. Grasser. *Collegium*, 1920, 80—81.

The damp pelt is pressed between dry cloth, cut into strips, and 5 g. heated with 3 c.c. of concentrated nitric acid and 50 c.c. of distilled water in a large covered beaker. In 10 mins. the pelt dissolves to a clear solution which is neutralised with ammonia and the calcium precipitated with ammonium oxalate. The calcium oxalate is ignited and the calcium determined as oxide.—D. W.

Aldehyde tannage; Formaldehyde titration of hide powder and —. O. Gerngross. *Collegium*, 1920, 2—11.

The author confirms Stiasny's statement (J., 1908, 113, 580) that hide powder which has been treated with formaldehyde adsorbs less acid than untreated hide powder. The diminution in the acid adsorbed shows no regularity with different acids, and it is concluded that the formation of acid from formaldehyde, as suggested by Moeller (*Collegium*, 1919,

270), does not take place. With increased hydron concentration the reaction between the hide powder and the formaldehyde is suppressed. The decreased acid adsorption of hide powder which has been treated with formaldehyde is not a proof of the presence of free amino groups, but only establishes the presence in collagen of basic groups capable of reacting with formaldehyde. Moeller (J., 1920, 36 A) claims that the hide fibres in aldehyde-tanned leather are surrounded by a sheath of formaldehyde polymers, which prevent the fibres from reacting with acid. If this were the case the hide fibres should likewise be protected from alkalis and show decreased alkali adsorption, whereas experiments show a slight increase in accordance with Stiasny's views as to the chemical nature of aldehyde-tannage (*loc. cit.*).—D. W.

Aldehyde tannage. W. Moeller. *Collegium*, 1920, 185—199. (See also J., 1920, 36 A.)

A REPLY to Gerngross (preceding abstract), in which the author affirms that the aldehyde combines with the hide decomposition products. The products of this reaction cannot tan the hide or render it insoluble in water, since they are readily soluble in water. The tanning agent consists of the insoluble polymers and condensation products formed from the excess of formaldehyde which form a protective covering round the fibres.—D. W.

Hydrolysis and adsorption; Relation between — [by hide powder]. I. W. Moeller. *Collegium*, 1920, 109—125, 152—166, 209—223, 267—276.

THE action of acids on hide powder is first one of hydrolysis, which proceeds much more quickly and strongly with inorganic than with organic acids. The presence of neutral salts appears to check the hydrolysis at first and then suddenly facilitates it. Besides the hydrolysis adsorption takes place between the non-hydrolysed protein micells, the products of hydrolysis, combined and uncombined with acid ions, and ionised and un-ionised protein molecules. The swelling is due to the increase in volume of the micellar strands caused by the adsorption of hydrated protein ions by the unattacked collagen micells in the hide substance. By the addition of neutral salts the hydrated hydrolysed collagen ions in the solution are dehydrated and no swelling can take place, although there may be subsequent adsorption. The effect of ampholytes on the hide powder is to restrict the hydrolysis. The apparent acidity of hide powder which has been in contact with acid for an hour and is then titrated in presence of different indicators differs from the actual acidity and forms no guide to the true conditions of the processes going on. With gelatin very different results are obtained owing to structural and chemical differences between gelatin and hide powder.—D. W.

Hydrolysis and adsorption; Relation between — [by hide powder]. II. W. Moeller. *Collegium*, 1920, 319—331, 382—387.

THE action of acids and alkalis on proteins, which is so frequently represented as being parallel to the adsorption process in dyeing and tanning, is quite different and cannot be cited as evidence for the chemical theory of tanning. The substances used in dyeing and tanning are either neutral or so feebly acid that hydrolytic effects on the animal fibres are either excluded or very much repressed.—D. W.

Nitrogen in leather; Rapid estimation of —. F. G. A. Enna. *Collegium*, 1920, 207—209.

One gm. of finely divided leather is heated with 8—10 g. of potassium sulphate and 20 c.c. of pure

concentrated sulphuric acid in a Kjeldahl flask over a large flame. After 10 mins. a small drop of mercury is added and the heating continued until the mixture becomes a very pale yellow. The flask is cooled, water added, followed by potassium sulphide and caustic soda solutions, and the ammonia is distilled into a saturated solution of boric acid. The determination can be completed in 2 hrs.—D. W.

Kjeldahl estimation of nitrogen [in leather]; Sources of error in the — and a modified method of expelling the ammonia. J. Kahn. *Collegium*, 1920, 367—371.

The chief sources of error are the irregularity of the ground leather sample, insufficient decomposition of the nitrogenous matter, the carrying over as spray of alkali, sodium sulphide, and even zinc particles in the ammonia distillation, and the escape of some of the ammonia which is unabsorbed. The modified method consists in fitting an Erlenmeyer flask with a dropping funnel containing the excess alkali protected from the air by means of a soda-lime tube fitted into the top of the funnel. A delivery tube from the flask leads to two wash bottles, one containing $N/1$ acid and the other water. The second bottle is attached to a filter pump. The solution for distillation is transferred to the Erlenmeyer flask which is placed in a water bath at $50^{\circ}C$. and gradually heated to boiling. Air is drawn through the apparatus for $1\frac{1}{2}$ —2 hrs. The alkali and sodium sulphide are gradually run in at the commencement from the dropping funnel.—D. W.

Free sulphuric acid in leather and animal hide; Determination and behaviour of —. C. Immerheiser. *Collegium*, 1920, 360—367.

A REPLY to Moeller's criticisms (J., 1919, 227 A). Leather treated with known amounts of sulphuric acid when analysed by the author's method has given an amount equal to that absorbed. The method is not influenced by any chlorides present. Experiments have shown the great reactivity of leather and hide for free acid, that of the latter being diminished by tanning in proportion to the acidity of the tanning agent employed. It is advisable to determine the nitrogen content of the aqueous extract of leathers under examination for harmful ingredients to determine whether the acid has decomposed the tissue.—D. W.

Gelatins and glues; Determination of the jellifying power of — by means of the polariscope. C. R. Smith. *J. Ind. Eng. Chem.*, 1920, 12, 878—881.

GELATINS of the highest jelly strength approximate to a definite and maximum value of mutarotation between 36° and $15^{\circ}C$. (cf. J., 1919, 228 A), and the polarimetric examination of gelatin in conjunction with certain other physical tests gives an indication of the relation of time, concentration, and temperature in the development of jelly strength. The smallest amounts to produce a standard jelly are required in the case of gelatins showing the greatest mutarotation at a concentration of 3 g. per 100 c.c.; as the mutarotation decreases, the amounts required to produce the standard jelly increase. For a gelatin which polarises $-20.5^{\circ}V$. at $35^{\circ}C$. and $-10.0^{\circ}V$. at $15^{\circ}C$., in a 200 mm. tube, it is suggested that the jelly strength be expressed as 19.5 points (i.e., the increment in rotation in Ventzke degrees); the maximum strength was found to be 25 points.—W. P. S.

Tannic acid from lignosulphonic acids. Hönig and Fuchs. See V.

PATENTS.

Leather; Method of drying varnished (patent) —. H. Schulz, Assr. to C. Heyl. U.S.P. 1,353,937, 28.9.20. Appl. 12.12.16.

SEE E.P. 113,622 of 1918; J., 1918, 431 A.

Glue from bones. G.P. 322,159. See XIXA.

XVI.—SOILS; FERTILISERS.

Fertiliser prepared by treating lime with end-liquors from potash works; Composition of —. J. Görbing. *Z. öffentl. Chem.*, 1920, 26, 205—213.

LIME fertilisers, prepared by sprinkling burned lime with potash end-liquors containing about 30% of magnesium chloride, are of complex composition, the assumption that they are merely mixtures of calcium hydroxide and magnesium chloride, with traces of magnesium hydroxide and calcium chloride, being erroneous. A sample examined consisted of a mass of damp, crumbly portions interspersed with hard lumps. An average sample had the following composition: Moisture (at $120^{\circ}C$), 30.41; CaO, 27.49; MgO, 11.37; K_2O , 0.58; Na_2O , 0.42; Fe_2O_3 and Al_2O_3 , 1.18; SiO_2 (sol.), 1.24; SiO_2 (insol.), 0.66; Cl, 11.87; CO_2 , 4.08; SO_3 , 1.76; P_2O_5 , Br. and organic impurities, traces; water of crystallisation and H_2O combined with lime and magnesium hydroxide (by difference), 7.83. A sample taken from the hard lumps gave: Moisture (at $120^{\circ}C$), 31.86; CaO, 36.63; MgO, 5.91; Cl, 4.89; CO_2 , 1.85. This sample gave a dry, non-hygroscopic powder which implies presence of oxychlorides. The decomposition of these in the soil is dependent on conditions of time, temperature, and concentration. A laboratory sample of the fertiliser treated with water on a filter gave a solution containing: CaO, 15.12; Cl, 11.75; K_2O , 0.60; Na_2O , 0.39; SiO_2 (sol.), 0.42; SO_3 , 1.39; with traces only of MgO, thus confirming the absence of magnesium chloride from the original material. The residue contained CaO, 12.4; MgO, 11.4%, and was of a light, pulverulent nature, and this condition was also produced with material exposed in the open for about six months, owing to atmospheric influences liberating the magnesia, which in the original material is present in a very finely divided condition and enclosed by calcium chloride. The liberated magnesia is in a very suitable form for distribution throughout the soil. Some photo-micrographs are appended.—W. J. W.

Humic acids. Eller and Koch. See III.

Ripening of wheat. Rousseaux and Sirot. See XIXA.

XVII.—SUGARS; STARCHES; GUMS.

Dextrose; Mutarotation of — in alkaline solution. H. Murschhauser. *Biochem. Zeits.*, 1920, 166, 23—36.

THE author has measured the rate at which the mutarotation of dextrose disappears in distilled water and in $N/10$ — $N/7000$ sodium carbonate solutions at $20.4^{\circ}C$. The values of the velocity constant of the change of mutarotation for different concentrations of sodium carbonate are: water, 7.11; $N/7000$, 7.32; $N/6000$, 9.32; $N/1500$, 12.70; $N/2000$, 24.4; $N/1000$, 56.5, and $N/500$, 93.1; these values give nearly a straight line when plotted against the concentrations of alkali.—T. H. P.

Maltose; Preparation of — [from starch]. M. Falch. Z. ges. Brauw., 1920, 43, 281—283, 289—291, 297—299, 306—309.

THE following method gives a yield of 30 pts. of pure maltose per 100 pts. of air-dry starch:—300 g. of potato starch is mixed with 1 l. of water and 30 c.c. of malt extract (made by digesting 1 pt. of pale kilned malt or green malt with 4 pts. of water for 3 hrs.), and the mixture is poured quickly into 2 l. of boiling water, with stirring, whereupon the starch is rapidly liquefied. After the temperature has fallen to 60° C., 30 c.c. of malt extract is added and the temperature is maintained for 2 hrs. after the liquid has ceased to react with iodine. The liquid is then heated to boiling, filtered, diluted to 7° Balling (sp. gr. 1.028), treated with 240 c.c. of malt extract, and with 7 mg. of hydrofluoric acid per 100 c.c., and maintained at 30° C. in a stoppered vessel for 3 days, during which no turbidity should appear. After addition of calcium carbonate the liquid is filtered, heated to boiling, evaporated to a water-content of 30%, preferably *in vacuo*, and then seeded with maltose crystals and maintained at 40° C. If crystallisation is very slow 3 separate quantities of 70 c.c. of 90% alcohol are added at intervals of 1 day. The crystals are collected with the aid of a pump, then mixed with 90% alcohol and again collected. 100 g. of the pressed product is dissolved in 30 c.c. of water on a water-bath, mixed with 260 c.c. of 90% alcohol, heated to boiling, and filtered. After cooling and seeding with maltose crystals, the liquid deposits pure maltose.—J. H. L.

PATENTS.

Sugar process. R. E. Pospisil and E. Kurek. U.S.P. 1,352,084, 7.9.20. Appl., 27.12.15.

WHITE granulated sugar is manufactured by treating raw juice to recover the first strike of sugar, boiling the syrup therefrom, together with a part of the dark sugar from a later massecuite, in a vacuum pan, subjecting the resulting sugar to water injections and raising the temperature to remove impurities, and re-melting the crystals and boiling to massecuite for white sugar.—J. H. L.

Explosives from carbohydrates. G.P. 298,949. See XXII.

XVIII.—FERMENTATION INDUSTRIES.

Fermentation in closed and open vats. E. Lühder. Z. Spiritusind., 1920, 43, 276, 283—284.

In distillery fermentations in large closed iron vats the temperature rises more rapidly during the early stages of fermentation and falls more rapidly during the later stages than in small open wooden vats. This difference cannot be altogether accounted for on physical grounds, e.g., loss of heat by conduction or surface exposure. Comparative fermentations of a filtered mash of rye and barley malt were carried out under identical conditions in similar, half-filled glass vessels, some of which were open and others closed by a stopper with glass tube and water seal. It was found that during the first 24 hrs. yeast reproduction (ascertained by counting cells) was almost twice as great in the closed as in the open vessels, e.g., 17-fold compared with 9-fold; but after 48 hrs. it was practically the same in both cases (18-fold compared with 17-fold), and no further change occurred afterwards. The yield of alcohol was the same in both cases, apart from a small loss by evaporation from the open vessels.—J. H. L.

Yeast; Rhythmic phenomena in the growth and fermentation of —. E. Köhler. Biochem. Zeits., 1920, 106, 194—206.

THE processes of fermentation and of yeast-growth are found to exhibit alternating maxima and minima of intensity; this periodicity appears to be due to alterations in the sugar and alcohol contents of the nutrient medium. The velocity with which yeast grows is conditioned by the concentration of the sugar and varies irregularly as the concentration increases.—T. H. P.

Alcoholic fermentation by yeast. E. Köhler. Biochem. Zeits., 1920, 108, 235—244.

THE course of alcoholic fermentation by yeast becomes rhythmic with increase in the concentration of the alcohol.—S. S. Z.

Methyl alcoholic fermentation; So-called —. E. O. von Lippmann. Biochem. Zeits., 1920, 106, 236—238.

OF 20 samples of genuine rums from the Lesser Antilles, 2 were found to contain from 1 to 2.5% of methyl alcohol, 7 from 2.5 to 5%, 5 from 5 to 7.5%, and 3 from 7.5 to 8.5%. In the district named the cane is largely pressed and the juice, being inadequately clarified, contains considerable proportions of very fine cane fibre, which passes to a large extent into the syrups and molasses. This fibre is rich in pectinous material and as the pectins consist of the methyl ester of pectinic acid, which is readily decomposed in various ways with liberation of methyl alcohol, the origin of the latter is evident.—T. H. P.

Maltose from starch. Falch. See XVII.

Enriching straw fodder. Pringsheim and Lichtenstein. See XIXA.

PATENTS.

Diastatic composition and process of making the same. J. Takamine. E.P. 131,579, 9.5.19. Conv., 20.8.18.

A DIASTATIC product is prepared by neutralising a diastatic extract, preferably with sodium bicarbonate, then adding a precipitant, such as magnesium or calcium chloride, and separating the precipitate. Claim is also made to a mixture of this precipitate with maize starch, sugar, and sodium phosphate, and to a process of bread making in which this mixture is employed without any further addition of sugar or malt extract and with less than the usual quantity of yeast.—J. H. L.

Acetone and butyl alcohol; Fermentation processes for production of —. C. Weizmann. E.P. 150,360, 25.5.17.

AMYLACEOUS materials of a nutty character, containing saponins, e.g., horse-chestnuts preferably freed from husk, are cooked under pressure, and the mash obtained is treated with malt and maintained at 60°—70° C. for some hours, after which it is cooled to 37° C. and fermented in accordance with E.P. 4845/15 and 9181/16 (J., 1919, 301A). The treatment with malt greatly reduces frothing during fermentation.—J. H. L.

Extracting alcohol from liquids; Process and apparatus for —. F. Fehr. U.S.P. 1,345,437, 6.7.20. Appl., 22.11.16.

THE liquid is heated in a closed tank to a temperature sufficient to volatilise the alcohol, without precipitating albuminoids or producing other changes in the character of the organic constituents of the liquid. An exhaust current is created from the upper end of the tank, so as to maintain a high vacuum and draw off the vola-

utilised alcohol, the upper end of the tank being heated to prevent premature condensation of the alcohol vapour. The liquid is circulated by drawing it off from the lower portion of the tank and discharging it in the form of spray into the upper portion above the body of the liquid. Air is drawn by the vacuum into the body of liquid in the tank, and into the circulating current of liquid prior to its encountering the spraying device, whereby a very fine spray is produced.—B. N.

XIXA.—FOODS.

Wheat; Nitrogenous substances and phosphoric acid in the ripening and germination of —. E. Rousseaux and Sirot. Comptes rend., 1920, 171, 578—580.

SAMPLES were examined taken from the time of the formation of grain on through harvest and from ears in the stook which had germinated owing to rain after the cutting. It was found that the total nitrogen and phosphoric acid of the grain vary little during this period. The proportion of the total nitrogen which is soluble diminishes rapidly, however, as the grain fills out and until the commencement of ripening, when it rises slightly and remains constant until the germination in the stook takes place. The same is practically true of the proportion of soluble phosphoric acid. During ripening the acidity of the grain diminishes steadily but increases again when the grain germinates.—W. G.

Wheat flour milling; Laboratory control of —. B. R. Jacobs and O. S. Rask. J. Ind. Eng. Chem., 1920, 12, 899—903.

THE inner endosperm, or true flour-producing portion of the wheat grain contains all the starch (the starch content is about 70% of the air-dried substance) and only very small quantities of fat (0.75%) and pentosans (2.25%); the bran tissue, including the aleurone layer, contains most of the pentosans, 5—6% of fat, and no starch, whilst the germ contains 12.5% of fat, no pentosans, and no starch. The pentosan content of offal tissue is fairly constant at 28.5%. The amount of fat and pentosans in a flour is, therefore, a measure of the amount of offal present; the percentage of starch is a measure of the quantity of inner endosperm or true flour in a sample.

—W. P. S.

Blood; Preservation of —. C. Oppenheimer. Biochem. Zeits., 1920, 105, 145—151.

BLOOD to be dried for use as human food has often to be kept for some time prior to drying. In order to prevent it from undergoing change it must be treated with a preservative which should, for convenience, either not require to be removed subsequently or be removed during the process of atomising the blood. For this purpose the author recommends the use of the following mixture, the quantities given being sufficient for all the blood of an ox, that is about 10 l.: 83 g. of commercial pure sodium sulphite (or about 40 g. of the anhydrous salt) is dissolved in 0.5—1 l. of water and the solution treated with about 52 c.c. of 38% hydrochloric acid, or 18.2 c.c. of concentrated sulphuric acid, or 49.3 g. of tartaric acid, or 41.4 g. of oxalic acid.

—T. H. P.

Straw fodder; Enrichment of — with the proteins of moulds. H. Pringsheim and S. Lichtenstein. Cellulosechemie, Beiblatt zu Papierfabr., 1920, 1, 29—39. Chem. Zentr., 1920, 91, IV., 414—415.

In experiments with a non-pathogenic *Aspergillus*

species it was found possible to use ammonium chloride and a mixture of kainite and superphosphate instead of ammonium sulphate and potassium phosphate in the nutrient solution. The solution is poured over the straw only once at the commencement, and subsequent daily turning of the couch is unnecessary. The depth of the couch should not exceed about 30 cm., in order to avoid undue rise of temperature, which should be kept below 40° C. Aeration of the resting couch with moist air appears very beneficial. Products containing 8% of protein can be obtained.—J. H. L.

Yellow AB and Yellow OB. Mathewson. See IV.

PATENTS.

Evaporator and pasteuriser [for milk]. J. F. Ruff. U.S.P. 1,354,342, 28.9.20. Appl., 16.1.19. Renewed 26.7.20.

AN apparatus for evaporating and pasteurising milk and liquid milk products comprises a vat having a number of internal rotary heating drums, through which high-pressure steam is passed. The drums are provided with longitudinal cleaning bars bearing on the outer surface, and the bars are provided with cross connecting members, so that the cleaning means corresponding to a given longitudinal portion of the drums may be removed as a unit. The individual particles of milk are thus subjected to repeated momentary contacts with the drum and are immediately removed. Air is blown through the liquid to prevent the temperature from rising materially above that at which pasteurisation takes place.—W. F. F.

Bones; Production of feeding-stuff, fat, and glue from —. H. Goslar. G.P. 322,459, 16.2.17.

THE coarsely crushed bones are softened by a steaming process and triturated in warm water.

—J. H. L.

Diastatic composition. E.P. 131,579. See XVIII.

XIXB.—WATER PURIFICATION; SANITATION.

Water softener; Data on operation of continuous type lime soda-ash —. R. F. Catherman and H. C. Fisher. Chem. and Met. Eng., 1920, 23, 526—528.

IT was found that the plant could be effectively controlled by testing the treated water at intervals and adjusting the addition of reagents from the results of the tests, the best figures for the hardness, causticity, and alkalinity having been determined by a series of preliminary tests.

—W. H. C.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Alkaloids and their salts; Conductometric titration of —. I. M. Kolthoff. Z. anorg. Chem., 1920, 112, 196—208.

THE conductometric method of titration has been extended successfully to alkaloids and their salts in dilute solutions. For conductometric titrations of the alkaloid salts with alkali no alcohol need be used in most cases, but for acid titration of the alkaloid bases these are generally dissolved in 50% alcohol. The alkaloid salts tested included hydrochlorides of quinine, cocaine, tropacocaine, novocaine, morphine, and ethylmorphine, strychnine nitrate, and atropine sulphate. In the case of morphine hydrochloride no distinct break in the conductivity curve was found at the neutral point,

but since morphine has acidic properties, further titration with alkali gives a sharp break at the point where the formation of sodium morphinate is complete. For this titration 50% alcohol is used as solvent. The alkaloid bases examined included quinine, hydrastine, codeine, morphine, nicotine, theobromine, and caffeine. The last two have acidic properties, but since theobromine is a much stronger acid than caffeine, the former can be estimated in presence of the latter by dissolving the mixture in alkali and titrating with acid. The conductometric method may in certain cases be applied for the estimation of alkaloids in extracts, etc. Thus strychnine was successfully determined in a 10% extraction strychni solution.—E. H. R.

Tetranitromethane. II. Tetranitromethane as a nitrating agent. E. Schmidt and H. Fischer. Ber., 1920, 53, 1529—1537.

DECOMPOSITION of tetranitromethane into nitroform and nitric acid, which has been shown to occur almost exclusively, under the influence of very concentrated aqueous alkali, is found to take place in the presence of a weak base if solutions in acetone or alcohol are employed, the base becoming nitrated during the process and separating as its nitroform salt. Suitable non-basic substances can be similarly nitrated if the reaction is carried out in the presence of pyridine, when pyridine-nitroform is simultaneously produced. The process thus constitutes the first method of introducing the nitro-group into the aromatic nucleus in a non-acid medium. Its application to dimethyl-*p*-toluidine, diethyl-*p*-toluidine, dimethyl-*p*-anisidine, *p*-cresol, isosafrole, and anethole is described. (Cf. J.C.S., Nov.)—H. W.

Oxalic acid; Colour test for —. L. H. Chernoff. J. Amer. Chem. Soc., 1920, 42, 1784—1785.

A FEW crystals of resorcinol are dissolved in 5 c.c. of the solution by warming gently. The liquid is cooled and an equal volume of concentrated sulphuric acid slowly poured in to form a layer. A blue ring is formed at the junction of the layers if oxalic acid is present. If a blue colour does not appear the liquids are mixed by shaking and, after cooling somewhat, another 5 c.c. of sulphuric acid is added. If the colour still fails to appear the liquid is warmed over a flame (not boiled), when an indigo-blue colour appears. If the mixture is boiled the colour becomes dark-green. By warming the dry substance with 2 drops of a 10% resorcinol solution and adding sulphuric acid drop by drop, 1 mg. of oxalic acid can be detected.—J. R. P.

Aldehydes; Dehydrocoulation of —. Mechanism of oxidation. E. Müller. Annalen, 1920, 420, 241—261.

FORMALDEHYDE, acetaldehyde, and benzaldehyde are decomposed in alkaline solution either by electrochemical or purely chemical oxidation (by CuO , Cu_2O , Ag_2O) into acid and gaseous hydrogen. The latter is also evolved in small amount from alkaline solutions of formaldehyde in the presence of certain metals which also catalytically accelerate the Cannizzaro reaction. The primary product is considered to be an aldehyde peroxide. (Cf. J.C.S., Nov.)—H. W.

Ether apparatus and vagrant electric currents. O. Schaal. Chem.-Zeit., 1920, 44, 741.

To prevent the formation of electric currents and the consequent destruction of lead plant used in the manufacture of ether, all parts of the apparatus should be made of purest lead of the same quality and the plant connected with the earth in several places. Experiments have shown that weak currents (0.01 amp.) are generated in a mixture of sulphuric acid and alcohol by using hard lead as cathode and soft lead as anode.—C. A. M.

Phenols. Kolthoff. See III.

PATENTS.

Camphor; Recovery of — from gaseous mixtures. J. H. Bregeat. E.P. 150,651, 7.5.20.

GASES containing camphor, e.g., from the manufacture of celluloid etc., are passed through scrubbers moistened by a counter current of an absorbent consisting of phenol or its homologues, especially cresols. The camphor may subsequently be separated from the phenolic absorbent by the direct action of steam or by indirect heating, or by dissolving the phenols in alkali, in which case the phenols may be recovered for further use by acidification, preferably with an acid salt. (Cf. E.P. 127,309, 128,640, and 131,938; J., 1919, 521 A, 612 A, 751 A.)—J. H. L.

Acetaldehyde; [Apparatus for use in the] production of —. M. Soller and J. L. R. Hotz. E.P. 151,086, 14.6.19.

THE lead linings of the vessels used in the process described in E.P. 105,061 (J., 1918, 222 A) are protected from amalgamation with mercury by electrolytically coating the surface with a layer of lead peroxide. The vessel itself, filled with 5—10% sulphuric acid, is used as the anode, and a sheet of lead serves as cathode. A current of 100 amps. and 5 volts is passed through for about 10 hrs., and, after washing and drying the layer of lead peroxide, the process is repeated.—L. A. C.

Ethylidene diacetate; Manufacture of —. M. Beudet, Assr. to Soc. Chim. des Usines du Rhône, anc. Gilliard, P. Monnet et Cartier. U.S.P. 1,351,990, 7.9.20. Appl., 22.3.19.

CRUDE ethylidene diacetate, obtained by the action of acetylene on acetic acid in presence of mercury compounds, is decanted and diluted, and the resulting solution is agitated with an immiscible solvent of ethylidene diacetate; the latter is recovered by washing, drying, and distilling the extract.

—J. H. L.

Tropine; Homologue of — and process of producing same. E. Müller, Assr. to Hoffmann-La Roche Chemical Works. U.S.P. 1,352,082, 7.9.20. Appl., 16.12.16.

A HOMOLOGUE of tropine is produced by the action of sodium on dihydroanhydroecgonine alkyl ester dissolved in absolute alcohol. It is readily soluble in ether, benzene, alcohol, and water, slightly soluble in petroleum ether, crystallises from ligroin in long needles, m.p. 85° C., is dextrorotatory, and yields a hydrochloride, m.p. 210° C., soluble in water and alcohol and insoluble in ether, and a platinum compound and picrate slightly soluble in water.—J. H. L.

Tannate-lime compound difficultly soluble in dilute acids; Process for producing —. R. Tambach, Assr. to Chemical Foundation, Inc. U.S.P. 1,352,768, 14.9.20. Appl., 10.3.17.

SEE G.P. 307,857 of 1916; J., 1918, 749 A.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENT.

Colour photography. W. Friese-Greene and L. O'Malley. E.P. 150,819, 10.6.19.

THE negatives in a two-colour process of photography are produced by the simultaneous exposure of two sensitive coatings either on separate supports with the sensitive surfaces together or on opposite sides of the same support, the sensitive film nearer the lens being non-colour-sensitive and the other film being colour-sensitised by the process described in E.P. 134,238 (J., 1919, 963 A).

—B. V. S.

XXII.—EXPLOSIVES ; MATCHES.

Tetranitromethane as nitrating agent. Schmidt and Fischer. See XX.

PATENTS.

Explosives from carbohydrates; Production of —. Harburger Chem. Werke Schön und Co., and W. Daitz. G.P. 298,949, 9.3.16.

A SOLUTION of carbohydrates (sugar, starch, cellulose) is saturated with hydrogen with the aid of a catalyst, and after the removal of the latter the product is nitrated. The catalyst is prepared by treating a solution of nickel sulphate with an excess of magnesia. An alkaline reaction accelerates hydrogenation.—J. H. L.

Explosive which can be detonated by mercury fulminate and containing no nitric acid residue; Production of —. J. D. Riedel A.-G. G.P. 301,799, 24.12.14.

KIESELGUHR or similar material is impregnated with alkylchloramines, e.g., methylchloramine.—J. H. L.

Signal lights and fire balls; Manufacture of — free from metals and shellac. Geka-Werke Offenbach G. Krebs. G.P. 322,779, 24.3.18. Addn. to 314,868 (J.), 1920, 430 A).

STRONTIUM carbonate and potassium chlorate or perchlorate are used instead of strontium nitrate and barium peroxide, and the organic solvent is replaced by water, which evaporates on drying so that the binding of the materials depends solely on the softening of the pitch.—J. H. L.

XXIII.—ANALYSIS.

Phosphoric acid and its salts; Conductometric titration of —. I. M. Kolthoff. Z. anorg. Chem., 1920, 112, 165—171.

PHOSPHORIC acid, in common with other strong acids, cannot be titrated conductometrically in concentrated solution. In dilute solution it can be titrated in exactly the same way as mono- or dibasic acids. The first break in the curve, corresponding with primary salt formation, is sharp, the second less distinct, and the third very uncertain owing to the hydrolysis of tertiary phosphates. The conductometric method is superior to the indicator method for titrating dilute solutions of phosphoric acid, but, for more concentrated solutions, indicators are to be preferred. Secondary phosphates, e.g., Na_2HPO_4 , can be titrated conductometrically with acid when the concentration is less than 0.01 molar, but with greater concentration the break in the curve corresponding with NaH_2PO_4 is indistinct owing to the dissociation of phosphoric acid. Dilute solutions of tertiary pyrophosphates can be titrated satisfactorily with acid to the secondary salt.—E. H. R.

Ammonia; Determination of — in urine, in serous liquids, and in the oxidised liquid of the Kjeldahl method. A. Hahn and E. Kootz. Biochem. Zeits., 1920, 105, 220—228.

WITH Hahn's modification of the Krüger, Reich, and Schittenhelm method, all the ammonia is expelled in 5 min. from a solution containing 0.01 g. of ammonia as ammonium sulphate, 25 c.c. of water, 10 g. of sodium chloride, 1 g. of sodium carbonate, and 30 c.c. of 96% alcohol, if the distillation flask is heated in a boiling-water bath and the distillation is carried out in the maximum vacuum given by a water pump. Under these conditions urea (2 g.) yields no ammonia. Cooling of the receiver con-

taining the standard acid for absorbing the ammonia is unnecessary. Methods based on the above results are given for the determination of ammonia in albumin-free urine, albuminous urine, and blood serum.—T. H. P.

Carbonic acid and its salts; Conductometric determination of —. I. M. Kolthoff. Z. anorg. Chem., 1920, 112, 155—161.

SOLUTIONS of carbonic acid from 0.0015 to 0.02 molar in strength can be titrated conductometrically with alkali. The breaks in the curve, corresponding with bicarbonate and carbonate formation, are not very sharp, but their sharpness can be considerably increased by titrating in presence of a calcium salt, e.g., calcium chloride. Time must be allowed during the titration for the calcium carbonate to be precipitated. Carbonate solutions can be satisfactorily titrated with acids. The bicarbonate break in the conductivity curve is not sharp, but the neutral point is very sharp, even in extremely dilute solutions. Alkali can be determined in presence of carbonate by acid titration, whilst bicarbonate in carbonate can be titrated with acid or alkali. To obtain accurate results, however, the proportion of alkali or bicarbonate in the carbonate must not be too small, or the direction of the corresponding portion of the conductivity curve cannot be accurately determined. (Cf. J.C.S., Nov.)—E. H. R.

Tin; Volumetric determination of —. J. G. F. Druce. Chem. News, 1920, 121, 173—175.

TIN may be accurately determined by titrating stannous solutions with standard iodine solution in the presence of hydrochloric acid. Good results are also obtained by titration with potassium permanganate in the presence of sulphuric acid. (Cf. J.C.S., Nov.)—C. A. M.

See also pages (A) 713, *Sulphur forms in coal* (Powell). *Sp. gr. of coal etc.* (Hailstone); 717, *Phenols* (Kolthoff), *Yellow AB and Yellow OB* (Mathewson); 718, *Reducing value of cellulosic substances* (Kuecht and Thompson), *Cellulose in woods* (Mahood); 720, *Persulphuric acid* (Palme); 721, *Gold, silver, rubidium, and caesium* (Bayer, also Emich); 721, *Electrical conductivity* (Haughton); 727, *Ammonia in oil preparations* (Welwart); 728, *Sulphur in rubber* (Kelly); 729, *Artificial tannins* (Lauffmann); 730—1, *Tannin analysis* (Möcller), *Lime in pelts* (Grasser), *Nitrogen in leather* (Enna, also Kahn), *Sulphuric acid in leather* (Immerheiser), *Gelatins and glues* (Smith); 733, *Alkaloids* (Kolthoff); 734, *Oxalic acid* (Chernoff).

PATENT.

Hardness of metal parts; Ascertaining the —. E. Schneider. Assr. to Schneider et Cie. U.S.P. (A, B, C) 1,351,216—8, 28.9.20. Appl. (A) 21.8.18, (B, C) 16.6.19. (B, 4) Renewed 12.8.20.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

British Thomson-Houston Co. (General Electric Co.). Removing gases from solids. 29,309. Oct. 16.

Comp. Gén. d'Electricité. 29,075. *See* X.
Conover. Apparatus for bringing about reactions between gases. 29,641. Oct. 20. (U.S., 30,419.)

Duckham, and Woodall, Duckham, and Jones. Furnaces for producing chemical changes. 29,194 and 30,059. Oct. 14 and 23.

Duckham, Morgan, and Thermal Industrial and Chemical Research Co. Heating materials at successively different temperatures. 29,678. Oct. 20.

Fischer. Pyrogenic reactions. 29,911. Oct. 22. (Ger., 22.10.19.)

Foster. Apparatus for cleaning gases. 29,275. Oct. 16.

Koppers Co. Purification of liquids. 29,013. Oct. 13. (U.S., 15.2.19.)

Morgan, and Thermal Industrial and Chemical Research Co. Evaporation of liquids. 29,677. Oct. 20.

Norsk Hydro-Elektrisk Kvaelfstafaktieselskab. Impregnation of liquids. 30,052. Oct. 23. (Norway, 24.11.19.)

Pyzel. Distillation. 29,525. Oct. 19.

Soc. des Gaz Radioactifs Naturels de Colombières sur Orb. Cooling liquids and charging same with gas. 29,815. Oct. 21. (Fr., 22.10.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

13,137 (1918). Windram. Separating crystals from mother liquors containing more than one salt. (152,041.) Oct. 20.

2482 (1919). Lewis and Green. Distilling or recovering volatile inflammable liquids or solvents. (152,374.) Oct. 27.

17,113 (1919). Hall. Ball or tube mills. (152,398.) Oct. 27.

18,584 (1919). Reol. *See* X.

24,048 (1919). Imray (Secord, Peters, and Paterson). Drying apparatus. (152,505.) Oct. 27.

31,371 (1919). Norske Akt. for Elektrokemisk Industri. Manufacture of porous material. (136,818.) Oct. 20.

32,433 (1919). Henning. Solvents for extraction purposes. (152,550.) Oct. 27.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Asiatic Petroleum Co., and Cameron. Dehydrating emulsions and/or distilling oils etc. 29,821. Oct. 21.

Bamber and Parker. Gas-producers etc. 29,647. Oct. 20.

Bartolomeis, and Colombo & Ing. de Bartolomeis. Distillation of fuels. 29,365. Oct. 8. (Ital., 18.10.19.)

Coke Oven Construction Co., and Marr. Coke ovens. 29,412. Oct. 18.

Comerford. Fuel. 29,329. Oct. 18.

Davis and Twigg. Producer-gas plant. 29,557. Oct. 19.

Docking, and Sharp and Preston. Rotary retort furnaces. 30,011. Oct. 23.

Fabry. Regenerative coke ovens. 29,748. Oct. 21.

Farbenfabr. vorm. F. Bayer u. Co. 29,215. *See* VII.

Fischer. 29,911. *See* I.

Fonblanque and Moeller. Manufacture of illuminating gas and by-products. 29,250. Oct. 16.

Fonblanque and Moeller. Treatment of peat. 29,253. Oct. 16.

Foster. Gas for power purposes. 29,459. Oct. 19.

Gibson. Coal-saving composition. 28,958. Oct. 13.

Glover, West, and West's Gas Improvement Co. Vertical retorts for destructive distillation of carbonaceous material. 29,307. Oct. 16.

Hart. Production of carbon. 29,119. Oct. 14.

McEwen, and Underfeed Stoker Co. Carbonisation of subdivided fuel. 28,778. Oct. 11.

Marks (U.S. Industrial Alcohol Co.) Non-freezing fuel. 28,995. Oct. 13.

Strache. Combustion of bituminous fuels with recovery of by-products. 29,551. Oct. 19. (Austria, 6.8.19.)

Worthington. Utilising peat or turf for heating and for generating steam, electricity, or gas. 29,701. Oct. 21.

COMPLETE SPECIFICATIONS ACCEPTED.

1166 (1918). Litwin. Gas-producer furnaces for heating gas retorts. (152,369.) Oct. 27.

12,278 (1919). Dille. Suction gas plants. (152,380.) Oct. 27.

17,547 (1919). Roberts. Recovery of by-products of the destructive distillation of wood in suction gas plants. (152,420.) Oct. 27.

17,810 (1919). Wallace. Apparatus for recovering oil from shale or other carbonaceous materials. (129,996.) Oct. 20.

17,811 (1919). Wallace. Distilling carbonaceous materials. (129,997.) Oct. 20.

24,906 (1919). Long. Manufacture of fuel from peat. (152,515.) Oct. 27.

26,013 (1919). Siemens u. Co. Electric light carbons. (134,225.) Oct. 27.

30,379 (1919). Grisard. Neutralising mineral oils which have been treated with acid. (136,169.) Oct. 27.

32,220 (1919). Woodall, Duckham, and Jones, and Gardner. Vertical retorts for carbonising coal. (152,548.) Oct. 27.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

Atack. 28,692. *See* XX.

Johnson (Badische Anilin u. Soda Fabr.). Production of soluble condensation products. 28,749. Oct. 11.

COMPLETE SPECIFICATIONS ACCEPTED.

10,918 (1919). Stephenson. Dealing with acid sludge produced in purifying benzol, and recovering benzol and its homologues. (152,054.) Oct. 20.

17,849 (1919). Morgan, and British Dyestuffs Corp. Manufacture of 4-nitro- β -naphthol. (152,437.) Oct. 27.

20,217 (1919). South Metropolitan Gas Co., Evans, and Hollings. Purification of benzole or the like. (152,470.) Oct. 27.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

Cassella und Co., and Herz. Manufacture of dyestuffs. 29,096. Oct. 14.

Johnson (Badische Anilin u. Soda Fabr.). Manufacture of anthraquinone derivatives. 29,517. Oct. 19.

COMPLETE SPECIFICATION ACCEPTED.

3557 (1914). Newton (Bayer u. Co.). Manufacture of azo dyestuffs. Oct. 20.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

British Cellulose and Chemical Manuf. Co., and Palmer. Manufacture of textile products from cellulose derivatives. 29,629. Oct. 20.

British Cellulose and Chemical Manuf. Co., and Shuttleworth. Treatment of artificial threads. 29,630-1. Oct. 20.

Chadwick and Chadwick. Preparatory treatment of cotton etc. 29,873. Oct. 22.

Clotworthy. Treatment of artificial filaments or threads and fabrics containing same. 29,630. Oct. 20.

Edmunds. Treatment of jute waste. 29,751. Oct. 21.

Glanzfüden A.-G. Producing spun material resembling wool or cotton from viscose solutions. 28,982-1. Oct. 13. (Ger., 27.3, 6.4, and 15.8.18.)

Sandeman. Treatment of jute etc. 30,041. Oct. 23.

Tootall Broadhurst Lee Co., and Willows. Treatment of yarn and fabrics of cotton etc. 29,039. Oct. 14.

COMPLETE SPECIFICATIONS ACCEPTED.

24,788 (1919). Poole and Davis. Washing or scouring wool. (152,513.) Oct. 27.

706 (1920). Great Northern Paper Co. Method and machine for making paper. (137,539.) Oct. 27.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Blanc and Jourdan. Treating potassic rocks etc. to obtain constituents in soluble form. 29,235. Oct. 15.

Craig. Recovering potash etc. salts from blast furnace gases. 29,710. Oct. 21.

Davis and Levy. Generation of oxygen. 29,671. Oct. 20.

Farbenfabr. vorm. F. Bayer u. Co. Obtaining sulphur or treating and purifying gases containing hydrogen sulphide. 29,215. Oct. 15. (Ger., 30.10.19.)

Foster. Producing oxygen and agricultural lime. 29,029—29,030. Oct. 13.

Johnson (Badische Anilin u. Soda Fabr.). Extraction of sulphur. 28,895. Oct. 12.

Mathieson Alkali Works. Production of barium chloride. 29,882. Oct. 22. (U.S., 22.1.20.)

Schott u. Gen. Rendering boronatrocalcite soluble. 28,723. Oct. 11. (Ger., 24.10.19.)

Soc. l'Air Liquide. Catalytic materials for synthesis of ammonia. 29,850. Oct. 21. (Fr., 28.10.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

11,955 (1917). Wade (Deuts. Gold- u. Silber-Scheide-Anstalt). Manufacture of alkali percarbonates. (152,366.) Oct. 27.

15,045 (1919). Richards and Hutchinson. Recovery of soluble nitrogen compounds from solutions. (152,387.) Oct. 27.

17,246 (1919). Ashcroft. Production of anhydrous magnesium chloride or anhydrous double chlorides of magnesium. (152,401.) Oct. 27.

17,247 (1919). Ashcroft. Electrolytic decomposition of anhydrous magnesium chloride and production of magnesium and chlorine. (152,402.) Oct. 27.

18,255 (1919). Collins. Separating sulphur from gases obtained in roasting and smelting sulphur-bearing ores. (152,447.) Oct. 27.

26,317 (1919). Uchino. Refining arsenious acid. (152,526.) Oct. 27.

366 (1920). Toniolo, and Officine Elettrochimiche Dr. Rossi. Production of hydrogen. (152,554.) Oct. 27.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Bailey. Kilns for firing pottery etc. 29,700. Oct. 21.

Friedrich. 29,370. See X.

COMPLETE SPECIFICATION ACCEPTED.

24,383 (1919). Priest. See X.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Curtis. Cementitious compositions for flooring, paving, etc. 28,952. Oct. 13.

Jager. Production of artificial marble. 29,077. Oct. 14. (Holland, 14.10.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

16,817 (1919). Lesley. Utilising low-grade carboniferous material in the manufacture of Portland cement. (152,106.) Oct. 20.

17,638 (1919). Slade. Colouring wood grey or black. (152,427.) Oct. 27.

23,277 (1920). Boverouille. Manufacture of cement from slag. (149,988.) Oct. 20.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Abraham and Thain. Separating minerals etc. 29,475. Oct. 19.

Bengough. Coating metals with lead. 29,953. Oct. 22.

British Thomson-Houston Co. (General Electric Co.). Melting pots. 29,633. Oct. 20.

Comp. Gén. d'Electricité. Pot or crucible furnaces. 29,075. Oct. 14. (Fr., 20.10.19.)

Craig. 29,710. See VII.

Electrolytic Zinc Co. Treatment of finely-divided zinc. 30,057. Oct. 23. (Australia, 24.10.19.)

Friedrich. Production of glaze-like mineral coatings for iron. 29,370. Oct. 18. (Ger., 18.10.19.)

Hall, and Rolls-Royce Ltd. Aluminium-bronze alloys. 29,673. Oct. 20.

Johnston. Apparatus for separating metals from ores. 30,026. Oct. 23.

Lockwood. Treating ores, concentrates, tailings, etc. 29,214. Oct. 15.

Morton (Gibson). Alloys. 29,200. Oct. 15.

Rheinisch-Nassauische Bergwerks u. Hütten A.-G. Rotary roasting and calcining furnaces. 29,532. Oct. 19. (Ger., 20.10.19.)

Richards. Manufacture of steel. 29,679. Oct. 20.

Vickers, Ltd., and Wilson. Anti-corrosive composition. 29,550. Oct. 19.

COMPLETE SPECIFICATIONS ACCEPTED.

9268 (1918). British Thomson-Houston Co. (General Electric Co.). Manufacture of steel. (152,371.) Oct. 27.

20,840 (1918). Rossi. Production of an acid-proof alloy. (121,730.) Oct. 20.

16,514 (1919). Rouse. Extraction of iron and steel from ore and formation of alloys thereof. (152,073.) Oct. 20.

17,190 and 20,201 (1919). Ballantine. Production of chrome iron or chrome steel alloys. (152,399.) Oct. 27.

17,247 (1919). Ashcroft. See VII.

17,248 (1919). Ashcroft. Production of magnesium or its alloys. (152,403.) Oct. 27.

18,255 (1919). Collins. See VII.

18,584 (1919). Reol. Apparatus for dehydrating, reducing, calcining, or roasting minerals or other pulverulent material. (130,978.) Oct. 27.

19,262 (1919). Dudzele et Cie. Coating articles with metal. (139,143.) Oct. 27.

22,057 (1919). Ansell. Combined flux solder for aluminium. (152,486.) Oct. 27.

24,383 (1919). Priest. Continuous regenerative gas-fired kilns for burning ore-briquettes and high-temperature refractory materials. (152,509.) Oct. 27.

4975 (1920). Gerber. Manufacture of aluminium. (139,194.) Oct. 27.
 10,125 (1920). Bangerter. Alloy. (152,277.) Oct. 20.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Mond (Internat. Precipitation Co.). Electrical precipitation of suspended particles from gaseous media. 29,185. Oct. 15.
 Selvatico. Manufacture of electric furnaces and ovens. 29,640. Oct. 20.
 Smalley. Electric furnaces. 29,780. Oct. 21. (U.S., 21,10,19.)

COMPLETE SPECIFICATIONS ACCEPTED.

9489 (1919). Lodge, Lodge, and Lodge Fume Deposit Co. Electrical deposition of particles from gases. (152,051.) Oct. 20.
 17,247 (1919). Ashcroft. *See* VII.
 18,016 (1919). Electro-Metals, Ltd., and Robertson. Electric furnaces. (152,145.) Oct. 20.
 19,571 (1919). British Thomson-Houston Co. (General Electric Co.). Electric induction furnaces. (152,157.) Oct. 20.
 21,342 (1919). Heyen. Electric arc furnaces. (152,176.) Oct. 20.
 24,813 (1919). Soc. Anon. des Ateliers de Secheron. Electric resistance heaters and furnaces for high temperatures. (133,706.) Oct. 27.
 28,743 (1919). Soc. Anon. des Ateliers de Secheron. Electric furnaces. (138,858.) Oct. 27.
 28,744 and 30,520 (1919). Soc. Anon. des Ateliers de Secheron. Electric resistance heaters and furnaces. (138,859 and 137,276.) Oct. 27.

XII.—FATS; OILS; WAXES.

COMPLETE SPECIFICATIONS ACCEPTED.

16,741 and 20,192 (1919). Howse. Nitration of oils or fatty acids. (152,095.) Oct. 20.
 19,901 (1919). Morel. Manufacture of soap. (131,881.) Oct. 27.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Frood. Production and utilisation of phenol-aldehyde condensation products. 28,929. Oct. 13.
 Vickers, Ltd., and Wilson. 29,550. *See* X.

COMPLETE SPECIFICATION ACCEPTED.

14,694 (1919). Weller and Bindley. Phenol-formaldehyde condensation products and the production thereof. (152,384.) Oct. 27.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATION.

Campbell and Pheazey. 29,501. *See* XV.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Adachi and Nakayama. Utilisation of leather waste. 28,712. Oct. 11.
 Campbell and Pheazey. Manufacture of artificial leather and substitutes for rubber, gutta-percha, etc. 29,501. Oct. 19.
 Jacobsen. Impregnating and greasing leathers. 28,912. Oct. 12.
 Röhm. Depilating, neutralising, and bating hides and skins. 28,904. Oct. 12. (Ger., 31,12,19.)

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Alby United Carbide Factories, and Dodd. Manufacture of fertilisers containing nitrogen and phosphate. 29,090. Oct. 14.
 Foster. 29,029 and 29,030. *See* VII.
 Messerschmitt. Manufacture of manures. 29,969. Oct. 22.

XVII.—SUGARS; STARCHES; GUMS.

COMPLETE SPECIFICATION ACCEPTED.

1976 (1920). Verein. Chem. Werke. *See* XVIII.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATION.

Dixen and Moore. Manufacture of yeast, ales, stout, etc. 28,695. Oct. 11.

COMPLETE SPECIFICATION ACCEPTED.

1976 (1920). Verein. Chem. Werke. Manufacture of glycerol from sugar. (138,330.) Oct. 27.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Candy. Apparatus for purifying water. 28,927. Oct. 13.
 Candy. Apparatus for proportionate addition of liquid to flowing water. 29,078. Oct. 14.
 Chaviara, Clayton, and Nodder. Manufacture of margarine etc. 28,951. Oct. 13.
 Solomides. Insecticides and fungicides. 29,410. Oct. 18.
 Thomson. Alcoholic solution of animal etc. proteins for human use. 30,048. Oct. 23.

COMPLETE SPECIFICATION ACCEPTED.

20,134 (1919). Beckmann, and Veredelungsges. f. Nahrungs- u. Futtermittel. Manufacture of fodder from straw. (151,229.) Oct. 20.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Atack. Oxidation of hydrocarbons having side chains. 28,692. Oct. 11.
 Imray (Meister, Lucius, u. Brüning). Manufacture of therapeutically active acridine compounds. 29,106. Oct. 14.
 Wacker Ges. f. Elektro Chem. Ind. Manufacture of dichloroethylene. 29,956. Oct. 22. (Ger., 23,12,19.)

COMPLETE SPECIFICATION ACCEPTED.

22,739 (1919). Damiens, Loisy, and Piette. Manufacture of alcohol or ether. (152,495.) Oct. 27.

XXII.—EXPLOSIVES; MATCHES.

COMPLETE SPECIFICATION ACCEPTED.

24,120 (1919). Muller. Manufacture of ammonium nitrate explosives. (152,199.) Oct. 20.

XXIII.—ANALYSIS.

APPLICATION.

Hilger, Ltd., Smith, and Williams. Polarimeters of polarimeters, saccharimeters, etc. 29,333. Oct. 18.

I.—GENERAL; PLANT; MACHINERY.

PATENTS.

Pulverising apparatus. J. Aube. E.P. 151,072, 12.6.19.

Is an apparatus employing air or other gas for separating the ground material, the fan and dust separator are external to the grinding mill, the gas circulating in a closed circuit. A rotating gas distributor is keyed to the central driving shaft of the mill and distributes the inlet air, and the outlets for air and dust are controlled by dampers.

—B. M. V.

Pulverisers. A. E. Davis. E.P. 151,440, 16.8.19.

Is pulverisers for coal and similar materials, the ground material is drawn by suction from the beater chamber through a vertical screen arranged in a chamber above and of the same width as the beater chamber. Baffles are provided to prevent large pieces being flung directly from the beater blades on to the screen, but permitting the oversize material to drop back directly into the beater chamber. Means are provided for rapid removal of the screen for cleaning or repairs.—B. M. V.

Rotary machines for treating air or gases with liquid, and vice versa. W. J. Bulgin, E. A. Hall, and G. Searle. E.P. 151,314, 21.5.19.

The effective surface of a rotary machine for the treatment of air or gas with liquid, and vice versa, is constituted of helically twisted or corrugated metal strip, expanded metal, or metallic wool wound in layers, carried by a cylindrical support, through the surface of which the air or gas to be treated passes substantially radially. The cylindrical support rotates about a horizontal axis within a casing, so that the effective surface just dips into liquid contained in the lower part of the casing. Alternatively, the liquid may be supplied by pipes to the effective surface.—J. S. G. T.

Furnaces [; Recuperative —]. A. Smallwood. E.P. 151,366, 24.6.19.

Is the type of furnace where a working chamber, gas chamber, and recuperator are arranged one above the other, the heated air is led up flues on each side of the furnace to ports in the working chamber which, together with the adjacent gas ports, are individually controlled by dampers; the waste gases leave the furnace at one end and pass to the recuperator.—B. M. V.

Furnaces [; Recuperative gas —]. A. Smallwood. E.P. 151,367, 24.6.19.

Is a furnace of the type in which recuperators are arranged under the working chamber, the waste gases are withdrawn through outlets disposed along the sides of the working chamber, while the gas and heated air are admitted through ports in the floor of the chamber from their appropriate flues underneath.—B. M. V.

Heat exchange devices. Maison F. Fouché. E.P. 151,551, 20.3.20. Conv., 12.2.20.

A HEAT exchanger, more particularly applicable to steam condensers and coolers for liquids and gases, comprises a number of juxtaposed or superposed radiator elements each composed of a coil of high-pressure tubing having a connecting sleeve at each end. These sleeves are hollow and comprise an inner and outer wall, and stepped end walls which fit tightly against each other. The sleeves slide along a collector tube in which there is a longitudinal slot, and are clamped in position by a nut or other device. Distance rings can be inserted

between two sleeves and are provided with joint faces corresponding to those of the sleeves.

—J. S. G. T.

Heat-insulating material for steam and other pipes, boilers, cylinders, and the like; Manufacture of —. J. D. Gilmour. E.P. 151,738, 3.7.19.

THE waste products, consisting mainly of calcium carbonate, obtained in the manufacture of caustic soda and sodium hypochlorite, and in the leaching of bleaching powder, are mixed with 10% of fibrous material (preferably asbestos) and with water to the consistency of stiff mortar. The mixture is cast into moulds, transferred from the mould to a flanged semi-cylindrical core, and dried in an oven. Before mixing with the fibrous substance the waste materials may be treated with carbon dioxide from waste chimney gases to convert any calcium hydroxide into flocculent calcium carbonate. If asbestos is not used, a veneer of waste material with asbestos is applied, so that the latter only is in contact with the heated surface.—H. S. H.

Mixer. J. Johnson. U.S.P. 1,353,165, 21.9.20. Appl., 21.2.19.

A ROTARY mixer is mounted on a vertical shaft and arranged at the bottom of a vessel containing the material to be mixed. Material is directed outwards through the mixer by centrifugal force, circulates upwards through the vessel at the periphery, and then downwards at the centre to the central opening of the mixer. Means are arranged at the inlet of the mixer to impose on the incoming material a movement radially inwards.—W. F. F.

Dissolver; Rapid —. J. Johnson. U.S.P. 1,353,166, 21.9.20. Appl., 21.2.19.

A CONTAINER has pockets at the bottom formed by radial partitions extending inwards about half the radius of the container, and the material to be dissolved is placed in these pockets. A central rotating wheel and guide blades are arranged to direct the solvent liquid into the pockets.—W. F. F.

Dehydrator. W. M. Luther. U.S.P. 1,353,167, 21.9.20. Appl., 26.9.19.

A CASING contains a series of L-shaped heating pipes attached at one end to a transverse inlet header, and at the other end to a longitudinal outlet header. A reducing valve is placed in the inlet pipe. Superposed horizontal conveyors are arranged so that the uppermost projects through one end of the casing and the lowermost through the other end. A spreader is arranged at the inlet of the uppermost conveyor, and the material passes from each conveyor to the next below. The conveyors are driven through variable speed gearing, and travel alternately in opposite directions with a wave-like motion.—W. F. F.

Drying or cooling apparatus. P. Steinfeldt-Lindholm. U.S.P. 1,353,358, 21.9.20. Appl., 11.3.19.

THE material to be dried is supported on shelves arranged to form a zig-zag channel for the passage of air heated to the desired temperature. The air is heated in a passage located on one side of and below the drying chamber, and after passing over the material is led through a chamber above the drying chamber, containing hygroscopic material.

—J. S. G. T.

Dryer. F. C. M. Yahn, Assr. to Buffalo Foundry and Machine Co. U.S.P. 1,353,980, 28.9.20. Appl., 27.1.19.

THE material is dried on a moving surface, from which it is scraped and falls off between plates which form the walls of a jacket through which a cooling fluid is passed.—B. M. V.

Leaching machine. G. S. Dyer. U.S.P. 1,353,463, 21.9.20. Appl., 27.8.19.

MATERIAL to be treated is fed at one end into a rotary cylindrical leaching chamber. A longitudinal series of transverse annular partitions divides the chamber into compartments, and diametrically opposite shifter plates, spaced at their outer edges from the partitions and extending at their inner edges through central openings in the partitions, are arranged adjacent to each partition and oblique thereto. A pipe for supplying water is provided at the other end of the chamber. Each compartment of the chamber can be supplied independently with steam through a pipe placed outside the peripheries of the openings in the partitions. At the end of the chamber at which water is supplied, the steam pipes are jacketed by a washing chamber.
—J. S. G. T.

Catalytic reactions in the vapour phase; Producing —. F. A. Canon, Assr. to The Barrett Co. U.S.P. 1,355,105, 5.10.20. Appl., 18.4.19.

A CIRCULATORY motion is imparted to a catalyst suspended in a gas mixture.—L. A. C.

Atomising liquids, or solids dissolved or suspended in them; Process for —. H. Seiferheld. G.P. 302,554, 20.2.17.

ATOMISING of liquids may be promoted by dissolving a gas in the liquid and so raising its vapour tension considerably. Solids may be atomised by dissolving them in a liquid and then treating this as above. In the application of the method to atomising water for use in sulphuric acid chambers, or for washing gases in the contact process, sulphur dioxide is dissolved in the water. The process is applicable to alcohols, ether, olefines, and mineral acids and to naphthalene, camphor, pinacones, and phenol, and, in addition to sulphur dioxide, carbon dioxide and monoxide, nitrogen oxides, or air may be employed.—W. J. W.

Gases; Process for cooling — previous to liquefaction. Chem. Fabr. Griesheim-Elektron. G.P. 324,266, 16.2.17.

THE dry waste gas leaving the apparatus is cooled by saturation with a cooling liquid, and is then used to cool the gas to be liquefied.—L. A. C.

Dryers; Rotary —. O. Imray. From W. J. Kuntz. E.P. 151,518, 31.12.19.

SEE U.S.P. 1,229,978 of 1917; J., 1917, 918.

Electrical deposition of particles from gases. O. and L. Lodge, and Lodge Fume Deposit Co., Ltd. E.P. 152,051, 14.4.19.

SEE U.S.P. 1,345,790 of 1920; J., 1920, 711 A.

Furnace; Mechanical —. A. Ramén. U.S.P. 1,353,268, 21.9.20. Appl., 1.5.18.

SEE E.P. 118,812 of 1918; J., 1918, 659 A.

Evaporating and distilling apparatus. A. Duodo. U.S.P. 1,353,521, 21.9.20. Appl., 16.11.17.

SEE E.P. 111,480 of 1917; J., 1918, 537 A.

Crystals; Method of and apparatus for forming large —. O. Dreihrodt, Assr. to Elektrochem. Werke Ges. U.S.P. 1,353,571, 21.9.20. Appl., 27.6.14. Renewed 6.2.20.

SEE G.P. 273,929 of 1913; J., 1914, 822.

Surface condenser. Condenser. R. N. Ehrhart. U.S.P. 1,353,641-2, 21.9.20. Appl., 21.8 and 8.11.18.

SEE E.P. 131,595 and 134,852 of 1919; J., 1920, 681 A.

Gases; Apparatus for treating — with liquids. G. H. Walker, Assr. to Heenan and Froude, Ltd. U.S.P. 1,353,685, 21.9.20. Appl., 31.5.16.

SEE E.P. 8676 of 1915; J., 1916, 822.

Drying sand; Machines for —. J. Southall. E.P. 151,891, 4.2 and 20.4.20.

Furnaces [; Heat-treating —] and discharging apparatus therefor. C. A. Parsons, S. S. Cook, A. Q. Carnegie, and J. Ford. E.P. 152,104, 4.7.19.

Digesters and other apparatus; Covers for —. Soc. d'Outillage Mecanique et d'Usinage d'Artillerie. E.P. 152,265, 20.2.20. Conv., 15.12.19.

Drying. E.P. 151,488. See XIX A.

II A—FUEL; GAS; MINERAL OILS AND WAXES.

Benzene formed in gas retorts and coke ovens; Origin of —. F. Fischer and H. Schrader. Brennstoff-Chem., 1920, 1, 4-6, 22-24.

PREVIOUS theories on the formation of benzene are shown to be untenable. It is shown experimentally that phenols when heated with hydrogen yield benzene, $C_6H_5(CH_3)OH + H_2 = C_6H_6 + CH_3 + H_2O$, and $C_6H_5CH_3 + H_2 = C_6H_6 + CH_4$, and this reaction may be of importance in the manufacture of benzene from tar. With iron tubes only poor yields of benzene were obtained and much carbon separated, but with tinned iron tubes, at 750° C., good yields (up to 78%) were obtained from phenols and from toluene and higher benzene homologues. Hexane yielded very little benzene, but high yields were obtained from diphenyl, aniline, diphenylmethane, and diphenylamine.—J. R. P.

Methane; Behaviour of — at high temperatures alone and in contact with the other constituents in firedamp explosions. H. Winter. Brennstoff-Chem., 1920, 1, 17-22.

IN mixtures of pure methane and air containing 5.0-9.2% CH_4 combustion takes place according to the equation $CH_4 + 2O_2 = CO_2 + 2H_2O$. In mixtures containing 9.2-14% CH_4 combustion of the methane is incomplete. In addition to carbon dioxide, water, and hydrogen, the residues contain carbon monoxide when the ratio $O_2:CH_4$ is somewhat greater than, equal to, or less than 2.0:1. When the concentration of oxygen becomes smaller, hydrogen appears in addition to carbon dioxide and monoxide, nitrogen, and water. The explosion under these conditions is also accompanied by deposition of carbon within a small area.—J. R. P.

Gasoline from natural gas. IV. Temperature correction and blending charts. R. P. Anderson. J. Ind. Eng. Chem., 1920, 12, 1011-1015.

A NOMOGRAPHIC chart based upon the relationship between the coefficient of expansion and the sp. gr. of gasoline is given; also temperature correction tables for Baumé gravity and volume extending from 30° F. to -20° F.; and a blending chart for blending two grades of gasoline, or gasoline and naphtha.—C. A. M.

Petroleum distillates; Use of alcohol in refining —. Z. Budrewicz. Przemysł Chem., 1920, 4, 63-64. Chem. Zentr., 1920, 91, IV., 448.

IN the refining of Baku solar oil with sulphuric acid, alcohol is added in some factories to remove the sulphonic acids, aqueous solutions of which form persistent emulsions with mineral oils. The oil, after treatment with sulphuric acid, is warmed to 50° C., treated with 0.25-0.3% of alcohol, shaken

for 1 hr., and allowed to stand. The sulphonic acids are then run off and the oil is washed. In this way 7% of sulphonic acids have been produced from Baku solar oil. They possess detergent and emulsifying properties in acid, neutral, and alkaline solutions and in hard waters.—J. H. L.

Paraffin wax; Influence of high temperatures on — H. Burstyn and W. Jakubowicz. *Przemysł Chem.*, 1919, 3, 36—42. *Chem. Zentr.*, 1920, 91, IV., 448—449.

Crude petroleum from Boryslaw, sp. gr. 0.856, after treatment with 50% of 98% sulphuric acid at 40° C. and subsequent washing, was distilled. According as the distillation was stopped at 150°, 200°, 250°, or 300° C., the proportion of paraffin wax obtained from the residue was 13.8%, 12.8%, 12.2%, or 10.1%. In each case the paraffin had a setting point of 51–59° C. Since no appreciable amount of paraffin wax passed into the distillates the differences in the amount of wax obtained are attributed to decomposition, which is stated to occur even at 125° C., mainly owing to the action of the air. On this account distillation *in vacuo* is recommended.

—J. H. L.

Ozokerite; Determination of paraffin wax in — A. S. Koss. *Przemysł Chem.*, 1920, 4, 74—76. *Chem. Zentr.*, 1920, 91, IV., 449—450.

From 2 to 5 g. of the paraffin distillate is mixed with 20% of animal charcoal previously dried at 140° C., and the mixture is extracted with petrol of b.p. 50°–60° C. The extract, containing the total paraffin wax and the paraffin oil, is freed from petrol, dissolved in warm acetone, and cooled to –22° C., whereupon the paraffin wax crystallises whilst the oil remains in solution. After filtering and washing with acetone at –22° C., the paraffin is dried at 100° C. The soft paraffins separate from the acetone solution only at –30° C. A sample of ozokerite showed 47.35% of paraffin by the Engler-Höfer method, and 65.85% by the method described above.—J. H. L.

Mineral oil products; Determination of the iodine value of — W. R. Röderer. *Z. angew. Chem.*, 1920, 33, 235—237.

The Hübl-Waller and Wijs iodine solutions are the most suitable for lignite tars and distillates. With the former the maximum values are obtained in 24 hrs. with a 5-fold excess of iodine, and with the latter in 16 hrs. with a 10-fold excess. Crude petroleum oils require a double excess of iodine with Wijs' solution and a period of 16 hrs., and montan wax a 10-fold excess. In the case of each product it is necessary to determine the effect of iodine excess and of time.—C. A. M.

Oil emulsions; New technical methods for separating aqueous — I. Maseicki and K. Kling. *Przemysł Chem.*, 1920, 4, 2—8. *Chem. Zentr.*, 1920, 91, IV., 448.

In accordance with the method previously suggested (J., 1919, 885A), aqueous mineral oil emulsions may be separated by maintaining them for 2–4 hrs. at 120°–140° C. under 2–6 atm. pressure in a perfectly air-tight pressure vessel. The emulsion may be forced continuously under pressure through a preheating system into a vertical holder, in which it slowly rises. The dimensions of the holder are such that the emulsion remains in it for several hours at a temperature high enough to effect separation. The separated oil is discharged through a pressure valve at the top of the holder, and the separated water is discharged from the bottom.—J. H. L.

Gelatin as an emulsifying agent [for kerosene]. H. N. Holmes and W. C. Child. *J. Amer. Chem. Soc.*, 1920, 42, 2049—2056.

In the emulsification of kerosene in water and salt

solutions by the aid of gelatin the maximum lowering of the surface tension should be obtained. This is secured just as well by 0.3–0.4 g. of gelatin per 100 c.c. of water as by 1.0 g. Acids, bases, and liquefying salts (sodium iodide, chloride, and nitrate) also lower it a little, whilst solidifying salts (sodium sulphate, tartrate, and citrate) raise it. Viscosity must not be increased more than a little beyond that of water. A small amount of gelatin only may therefore be used or the gelatin must be liquefied by suitable electrolytes. The latter method yields the better emulsion. An excess of acid, base, or liquefying salt must not be used. The main factor in oil-water emulsification with the aid of gelatin is viscosity—not the maximum but the most favourable viscosity. (Cf. J.C.S., Dec.)

—J. F. S.

See also pages (A) 746. *Ammoniacal liquor* (Frère); 751. *Blast-furnace gas* (Hutchinson and Bury); 763. *Ichthyl oil* (Scheibler); 768. *Absorbent for hydrocarbons* (Piechota), *Carbon monoxide* (Teague).

PATENTS.

Burning fuel. H. H. Hurt, Assr. to Robeson Process Co. U.S.P. 1,329,300, 27.1.20. Appl., 11.5.18.

FINELY divided fuel is wetted down with concentrated sulphite-cellulose waste lye and charged on to a fire bed.

Retorts for distilling carbonaceous materials. J. West, W. Wild, and West's Gas Improvement Co. E.P. 144,051, 7.5.19.

A SET of horizontal or inclined gas retorts discharge into a vertical chamber, heated externally, into which steam is injected to produce water-gas by interaction with the coke from the retorts. The water-gas passes through the retorts and mixes with the distillation gases. The vertical chamber may be discharged continuously or intermittently. The retorts are heated by products of combustion from chambers below, the gases, after passing back and forth across the retorts, being led down the opposite sides of the vertical coke chamber. The retorts are supported on walls which serve as baffles to equalise the heating.

Coke-oven and like gases; Recovery of ammonia from — Soc. Ind. de Prod. Chim. E.P. 136,834, 27.9.18. Conv., 27.5.18.

THE ammonia is absorbed in a solution of sodium bisulphate at 70° C. The precipitate of sodium-ammonium sulphate which forms is separated, dried, and heated, first at 100° C. to expel water of crystallisation, and then at 350°–600° C. to decompose it into ammonia and sodium bisulphate.

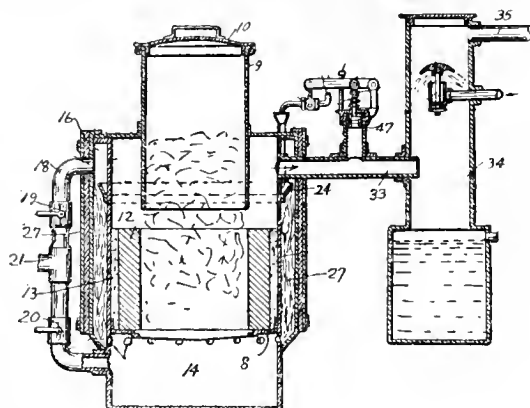
Gas producers. J. F. Wells. E.P. 141,520, 1.9.19.

AROUND the producer chamber is an annular water space through which tuyères extend to deliver air to the producer. The air is supplied by an injector, which may be operated by the exhaust of the engine or, before the injector comes into operation, by an auxiliary fan. The air may first be drawn over the surface of the water in the annular space to become charged with steam, or the annular space may be closed and the steam generated used to actuate the injector. The ashes are discharged by a screw conveyor working in a chamber below the producer.

Suction gas generators. J. Franklin. E.P. 151,329, 18.6.19.

A CYLINDRICAL generator is provided with a fire-brick lining, 12, surrounded by asbestos, 13, supported on the firebar supports, 8, and extending upwards to a level short of the bottom of the feed hopper, 9. The generator is surrounded by an annular space, 16, packed with wire netting, 27, and a pipe, 18, is provided connecting the upper

end of this space with the ashpit, 14. Gas is drawn from the generator through the pipe, 33, to the cooler and washer, 34, and thence to the main,



35. In operation the damper, 19, is closed and 20 opened, and the cover, 10, removed, so that air is drawn through the pipe, 21, and upwards through the fire. The damper, 19, is then opened and 20 closed, water is admitted to the channel, 24, and the cover, 10, replaced. Air and steam are thus drawn through the packing, 27, and ashpit, 14, to the fire to generate water-gas. The water supply is regulated automatically by the suction pressure of the engine, which acts upon the piston, 47.

—W. F. F.

Gas producer. F. J. Grocott. E.P. 151,520, 15.1.20.

A gas producer mounted on a truck is provided with a vertical partition extending downwards from the top for about half the height. A fuel supply hopper is arranged at the top of one of the compartments and a gas outlet at the top of the other. Another gas outlet at the side of the latter compartment is connected with a furnace where the gas is to be used as fuel. The gas flows to the outlet at the top as soon as it is opened, and hence connexion of the other outlet with the furnace is facilitated.—W. F. F.

Gas mixtures; Removal of carbon monoxide from — S. Fränkel. G.P. 303,931, 7.3.16.

In the use of chromic acid, in presence of sulphuric acid, as a gas absorption agent, the addition of catalysts is prescribed, suitable materials being mercuric oxide, silver oxide, palladium oxide, and oxides and compounds of metals such as iron, manganese, chromium, and tungsten, which combine with oxygen in various proportions.—W. J. W.

Gas cleaning apparatus. J. F. Wells. E.P. 151,443, 20.8.19.

An apparatus for cleaning gas and extracting tar is built up of separate elements mounted on a horizontal shaft, the number of elements being variable. The casing is divided into transverse compartments by annular partitions clamped between circumferential rings, and paddles are arranged to rotate in the compartments. Each paddle is provided with a central disc so that the gas is compelled to take a tortuous path alternately outwards and inwards over the paddles and through the central openings in the partitions. A propelling fan is provided in the end compartment and tar outlets at the bottom of each compartment.—W. F. F.

Naphthalene from gas obtained by the distillation of coal; Apparatus for the complete extraction of — D. Marbais and C. Deguido. E.P. 151,463, 13.9.19.

Coal gas containing naphthalene is passed through

a refrigerator, where it is cooled to 18° C., and then upwards through a scrubber in counter-current to anthracene oil which is sprayed into the top at 22° C. The anthracene oil may be mixed with about 4% of benzol to prevent absorption of benzol from the gas. The oil is subsequently distilled with steam to free it from naphthalene, and is then passed through a cooler supplied with cold water from the gas cooler first mentioned, where it is cooled to 22° C. and supplied again to the scrubber. (Cf. E.P. 118,730; J., 1918, 616 A.)—W. F. F.

Ammonia; Preparation of — from gas liquor. G. Schuchardt. G.P. 324,582, 16.7.19.

In the process in which the ammonia is driven off by means of hot air, both the air and liquor feeds are preheated by the heat of the retorts.—C. I.

[Petroleum] hydrocarbons; Apparatus for cracking — J. W. Coast, jun., Assr. to The Process Co. U.S.P. 1,353,316, 21.9.20. Appl., 27.9.17.

The oil is circulated through two cracking elements, one a pipe, and the other a relatively large still. The pressure is regulated in the still by an outlet valve, and in the pipe by a valve in one of the connexions between the two elements. Vapour passes out of the still into a condenser.—L. A. C.

Gasoline and other products; Apparatus for producing — from hydrocarbons. A. A. Daugherty. U.S.P. 1,353,638, 21.9.20. Appl., 23.5.17.

HYDROCARBON oils are heated above 700° F. (370° C.) and under a pressure of more than 300 lb. per sq. in., and are allowed to expand suddenly to atmospheric pressure. The expansion vessel is heated to vaporise any condensed liquid, and the vapour is withdrawn therefrom by suction and passed into a condenser.—L. A. C.

Hydrocarbons; Continuous production of light — from heavy hydrocarbons and waste products thereof by distillation under pressure. Zeller und Gmelin. G.P. 303,235, 29.10.15.

The oil is first subjected to a cracking process under a low excess pressure to remove substances which are liable to form coke and pitch, and then subjected to a continuous distillation process under a high pressure in an autoclave.—J. H. L.

Oils; Process and apparatus for separating — from mixtures by means of continuous distillation and concentration columns. Maschinenbau-A.-G. Golzern-Grimma. G.P. 324,280, 20.2.18.

THE residue leaving the concentrator passes into a reservoir heated by the distillation column, and either returns as vapour to the concentrator, or overflows into the distillation column.—L. A. C.

Lubricant; Production of a consistent — from mineral oil and resin oil. Dr. Graf und Co., and A. Fischer. G.P. 323,905, 12.2.18. Conv., 4.2.18.

An aqueous emulsion consisting of resin oil or distillation residues therefrom, mineral oil, and aqueous ammonia, is intimately mixed with milk of lime and then treated with an aqueous solution of aluminium or zinc sulphate. The greater part of the water is then separated, preferably by means of a filter-press, and the residue is intimately mixed with mineral oil and, if desired, with an aniline dye. On heating for several hours, the product undergoes a reaction, with elimination of water, and a lubricant very stable towards heat is produced which is suitable for high-speed engines, automobiles, etc.—J. H. L.

Coke; Manufacture of — and apparatus therefor. F. J. Sivyer. U.S.P. 1,354,809, 5.10.20. Appl., 15.11.17.

SEE E.P. 113,091 of 1918; J., 1919, 4 A.

Shale and other carbonaceous materials; Apparatus for recovering oil from —. Process for distilling carbonaceous materials. G. W. Wallace. E.P. 129,996—7, 16.7.19. Conv., 8.2 and 26.3.18. SEE U.S.P. 1,283,000—1 of 1918; J., 1919, 68 A.

Hydrogenating carbon compounds [c.g., coal, for preparation of liquid products]; Process of —. F. Bergius, Assr. to The Chemical Foundation, Inc. U.S.P. 1,342,790, 8.6.20. Appl., 18.4.16. SEE E.P. 18,232 of 1914; J., 1916, 167.

Briquettes; Presses for forming —. J. Réol. E.P. 128,956, 27.6.19. Conv., 20.1.11.

Hydrogenation of naphthalene. G.P. 300,052. See III.

Oil varnish substitute. G.P. 323,155. See XIII.

Fatty acids etc. G.P. 321,663. See XX.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Acetone. Duclaux and Lanzenberg. See XX.

PATENTS.

Metallic-filament electric lamps; Removing film from [bulb of] used —. F. Harrison. U.S.P. 1,353,654, 21.9.20. Appl., 8.7.19.

THE inner surface of the bulb is washed with hydrogen peroxide.—J. S. G. T.

Electrical discharge tubes. Siemens und Halske A.-G. G.P. 323,205, 11.3.13.

IN discharge tubes conductivity is caused by nitrogen obtained by the use of electrodes of a metal such as tantalum, which absorbs large volumes of the gas and again evolves it on heating.—W. J. W.

Electrodes for discharge tubes; Manufacture of — from light metals. Studien-Ges. für elektrische Leuchtröhren m.b.H. G.P. 323,494, 3.11.17.

COMPOUNDS of the desired metals which are readily decomposed, or mixtures of these, are decomposed in the discharge tubes or in vessels in connection with them. Suitable compounds are metallic azides or a mixture of sodium azide and barium chloride.—W. J. W.

Products from carbohydrates. U.S.P. 1,327,737—8. See XI.

III.—TAR AND TAR PRODUCTS.

Aromatic compounds; Use of catalysts in the sulphonation of —. J. A. Ambler and W. J. Cotton. J. Ind. Eng. Chem., 1920, 12, 968—969.

BENZENE was dissolved in 70% sulphuric acid and sulphonated at 243° to 259° C. in the presence of various catalysts (0.1% of the active element of the weight of sulphuric acid). Compounds of copper, mercury, vanadium, chromium, potassium, and lithium had a slight catalytic action, whilst mixtures of sodium sulphate and vanadium pentoxide were the most active. The increased sulphonation caused by the presence of sodium and potassium sulphates in the sulphonation mixtures was not entirely due to the increase in the boiling point of the sulphuric acid. The formation of disulphonic acid was promoted by the presence of sodium or lithium sulphate, and still more so by the mixture of vanadium pentoxide and sodium sulphate, but was inhibited by the other catalysts.—C. A. M.

Formation of benzene. Fischer and Schrader. See IIA.

Iodine value of mineral oils. Röderer. See IIA.

PATENTS.

Anthranol; Manufacture of —. A. G. Perkin. E.P. 151,707, 26.6.19.

ANTHRANOL is obtained by heating anthraquinone (60 pts.) with glucose (60 pts.) and a 30% solution of sodium hydroxide (650 pts.) in an autoclave provided with stirring gear at 230° C. for 5—10 hrs. The anthranol is precipitated from the reaction mixture by the addition of hydrochloric acid or by leading in carbon dioxide. Instead of glucose, sucrose, molasses, maltose, lactose, and the like may be used with similar results.—G. F. M.

Anthracene; Catalytic oxidation of —. J. M. Weiss and C. R. Downs, Assrs. to The Barrett Co. U.S.P. 1,355,098, 5.10.20. Appl., 1.11.18.

ANTHRACENE vapour is treated with oxygen in the presence of a vanadium oxide at 300°—500° C.—L. A. C.

Coal-tar product; Process of making a —. J. M. Weiss, Assr. to The Barrett Co. U.S.P. 1,355,099, 5.10.20. Appl., 27.10.19.

THE constituents of tar bitumen soluble in coal-tar naphtha are treated with petroleum naphtha, and the soluble portion is separated from the residue.—L. A. C.

Coal-tar distillates; Treatment of high-boiling — for the production of resinous products. G. C. Bailey and F. Boettner, Assrs. to The Barrett Co. U.S.P. 1,355,103, 5.10.20. Appl., 27.10.19.

A HARD, transparent, red resin, m.p. above 40° C., which does not darken on exposure to air and light, is prepared by extracting a high-boiling solid to semi-solid coal-tar bitumen with a solvent, filtering the solution, and, after separation of the solvent by distillation, purifying the product by fractional distillation *in vacuo*.—L. A. C.

Chlorobenzoyl-benzoic acid; Production of —. J. M. Weiss, G. C. Bailey, and R. S. Potter, Assrs. to The Barrett Co. U.S.P. 1,355,100, 5.10.20. Appl., 12.12.19.

As a step in the production of chlorobenzoyl-benzoic acid, a mixture of phthalic anhydride and aluminium chloride, from which all particles small enough to pass through a 20-mesh sieve have been removed, is added to chlorobenzene at a temperature above 110° C.—L. A. C.

Hydronaphthalenes; Production of —. Tetralin Ges.m.b.H. G.P. (A) 299,012, and (n) 299,013, 2.8.16.

(A) VAPORISED naphthalene is brought into intimate contact with finely-divided or easily fusible metals, alloys, or feeble non-saline compounds of such metals, either with or without the additional presence of finely-divided or porous materials such as fuller's earth; after this process of purification, which occurs more rapidly in the gaseous than in the liquid state, the material is reduced with hydrogen and a catalyst. Sodium or finely-divided iron and nickel are suitable for the preliminary purification; the subsequent reduction with a nickel catalyst can be effected at 100° C. (n) Instead of vaporised naphthalene, a solution in a suitable organic solvent, such as tetrahydronaphthalene, may be used for the purification process.—D. F. T.

Naphthalene; Hydrogenation of —. K. Wimmer. G.P. 300,052, 14.3.15.

THE hydrogenation catalyst is obtained by heating an organic salt, such as nickel formate, in a saturated glyceride with hydrogen. The product permits the reduction of naphthalene at 180° C., by hydrogen under a pressure of 15 atm., to deca-

hydronaphthalene. A mixture of the hydronaphthalenes with benzene or petroleum spirit can be used as motor fuel.—D. F. T.

Chlorinated derivatives of naphthalene of waxy consistency; Production of —. Chem. Fabr. Griesheim-Elektron. G.P. 322,794, 10.12.18. Addn. to 319,253 (J., 1920, 442 A).

In the process described in the original patent, the chlorination may be checked when the product has a solidifying point of 110°–118° C., after which air or an inert gas is blown through it at 200° C. The chlorine-content of the product may reach 58%.—W. J. W.

Benzol refining; Still for recovery of sulphuric acid used in —. O. Schröder. G.P. 324,731, 16.9.19.

The still can be tipped, and a removable lid carries the steam jet and vapour exit pipe.—C. I.

Distilling oils. G.P. 324,280. See IIa.

Artificial resin. U.S.P. 1,353,220. See XIII.

Oil varnish substitute. G.P. 323,155. See XIII.

Fatty acids etc. G.P. 324,663. See XX.

IV.—COLOURING MATTERS AND DYES.

PATENTS.

Colours soluble in fats and oils and process of preparing the same. W. Clark. From Chem. Fabr. Worms A.-G. E.P. 151,657, 8.5.18.

COLOURS suitable for printing or for colouring resinous and fatty materials are prepared by treating the colour bases of basic dyes with a naphthenic or montanic acid, or by treating alizarin dyes with an alkali salt of a naphthenic or montanic acid and with aluminium sulphate.—L. A. C.

Diphenylamine derivatives [dyestuffs]; Manufacture of —. British Dyestuffs Corp., Ltd., J. Turner, and L. G. Badier. E.P. 151,868, 17.12.19.

New dyestuffs are obtained by bringing into reaction with alkali cyanides the ammonium or sodium salts of a nitrated diphenylamine derivative which yields water-soluble alkali salts, such as hexanitrodiphenylamine, tetranitrothio-oxydiphenylamine, or the sulphonic acids of dinitrodiphenylamine. The products are precipitated from the reaction mixture by the addition of common salt. They produce, on wool, brown to purple shades, fast to light.—G. F. M.

Dye-soap; Process of manufacture of —. C. S. Robison, Assr. to Aladdin Products Co. U.S.P. 1,353,182, 21.9.20. Appl., 25.10.17.

A MIXTURE of coconut oil (10 pts.), castor oil (10 pts.), and tallow (8 pts.) is saponified at 50° C. with 14 pts. of sodium hydroxide solution (38° B., sp. gr. 1.357), a solution of 8 pts. of granulated sugar in 8½ pts. of water at 85° C. is added, the mixture is neutralised with coconut oil and fatty acid, and an alcoholic solution of a dye is mixed with the soap solution at 60° C.—L. A. C.

Dyes of the Magenta type; Process of producing —. J. L. Kane. U.S.P. 1,355,048, 5.10.20. Appl., 21.12.19.

As a step in the production of alkyltriarylamine dyes, a salt of an alkyltriarylamine is produced by the interaction of an aromatic amine, an aromatic nitro compound, and an acid in the presence of a starchy material.—L. A. C.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Reeds (Arundo phragmites L.); Utilisation of —. E. Heuser. Papierfabr., 1920, 115. Chem.-Zeit., 1920, 44, Rep., 236.

THE cellulose content of the root stems of the common reed (*Arundo phragmites*) was determined by Cross and Bevan's chlorination method. The cellulose obtained contained 1.09% of ash and 24.74% of pentosan. The chlorination method gives therefore not the percentage of pure cellulose, but of crude cellulose. Calculated to an air-dried and ash-free basis the following figures were obtained:—25.42% pure cellulose, 44.02% lignin, 14.78% pentosan, 10.68% moisture. By fermentation of the aqueous extract two experiments gave an average yield of 9.28 l. of alcohol from 100 kg. of dry substance. A similar plant, the reed mace (*Typha latifolia*), was worked up in Germany during the war to hemicellulose for cattle food. This hemicellulose had the composition 58.70% pure cellulose, 28.05% pentosan, 9.45% lignin, and 3.80% ash.—G. F. M.

PATENTS.

Spanish moss; Method of utilizing —. M. W. Marsden. U.S.P. 1,327,873, 13.1.20. Appl., 18.7.18.

THE material is boiled with dilute sulphuric acid, and the liquid is separated and used for production of sugar, alcohol, etc. The cellular residue is suitable for conversion into nitrocellulose, artificial silk, etc.; or after boiling with a solution of soap and glycerin, washing, and drying, may be used as a filling for mattresses etc.

[Pile] fabrics; Colloid-treated — and method of producing the same. S. P. Lovell. U.S.P. 1,353,599, 21.9.20. Appl., 16.1.20.

VILLOUS pile fabric, impregnated with a solution of colloidal matter, is passed through a pile-opening mechanism and thence through a precipitating bath, whereby a colloidal material, insoluble in water, is deposited between the fibres of the pile.

—A. J. H.

Cellulose fibre; Method of producing —. J. A. de Cew. U.S.P. 1,354,731, 5.10.20. Appl., 21.10.19.

LIGNOCELLULOSE is impregnated with a solution containing 15% of caustic soda under the usual conditions of temperature and pressure of pulp cooking. Excess of alkali is then removed so that the lignone is attacked only by the alkali absorbed.

—A. J. H.

Broom; Production of textile fibres from —. A. Hammer. G.P. 323,607, 22.3.18.

THE plants are boiled, preferably under pressure, in a sodium hydroxide solution of about 5° B. (sp. gr. 1.036) until the cortex is disintegrated and can be removed by washing; the fibres freed from cortex are separated from the woody stems by combing with hackles of graded fineness.—J. H. L.

Straw and similar materials; Production of textile fibres from —. G. Strauss. G.P. 323,669, 21.9.19.

STRAW is steeped for a long time in a mixture of aqueous ammonia, sodium hydroxide, and tanning material.—J. H. L.

Cellulose acetates; Process for the manufacture of —. H. A. Levey. U.S.P. 1,330,543, 10.2.20. Appl., 13.7.18.

CELLULOSE is treated with chlorinated acetic acid, i.e., glacial acetic acid which has been treated with chlorine until it is distinctly yellow, and when dis-

integrated the excess acid is expressed and the mass dissolved in a mixture of acetic acid and zinc chloride at 55°–70° C. and further treated with chlorinated acetic acid for 12–36 hrs., at 50°–70° C. The product is then washed and dried at 60°–75° C. It is soluble in ethyl acetate, ethyl butyrate, and pyridine, and insoluble in benzene, carbon tetrachloride, and alcohol.

Cellulose-nitrate composition, and process of making the same. Mixed cellulose-ester composition, and process of making the same. S. J. Carroll, Assr. to Eastman Kodak Co. U.S.P. (a) 1,351,725 and (n) 1,351,726, 5.10.20. Appl., 2.1.20.

A COLLOIDAL mixture of tetrachloronaphthalene and triphenyl phosphato is combined with a solution containing (a) nitrocellulose and fusel oil, or (n) nitrocellulose, cellulose acetate, and fusel oil.

—A. J. H.

Cellulose; Production of (A) soda — from vegetable materials of all kinds, (n) pure soda-celluloses from wood, straw, esparto, rushes, bamboo, manila and other vegetable materials by double digestion with alkalis and acids. M. Müller. G.P. (a) 323,743, 18.6.19, and (n) 323,744, 21.8.19.

(A) DIGESTION is effected by means of soda or sulphate solutions to which alkali or alkaline-earth salts of lower fatty acids have been added or in which such salts have been formed by addition of lower fatty acids. Crude calcium acetate which contains calcium phenolate has a very favourable influence, and the addition of tar oils assists digestion. (n) Soda-cellulose produced by known methods of alkaline or neutral digestion is further treated with mixtures of inorganic and organic acids or their salts. The mother liquors from this second treatment contain precipitable substances which can be used for loading and sizing paper, etc.

J. H. L.

Cellulosic materials; Digestion of wood and other — with the aid of chlorine. A. Franz. G.P. 323,936, 18.9.19.

THE chlorine is used dissolved in an indifferent solvent, such as carbon tetrachloride. After extraction of the product with a 2% aqueous sodium hydroxide solution pure cellulose is obtained.

—J. H. L.

Sulphite-waste-liquor preparation and process of making the same. F. E. Coombs. U.S.P. 1,327,862, 13.1.20. Appl., 12.9.19.

SULPHITE-cellulose waste liquor is reduced with iron and acid and then concentrated, or the liquor is first concentrated and then reduced. The product, after dilution, may be used for dyeing unmordanted wool greenish-brown, and is also useful as an insecticide and for dressing the surface of roads.

Sizing of paper, cardboard, textile fabrics, etc., by means of sulphite-cellulose waste liquors. E. Rasch. G.P. 323,627, 31.5.16.

CONCENTRATED waste liquors are used without removal of the bases present, and if necessary with addition of further bases. If the calcium compounds present in the liquors are decomposed and the lime removed, the adhesive properties are impaired.—J. H. L.

Cellulose acetate; Manufacture of compositions, preparations, or articles having a basis of —. H. Dreyfus. U.S.P. 1,353,384–5, 21.9.20. Appl., 15.4.19.

SEE E.P. 132,283 and 133,353 of 1918; J., 1919, 896A; 1920, 14A.

Paper-making machines and the like [; Arrangement of suction boxes in —]. J. Heess and R. J. Marx. E.P. 151,516, 15.12.19.

Burning fuel. U.S.P. 1,329,300. See IIA.

Aluminium compounds for sizing. G.P. 323,626. See VII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Discharging Indigo Blue with glucose; Methods for —. A. Scheunert and N. Wosnessensky. Sealed notes 1990, 6.4.10; 1991, 22.4.10; 2019, 20.7.10; and 2075, 15.3.11. Bull. Soc. Ind. Mulhouse, 1920, 86, 263–266. Report by M. Battegay, *ibid.*, 266–267.

WHITE discharge effects are obtained by printing a fabric dyed with Indigo Blue, with a paste containing 300 g. of glucose, 700 g. of thickening, and (optional) 100 g. of zinc oxide and 50 g. of Leucotrope W, passing it through a solution of caustic soda of 30° B. (sp. gr. 1.26) at 100°–110° C., and immediately through boiling water. A short steaming of the fabric previous to treatment with caustic soda improves the result. For coloured effects the discharge paste contains 250 pts. of a suitable dye, 300 pts. of glucose and 450 pts. of thickening. The process may be combined with the production of ingrain dyes, the discharge paste then containing 200 g. of glucose, 150 g. of a solution of diazotised α -naphthylamine, chloroaniline, or benzidine, 50 g. of Leucotrope W, and 600 g. of thickening, and being used on indigo-dyed cloth prepared with β -naphthol. A brilliant red discharge is obtained by use of a paste containing 250 g. of a solution of diazotised Azo Rose BBI, 200 g. of glucose, 500 g. of British gum, and 50 g. of Leucotrope W. The printed material is passed through a caustic soda bath at 100° C. If 50 g. of stannous chloride be added to the above discharge pastes, the quantity of glucose used may be reduced by one-half, the caustic soda solution may be at 20°–25° B. (sp. gr. 1.16–1.21) and the resulting colours are more brilliant and intense. Battegay reports that the processes are better than the usual one in which the discharge paste consists of a mixture of glucose and caustic soda.—A. J. H.

Discharges on tannin-antimony mordanted cotton; Production of white and coloured —. J. Pokorny. Sealed note 2063, 25.1.11. Bull. Soc. Ind. Mulhouse, 1920, 86, 260–261. Report by P. Binder, *ibid.*, 261–262.

COTTON fabric mordanted with antimony tannate is printed with a thickened solution of a vat dye-stuff, overprinted with a discharge paste containing caustic soda, steamed in a Mather-Platt, washed, soaped, and dyed with basic dyes. The caustic soda simultaneously fixes the vat dye and destroys the mordant. Improved effects are obtained by incorporating glucose with the mordant, by chroming after steaming, and by adding about 50 g. of an alcoholic solution of β -naphthol to the vat dyestuff. Binder reports favourably on the process, which has been previously suggested for discharges on indigo. The temperature should not exceed 30°–50° C. whilst after-chroming, in order to avoid dulling the coloured ground.—A. J. H.

Reserves under Indanthrene Blue; Process for printing —. J. Pokorny. Sealed notes 2180–1 and 2186–7, 23.5 and 26.6.12. Bull. Soc. Ind. Mulhouse, 1920, 86, 257–258. Report by P. Binder, *ibid.*, 258–259.

COTTON fabric is printed with a reserve paste containing manganous chloride without the addition of a bichromate, dried, and dyed with Indanthrene Blue. A suitable paste for white reserves consists of 370 pts. of manganous chloride, 550 pts. of gum tragacanth, and 80 pts. of water. For red reserves

on cloth prepared with β -naphthol the paste contains 293 pts. of manganous chloride, 509 pts. of gum tragacanth, 165 pts. of a solution of diazotised *p*-nitro-*o*-anisidine, and 33 pts. of sodium acetate. A paste suitable for yellow reserves consists of 240 pts. of manganous chloride, 200 pts. of Indanthrene Yellow R. paste, 500 pts. of gum tragacanth, and 60 pts. of water. The addition of 150 pts. of stannous chloride to 1000 pts. of one of the above pastes, allows reserves under Indanthrene Blue and overprinted azo dyes to be obtained simultaneously. Binder reports that the process works well.

—A. J. H.

PATENTS.

Dye jig rollers. W. Dean and T. Lyttle. E.P. 152,285, 19.6.20.

Dye-soap. U.S.P. 1,353,182. See IV.

Sulphite-waste-liquor preparation. U.S.P. 1,327,862. See V.

Washing and cleaning. G.P. 323,193. See XII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric acid; Method of recovering — from sodium bisulphate. I. Moscicki and W. Dominik. *Przemysl Chem.*, 1920, 4, 17—23. *Chem. Zentr.*, 1920, 91, IV., 430.

Sodium bisulphate is mixed with an equal quantity of sand or sodium sulphate, and the sulphuric acid is then distilled off, only about 0.1% H_2SO_4 remaining in the residue.—W. J. W.

Nitric acid; Fractionation of —. J. Baumann. *Chem.-Zeit.*, 1920, 44, 678.

By distilling 69.8% nitric acid (sp. gr. 1.4) using a dephlegmator heated to 100° C., equal volumes of distillate containing 0.02% of HNO_3 and of residue containing 69.5% were obtained. Distillation of 500 c.c. 91.5% acid using a dephlegmator heated at 86° C. yielded 140 c.c. of 70.1% acid (the constant boiling mixture), and a residue of 350 c.c. of 100.5% acid.

Chlorine; Preparation of — from hydrochloric acid or chlorides. J. Baumann. *Chem.-Zeit.*, 1920, 44, 677.

The whole of the chlorine in hydrochloric acid or chlorides can be liberated by means of nitric acid of about 52% strength, and the nitric acid recovered unchanged, if the chlorine compound is added slowly to the boiling acid, using a long dephlegmator, and passing a current of air into the reacting gases. (*Cf.* J.C.S., ii., 684.)

Ammonia; Very high pressures and the synthesis of —. G. Claude. *Bull. Soc. Chim.*, 1920, 27, 705—724.

The author gives an historical account of the developments in the synthesis of ammonia, and a description of the application of pressures up to 1000 atm. to this process. (*Cf.* J., 1919, 885A; 1920, 61A, 187A, 655A.)—W. G.

Ammoniacal liquor; Automatic supply of steam in distillation of —. G. Frère. *Soc. Techn. du Gaz.* Gas J., 1920, 150, 841—842.

The lower part of the boiler from which steam is supplied to the still is connected with a reservoir open at the top; the level of the water in the reservoir hence varies with the steam pressure. A pipe with its lower end cut at an angle extends into the reservoir so that its end is partly sealed by the water. This pipe is connected with a pipe leading to the

boiler furnace, and according to the steam pressure and hence the water level in the reservoir, the end of the first pipe is more or less uncovered and more or less air passes through to the furnace. A similar device may be used when the steam supply comes from an independent source, but in this case the reservoir is provided with a counterbalanced float which actuates a valve on the steam supply pipe.

Carbon dioxide; Determination of — in bicarbonates in the presence of carbonates. W. Hartmann. *Z. anal. Chem.*, 1920, 59, 289—297.

In a mixture of alkali bicarbonate and carbonate, the former may be decomposed completely by heating the mixture with 70% glycerin at 114°—117° C. for 10 mins., and the evolved carbon dioxide may be collected and weighed. At the end of the 10 mins., the temperature should be lowered to 100° C., and a current of air aspirated through the apparatus to carry over all the carbon dioxide into the potash bulb. The alkali carbonate present is not affected by this treatment, but the carbon dioxide in this salt may be determined in the same way by heating the residue at 180°—190° C. for 5 mins., then adding 2 drops of water, continuing the heating for 10 mins., cooling to 150° C., and aspirating air through the apparatus. When ammonium salts are present, the liberated carbon dioxide should be dried by means of sulphuric acid; it is not advisable, in this case, to condense the moisture. From 7 to 18 c.c. of 70% glycerin is required for each 0.1 g. of carbonate.—W. P. S.

Soda-lime as an absorbent for industrial purposes. R. E. Wilson. *J. Ind. Eng. Chem.*, 1920, 12, 1000—1007.

The absorptive capacity of soda-lime is, in general, inversely proportional to its hardness, the latter apparently depending upon the formation of sodium silicate, which tends to clog the pores of the material. This tendency is not directly proportional to the amount of silica in the lime, but to differences in its reactive power. Limes containing over 5% of magnesium oxide are less absorptive than those rich in calcium. A low content of carbon dioxide and ferric oxide is desirable. Extra fine lime yielded granules which were harder, but less absorptive than those from lime of normal fineness. Completeness of hydration appears important to insure stability and hardness of the granules. Up to 5% alkali content the hardness of soda-lime increases greatly with the addition of the alkali, but beyond 6% there is a slight but distinct decrease in hardness. For soda-lime in gas-masks an alkali content of 5% (dry basis) and water content of 10—13% is the most desirable for gases in general. For the absorption of carbon dioxide in steel analyses, oxygen helmet apparatus, etc., an alkali content of 4.5% and water content of 16—19% appear to be the best. For chlorine absorption 8—9% alkali might be used. A greater proportion would be liable to cause trouble from caking. If 2% of kieselguhr is used the optimum alkali content for general purposes is about 3% instead of 5%, but the increase of hardness thus obtained is at the expense of the absorptive capacity.—C. A. M.

Chlorate and perchlorate formation; Theory of electrochemical —. N. V. S. Knibbs and H. Palfreeman. *Faraday Soc.*, June, 1920. [Advance proof.]

RESULTS of the measurement of the conductivities of chlorate, chloride-chlorate, and chlorate-perchlorate solutions, the velocity constants of the reaction hypochlorite to chlorate, and the resistance and potential effects in technical cells are given, the data on conductivities and on the dynamics of the hypochlorite-chlorate reaction covering a wider range than any previously published. The gener-

ally accepted theory of chlorate formation of Foerster and Müller (J., 1903, 417) is reviewed, and the loss of efficiency in technical working is shown to be due mainly to discharge of hypochlorite ions. Regulated addition of hydrochloric acid was found to be the most satisfactory method of maintaining hypochlorous acid concentration. Insoluble impurities in the electrolyte increase the resistance of the solution and therefore the cell voltage, reduce the current efficiency by facilitating the loss of hypochlorous acid, and exert an erosive action on the electrodes, the last effect being of special importance when platinum anodes are used. The two theories of perchlorate formation advanced by Oechsli (J., 1903, 1248) and by Bennett and Mack (Trans. Amer. Electrochem. Soc., 1916, 29, 323) are discussed, and neither is considered to be adequate. It is maintained that perchlorate formation is due to chlorate ion discharge, and a mechanism similar to that of persulphate formation is suggested. The factors in the current efficiency of technical perchlorate formation are discussed, and chloride formation is shown to take place during electrolysis and to be dependent upon the temperature.—B. N.

Aluminates of sodium. Equilibria in the system $\text{Na}_2\text{O}-\text{Al}_2\text{O}_3-\text{H}_2\text{O}$. F. Gondriaan. Proc. K. Akad. Wetensch., 1920, 23, 129—142.

At 30° C. two stable aluminates occur in the system $\text{Na}_2\text{O}-\text{Al}_2\text{O}_3-\text{H}_2\text{O}$, viz., $4\text{Na}_2\text{O} \cdot 3\text{Al}_2\text{O}_3 \cdot 16\text{H}_2\text{O}$ and $4\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 10\text{H}_2\text{O}$. Both forms inconspicuously saturated solutions, i.e., are decomposed by water and dilute sodium hydroxide solutions. According to the mode of preparation aluminium hydroxide may be obtained in different forms. Under special conditions alumina forms a crystalline hydrate, $\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$. The gelatinous hydroxide must be considered as a metastable phase of variable composition. It adsorbs variable quantities of alkali. There is probably a continuous transition between these gelatinous hydroxides and the crystalline hydrate. Aluminium oxide is metastable at 30° C. in respect to the hydrate. The swelling exhibited by desiccated particles of the hydroxide and oxide is dependent on the alkalinity of the solution.

—J. R. P.

Calcium oxide; Extraction of — from calcined magnesite. L. H. Duschak. Chem. and Met. Eng., 1920, 23, 628.

CALCIUM oxide may be almost completely extracted from magnesite after calcining at 900°—950° C., by leaching for several hours with water free from carbon dioxide. If the magnesite has been calcined at higher temperatures, and especially if it is "dead-burnt," the extraction is very incomplete and is not improved by fine grinding, probably owing to the formation of an insoluble compound.

—A. R. P.

Zinc sulphide; Influence of crystalline form, flux and actual fusion on the phosphorescence of —. E. Tiede and A. Schleede. Ber., 1920, 53, 1721—1725.

NEITHER crystalline form of zinc sulphide in the pure state is phosphorescent, but when the pure sulphide is fused (cf. *supra*) it becomes strongly phosphorescent. The effect of a flux such as potassium chloride in inducing phosphorescence in zinc sulphide is therefore not due to the formation of a definite crystalline form, but to the lowering of the melting point. The pure fused sulphides of the alkaline-earth metals are also phosphorescent. (Cf. J.C.S., Dec.)—E. H. R.

Zinc, cadmium, and mercury sulphides; Fusion of —. E. Tiede and A. Schleede. Ber., 1920, 53, 1717—1721.

THESE sulphides, which sublime without melting under atmospheric pressure, were fused by heating

in a carbon-tube furnace under pressure in an atmosphere of nitrogen. Zinc sulphide melted under 100—150 atm. at 1800°—1900° C.; the fused solid had a light greenish-yellow lustrous appearance and proved to be the hexagonal modification. Cadmium sulphide melted at about 1750° C. under 100 atm.; it had a dark brownish-yellow lustrous appearance when solidified. Mercuric sulphide was fused at 1450° under 120 atm.; it formed a matt, steel grey solid which quickly became red when rubbed.—E. H. R.

White arsenic; Manufacture of —. E. C. Williams. Eng. and Min. J., 1920, 110, 671—673.

To obtain a pure, dense arsenic trioxide from blast-furnace flue dust and Cottrell plant dust careful regulation of the draught on the furnace from which the arsenic trioxide is sublimed, and efficient and quick cooling are essential. To accomplish this, the cooling chambers should be as deep as possible and their width should increase away from the furnace, with which they should be connected in a straight line by a flue, 2 ft. 6 in. square and about 20 ft. long.—A. R. P.

Tin; Precipitation of — by iron. I. M. Kolthoff. Rec. Trav. Chim., 1920, 39, 606—608.

WHILE agreeing with Bouman (J., 1920, 569a) that under certain conditions iron is capable of precipitating tin from solution, the author concludes from his own experiments that such precipitation does not occur in hydrochloric acid solution.—W. G.

Hydrogen cyanide; New synthesis of — by catalysis. A. Mailhe and F. de Godon. Sealed packet 223, 28.2.18. Bull. Soc. Chim., 1920, 27, 737—739.

WHEN carbon monoxide and ammonia are passed together over thoria at 410°—450° C., an excellent yield of ammonium cyanide is obtained.—W. G.

Cyanic acid; Detection of —. R. Fosse. Comptes rend., 1920, 171, 635—637.

IF the solution shows an increase in urea content, as evidenced by an increase in the weight of xanthylurea obtained in the xanthidol test, after heating with ammonium chloride for 1 hr., cyanic acid is present. A second method is to precipitate the cyanic acid as silver cyanate. The precipitate is divided into two parts; one is heated directly with aqueous ammonium chloride, the other is first heated with nitric acid and then with ammonium chloride. Both solutions are then tested for the presence of urea by means of xanthidol. A positive result with the first solution and a negative result with the second is evidence of the presence of cyanic acid in the original solution.—W. G.

Silicic acid; A new form of —. R. Schwarz and O. Liede. Ber., 1920, 53, 1680—1689.

By leading silicon fluoride into boiling water a new form of silicic acid was obtained, for which the term β -silicic acid is proposed. It dissolves less readily in hydrofluoric acid or in sodium hydroxide or ammonia solution than the normal α -acid. Conductivity experiments on solutions of the β -acid in ammonia indicate that it is a more highly polymerised form, analogous in some respects to meta-stannic acid. (Cf. J.C.S., Dec.)—E. H. R.

Lead hydride. F. Paueth and O. Nörring. Ber., 1920, 53, 1693—1710.

By methods similar to those employed for the detection of the hydrides of bismuth (Ber., 1918, 51, 1704, 1728) and tin (Ber., 1919, 52, 2020), the existence of a gaseous hydride of lead has been proved. The hydride was first prepared by the action of dilute hydrochloric acid on an alloy of bismuth and radio-lead (thorium B); a minute

trace of lead hydride in the resulting gas was detected by a test similar to the Marsh test for arsenic. The lead hydride could not be obtained by the action of acids on ordinary lead-magnesium alloys, but traces were obtained by a combined electrolysis-spark process. (*Cf.* J.C.S., Dec.)

—E. H. R.

Hydrides; Which elements form gaseous —?
F. Paneth. Ber., 1920, 53, 1710—1717.

THE discovery of volatile hydrides of tin, lead, bismuth, and polonium makes it possible to divide the periodic table into two portions, such that all the elements in one portion, and only one element, boron, in the other portion, form volatile hydrides. In other words, the four elements immediately preceding a noble gas in the table, and, in addition, boron, all form volatile hydrides. (*Cf.* J.C.S., Dec.)

—E. H. R.

Radium; Microchemical reactions of —: its differentiation from barium by iodic acid. G. Demigès. Comptes rend., 1920, 171, 633—635.

ALTHOUGH radium salts give results identical with those of barium salts in most microchemical tests for barium, these two series of salts may be differentiated by the use of iodic acid (J., 1920, 469 A). With this reagent the salts of the two metals give definite and distinctly different microcrystalline precipitates, providing the concentration of the salt solution does not exceed 0.3%.—W. G.

Gases; Examination of naturally occurring —.
F. Henrich. Ber., 1920, 53, 1940—1949.

METHODS for collecting and analysing natural gases are described, and particularly an apparatus for removing nitrogen from the rare gases. By circulating through a tube containing a mixture of calcium and sodium in the ratio 10:1, heated to 500° C., in 10 to 15 mins. the whole of the nitrogen can be removed from, for example, a mixture containing 1% of argon. The gas from a cold spring near Leupoldsdorf, Bavaria, was found to contain 13% of oxygen, 86% of nitrogen, and 1% of rare gas, chiefly argon. (*Cf.* J.C.S., Dec.)

—E. H. R.

Blast-furnace gas. Hutchinson and Bury. See X.

Determination of potassium. Morris. See XXIII.

Magnesium ammonium phosphate. Kunz-Krause. See XXIII.

PATENTS.

Nitric acid; Production of — [from ammonia].
General Chemical Co., Assees. of F. W. de Jahn. E.P. 124,759, 28.2.18. Conv., 3.11.17.

PURIFIED ammonia is oxidised by mixing it with air and passing the mixture over a catalyst. The nitrogen oxides formed are cooled and partly condensed, after which the residual gases are absorbed by water. The dilute nitric acid produced both by condensation and absorption is conducted into a tower packed with suitable trays containing hot sulphuric acid, and is vaporised by a downward stream of sulphuric acid, the vapours passing from the top of the tower to a condensing system. Nitric acid containing 97% HNO₃ is obtained by this process.—W. J. W.

Nitric acid and sulphuric acid; Apparatus for the recovery of — from waste acids. C. F. Baer. G.P. 323,416, 27.2.19.

A DENITRATION plant comprises a tower built up of sections with superposed, perforated plates arranged internally at 5—7.5 mm. distance from the walls. The plates have large central openings for the

passage of nitrous vapours, and smaller ones for the sulphuric acid flow, with partition walls between the sets of openings dividing the plates into two parts.—W. J. W.

Sulphuric acid and hydrochloric acid; Process of obtaining — from alkaline-earth salts. J. Behrens. G.P. 303,922, 30.6.17.

ALKALINE-EARTH salts are caused to interact with a weak basic oxide in a neutral salt solution and in presence of carbon dioxide. The alkaline-earth carbonate formed is separated and the metallic salt is decomposed by suitable means. Sulphuric anhydride is thus produced by introducing calcium sulphate into ferrous sulphate solution containing reduced ferrous oxide, and passing in carbon dioxide. Air is then blown through the filtered solution, and the ferric sulphate is decomposed. For the production of hydrochloric acid magnesium chloride is made to react with aluminium hydroxide in aluminium chloride solution.—W. J. W.

Automatic control of the production of a product [e.g., sulphuric acid by the contact process]; Means for — depending on the change produced in radiant energy by the presence of undesirable substances in the initial material. L. Logan. E.P. 151,328, 18.6.19.

LIGHT or other radiant energy is thrown upon a surface or container adapted to receive an automatically taken sample of the material to be controlled. A selenium or potassium-sodium alloy cell responds to changes in the transmitted light and regulates the supply of the material. For example, in the contact process for sulphuric acid the supply of burner gas can be automatically by-passed if arsenic is present by utilising the mirror of arsenic deposited in the Marsh test to intercept the light. The device is also applicable to impurities which alter the specific rotatory power or refractive index of a liquid, or which yield a coloured product with chemical indicators.—C. I.

Alkali aluminates; Preparation of pure —.
Rochette Frères. E.P. 141,666, 3.12.19. Conv., 16.4.19.

LIME or baryta and bauxite are fused with carbon in an electric furnace. Pure calcium or barium aluminate is formed and floats on the top, and a ferrous alloy containing silicon, titanium, etc., is drawn off at the bottom. From the calcium or barium aluminate alkali aluminates are obtained by interaction with alkali carbonate.—C. I.

Alkali silicates; Manufacture of readily soluble —.
F. J. Phillips and E. J. Rose. E.P. 151,339, 19.6.19.

A MIXTURE of silica and alkali containing 3 pts. of SiO₂ to 1 pt. of Na₂O or K₂O is mixed with 0.5—10.0% of borax and fused. The mass in a viscous state is rolled, dropped into water, and ground till dissolved. Sulphuric or other acid is then sprayed into the solution with continuous agitation until the gelatinous silica is redissolved, and the solution is evaporated.—C. I.

Comminution and mixing of materials [alkali silicates]. F. J. Phillips and E. J. Rose. E.P. 151,508, 28.11.19.

PARTIALLY disintegrated alkali silicate is introduced with water into a closed rotating cylinder, the axis of which is formed of a perforated pipe through which steam is supplied. The steam pipe may be surrounded by a perforated sleeve, and the perforations in the two pipes may be caused to register or not as desired. A safety valve may be provided to maintain a predetermined steam pressure in the cylinder.—B. M. V.

Absorbent [soda-lime]. R. E. Wilson. U.S.P. 1,333,524, 9.3.20. Appl., 15.11.18.

SODA-LIME in the form of hard, uniform grains, specially efficient as an absorbent, and offering low resistance to the passage of gases, is prepared by mixing 300 lb. of slaked lime with 60 lb. of a 20% solution of sodium hydroxide and 120 lb. of water, forming the mixture into slabs, 1½ in. thick, drying till the water content is reduced to 14%, allowing to cool for several hours, grinding, and screening to 8—14-mesh size.

Potassium chloride; Process for obtaining — [from furnace dust]. E. Anderson and F. S. Moon, jun., Assrs. to International Precipitation Co. U.S.P. 1,354,642, 5.10.20. Appl., 8.3.19.

FURNACE dust containing potassium sulphate and chloride is washed with calcium chloride solution to convert the potassium sulphate into chloride, after which the solution is separated from solid matter and the potassium chloride recovered.—W. J. W.

Potassium chloride; Method of recovering — from brine. J. L. Silsbee. U.S.P. 1,353,283, 21.9.20. Appl., 22.10.19.

BRINE containing sodium, potassium, and magnesium chlorides is evaporated at atmospheric temperature until nearly saturated with respect to potassium chloride, and after separating precipitated sodium chloride is further concentrated at atmospheric temperature to precipitate mixed chlorides of potassium and sodium without precipitating magnesium salts.—C. I.

Nitrogen compounds [cyanides] Process of forming —. C. B. Jacobs, Assr. to Air Reduction Co., Inc. U.S.P. 1,354,561, 5.10.20. Appl., 19.8.16.

CARBONACEOUS material, such as coke, is intimately mixed with an alkali metal compound, and the mixture, while in a pulverulent condition, is heated to expel its volatile constituents. The porous coke, enclosing the alkali metal compound, is then heated to, and maintained at, an elevated temperature, and nitrogen is introduced into the mixture.

—W. J. W.

Ammonia; Production of — [from crude cyanides]. F. J. Metzger, Assr. to Air Reduction Co. U.S.P. 1,354,574, 5.10.20. Appl., 15.10.18.

CYANIDE furnace products containing iron are subjected to the action of dry steam at a temperature above that of condensation of the steam and below 500° C., whereby loss of ammonia by formation of ferrocyanide is prevented.—A. de W.

Nitre-cake; Process of obtaining a liquid of high acidity from —. W. C. Holmes, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,354,649, 5.10.20. Appl., 27.11.18.

By continuous treatment of nitre-cake with water in such quantities as to prevent complete solution, a solution of 20—30% acidity is obtained.

—W. J. W.

Bismuth oxide; Making —. E. R. Darling, Assr. to Ellis-Foster Co. U.S.P. 1,351,806, 5.10.20. Appl., 4.1.19.

YELLOW bismuth oxide is prepared by fusing bismuth with sodium nitrate and passing chlorine into the fused material.—C. I.

Aluminium chloride; Method of manufacturing —. F. C. Frary, Assr. to Aluminium Co. of America. U.S.P. 1,354,818, 5.10.20. Appl., 26.5.20.

CHLORINE and a reducing agent are added to a pulverulent mass of aluminium dross.—C. I.

Aluminium compounds for sizing papers and other purposes; Preparation of —. G. Muth. G.P. 323,626, 19.8.19. Addn. to 319,420 (J., 1920, 517 A).

AN excess of acid substances is employed in decomposing the original material, and the excess is afterwards so far neutralised that the final product remains liquid. Neutralisation is effected by means of magnesium oxide or carbonate or materials containing these, or with mixtures of magnesium compounds with basic compounds of the alkaline earths or alumina, e.g., aluminates or soluble aluminium hydroxide.—J. H. L.

Alkali sulphates; Manufacture of — from alkali chlorides by decomposition with alkaline-earth sulphates or magnesium sulphate. Salzwerk Heilbronn, G. Kassel, and T. Lichtenberger. G.P. 299,775, 11.6.15. Addn. to 289,746 (J., 1920, 405 A).

A MOLTEN mixture of alkali chlorides and sulphates of the alkaline earths or magnesium is superheated and agitated with steam, after which it is allowed to settle, and the pure alkali sulphate is separated from the residual alkaline-earth oxido or magnesia.

—W. J. W.

Cyanamide solutions; Manufacture of — from crude calcium cyanamide. H. Danneek, and Elektrizitätswerk Lonza, A.-G. G.P. 302,495, 13.3.17.

CRUDE calcium cyanamido is treated with carbon dioxide, or gases containing it, before being mixed with water.—W. J. W.

Amides of alkalis and alkaline earths; Manufacture of —. Badische Anilin- und Soda-Fabrik. G.P. 323,656, 24.5.14.

DRY ammonia gas is passed into solutions of alkali or alkaline-earth metals in anhydrous caustic alkalis. As the formation of amides takes place at about 275° C., loss of ammonia through dissociation is avoided.—W. J. W.

Sodium percarbonate; Preparation of a substance containing — from sodium peroxide and carbon dioxide. L. Schwedes. G. P. 324,569, 1.1.18.

CARBON dioxide is passed into a paste of sodium peroxide and alcohol. The heavy yellow peroxide changes to a white flocculent mass containing 50% of percarbonate with about 7% active oxygen.

—C. I.

Sulphur; Process for obtaining — from hydrogen sulphide, or mixtures containing it. Chem. Fabr. Rhenania, and F. Projahn. G.P. 298,844, 1.2.16.

THE gases to be treated are passed downwards through a layer of contact material, such as bauxite, of 30 cm. thickness, at about 330° C., and then through a lower layer of such greater thickness as to allow the gases to cool down to the desired minimum temperature, approximately 200° C. A conical heap of the same contact material may be placed round the charging hole of the furnace. The main reaction, $H_2S + O = H_2O + S$, proceeds most favourably at 300° C. The injurious secondary reaction $2H_2O + 3S = 2H_2S + SO_2$, is only feeble at 200° C., and in the lower layer of the contact material is largely replaced by the reaction $2H_2S + SO_2 = 2H_2O + 3S$.—W. J. W.

Sulphuric acid; Concentration of —. A. C. Bohre; K. C. Bohre, extrix., Assr. to Norsk Hydro-Elektrisk Kvaelfstofaktieselskab. U.S.P. 1,353,448, 21.9.20. Appl., 27.3.19.

SEE E.P. 132,704 of 1919; J., 1919, 817 A.

Thorium [compounds]; Manufacture of —. H. Wade. From Lindsay Light Co. E.P. 151,854, 19.11.19.

SEE U.S.P. 1,329,747 of 1920; J., 1920, 232 A.

Radium; Producing compounds of —. H. O. Hedström. U.S.P. 1,353,532, 21.9.20. Appl., 21.6.19.

SEE E.P. 136,768 of 1919; J., 1920, 156 A.

Aluminium compounds; Process of producing —. H. J. Goldschmidt, Assr. to Det Norske Aktieselskab for Elektrokemisk Industri, Norsk Industri-Hypotekbank. U.S.P. 1,354,824, 5.10.20. Appl., 19.3.17.

SEE E.P. 113,276 of 1918; J., 1918, 466 A.

Atomising liquids. G.P. 302,554. See I.

Recovering ammonia. E.P. 136,834. See IIA.

Ammonia from gas liquor. G.P. 324,582. See IIA.

VIII.—GLASS; CERAMICS.

PATENTS.

Glass; Reduction in size of the bubbles contained in —. Schott und Gen. E.P. 147,487, 8.7.20. Conv., 7.12.14.

THE glass is subjected, when in the softened state, to a pressure of at least 200 atm. The reduction in size of the bubbles is partly due to the compression of the gases forming the bubbles, and partly to the absorption of the gases by the glass. —H. S. H.

Kilns. T. West and J. Hodgson. E.P. 151,709, 26.6.19.

THE kiln chamber is divided by partitions into five spaces, with fire-grates adjoining the outer spaces. The heating gases enter the latter near the top, descend, pass up through perforated floors into the intermediate spaces, and thence down the central chamber. From the bottom of this central space four flues extend to the top of each other chamber. Part of the heating gases from the central chamber passes through these flues and is used over again in the outer and intermediate chambers. Air enters under the fire-grate, passes up a hot air flue and enters the outer spaces at the top. Hot air is also allowed to enter the central chamber to mix with the heating gases which are conveyed by the four return flues to the outer chambers. Air flues above the fire-grate enable the process of "salting" to be carried out. —H. S. H.

Ovens; Continuous and intermittent —. Dressler Tunnel Ovens, Ltd., and J. Williamson. E.P. 151,722, 27.6.19.

TWO burners are placed at the middle of the length of the oven and on opposite sides of the goods spaces. They are fired from gas producers which form part of the side walls of the oven. Hot air can be supplied to the burners from the interior of the oven. The hot gases pass into heat accumulators built at the sides of the oven, and containing refractory chequerwork. These chambers are connected with an exhaust fan by means of horizontal pipes freely exposed to the interior of the oven at the inlet end, so that heat from the waste gases effects the preliminary heating of the goods. The heat accumulators are provided with inlet and outlet dampers which enable a transverse current of air, heated by the accumulator, to enter the oven and flow round the goods. When these dampers are closed the hot gases from the burner heat up the accumulator and pass to the

chimney. By means of reversing valves and dampers it can be arranged that one accumulator is heating up while the other discharges hot air into the oven. A more uniform and a higher temperature is attained by means of these devices, without increasing the amount of fuel used. When required, material for salt glazing is injected into the heat accumulator immediately after it is put into connexion with the oven. An oxidising or reducing atmosphere can be produced in the oven at will. —H. S. H.

Decorating kiln and the like. M. Solon, Assr. to The Potters Equipment Co. U.S.P. 1,354,518, 5.10.20. Appl., 8.12.17.

THE kiln is provided with heating flues in opposite walls, each flue having a heat supply connexion and an outlet connexion. A by-pass heating flue, placed in one of the outer walls, has its upper end connected with the other flues near the supply connexion, and its lower end connected with the same flues near the outlet connexion. —H. S. H.

Tunnel-kiln. P. Dressler, Assr. to American Dressler Tunnel Kilns, Inc. U.S.P. 1,354,541, 5.10.20. Appl., 22.1.19.

COMBUSTION chambers are arranged on each side of the interior of the inclosing structure of a tunnel kiln. Each combustion chamber is divided into two sections connected by a sleeve, which permits relative movement between the sections. Means are provided for introducing gas and highly heated air into one section of each chamber, and of projecting the gases longitudinally into the adjacent section of the chamber. —H. S. H.

Glass bait. E. C. R. Marks. From Universal Glass Co. E.P. 151,361, 23.6.19.

Glass; Manufacture of plate and other window — and apparatus therefor. J. Ramsey. E.P. 151,543, 13.3.20.

Glass bulbs and other necked glass vessels; Methods of and apparatus for operating upon —. British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 151,771, 25.7.19.

Glass gathering and blowing machines. W. E. Evans. From Europaischer Verband der Flaschenfabriken Ges. E.P. 151,831, 16.10.19.

IX.—BUILDING MATERIALS.

PATENTS.

Wood; Preserving and fireproofing of —. I. De Vecchis. E.P. 151,661, 3.3.19.

THE wood is placed in a hermetically sealed container and the sap removed by reducing the pressure. A little free alkali is added to a solution of double sulphates or chlorides of copper, zinc, and magnesium with ammonium, and a suitable anti-septic, and the solution is sucked into the container, which is then subjected to a pressure of 8 to 14 atm. The wood after removal is air-dried until it has been reduced to the weight which it would have had if it had been seasoned in the ordinary way. —H. S. H.

Road-making or paving materials, and method of manufacturing the same. C. Honda. E.P. 151,852, 15.11.19.

ROAD-MAKING or paving materials are made by mixing tarry matter with table salt, rosin saponified with slaked lime, and rosin sulphuretted with sulphur powder, clay being also added if desired. The salt increases the viscosity of the tar and improves its impregnating and solidifying power, the

saponified rosin acts as a binding material, and the sulphuretted rosin protects the surface from being affected by temperature changes.—H. S. H.

Calcium-sulphate products [plaster]; Retarder for —. F. E. Culver. U.S.P. 1,330,058, 10.2.20. Appl., 17.3.19.

The setting of plaster or the like is retarded by addition of vegetable gluten dissolved in potassium or sodium hydroxide solution and mixed with lime.

Cement raw materials; Treatment of —. H. Cappenberg. G.P. 323,290, 29.1.19.

Dry ferric chloride, or a mixture of aluminium chloride and ferric chloride, or a solution of both salts, is added to cement raw material before burning. The salts promote the evolution of carbon dioxide from the material, and a porous clinker is obtained.—W. J. W.

Concrete blocks, slabs, etc.; Manufacture of —. G. Schalk. G.P. 323,526, 12.4.19.

Pumice sand heated to 1000° C. is mixed with sand etc. in a drying and mixing machine, and the product is incorporated with a prepared mixture of cement and water at about 80° C.—W. J. W.

Light concrete for ship-building; Process for manufacturing —. A. Hambloch. G.P. 323,643, 13.3.19.

VOLCANIC sand, pumice, tufaceous limestone, or granulated slag is mixed with lime and formed into lumps by shaking, after which these are hardened by means of steam under pressure, reduced to small size, and mixed with cement.—W. J. W.

Potland cement; Utilisation of low grade carboniferous material in manufacture of —. R. W. Lesley. E.P. 152,106, 4.7.19.

SEE U.S.P. 1,323,294 of 1919; J., 1920, 65 A.

Sulphite - waste - liquor preparation. U.S.P. 1,327,862. See V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Iron ores; Microscopical examination of — with special reference to its value in dressing processes. H. Schneiderhöhn. Stahl u. Eisen, 1920, 40, 1261—1265.

THE author recommends the use of the microscope to investigate the nature of raw materials, products, and working methods. It is of service in determining if the concentration of low-grade ores is an economical proposition, in determining mineralogical composition, and in ascertaining whether ores are suitable for magnetic separation. It has also proved of value in determining the iron content of mixed iron ores. From the microscopical appearance the nature of the gangue can be ascertained, and hence its specific gravity and probable behaviour in wet mechanical separation. Separation processes may be controlled by microscopical examination of the products at different stages, and the method of combination of valuable and useless components in iron ores, e.g., the form in which manganese and phosphorus are present, may be ascertained. The microscope is also of service in examining the structure and intermixture of crushed and intermediate products, in controlling the briquetting and agglomeration of concentrates, and in the examination of slags.—T. H. Bu.

Blast-furnace gas; Rough-cleaning of — at Skinningrove by the Lodge electrostatic process. A. Hutchinson and E. Bury. Iron and Steel Inst., Sept., 1920. [Advance proof.] 18 pages.

THE electrostatic plant is divided into sixteen cleaning units, each with two discharge grids placed in series. This multiplicity of chambers was adopted to ensure maximum contact between the electrostatic discharge and the gas to be cleaned by splitting up the gas flow into multiple streams; also to avoid passing large quantities of dirty gas into the system when cleaning the chambers (during which operation the discharge must be cut off), which would have been the case if a smaller number of larger chambers had been employed. The pressure employed is 40,000 volts. The entire make of gas from 3000 tons of pig iron per week is treated with a consumption of 50 kw. Each cleaning chamber is provided with two cleaning hoppers, into which the dust is shaken from the grids by rappers. The dust content of the gas is reduced from 5–6 g. to 0.8–1.1 g. per cb. m. with 15–20% of the plant not yet in action. When the plant is complete it should be possible to keep down the dust content to 0.5–0.7 g. per cb. m. The dust (recovery, 48–50 tons per week) is removed every 8 hrs. and is taken to the potash extraction plant by scraper conveyors. The potash extraction plant consists of mixers for lixiviating the soluble salts from the dust, revolving filters for separating the soluble matter from the solution of chlorides, and two Kestner evaporators. Potassium and sodium chlorides are separated by fractional crystallisation from calcium chloride, and potassium chloride is further fractionated from sodium chloride. The potassium chloride recovered amounts to 27% of the dust. The sensible heat of the gas is not greatly diminished, the temperature of the raw gas being 220°–250° C. and of the cleaned gas 200°–220° C.—A. G.

Soft iron. F. Goerens and F. P. Fischer. Elektrochem. Zeits., 1920, 27, 1–5, 11–16.

TESTS were made on a soft iron prepared by the open-hearth process and having an average composition C 0.057%, Si 0.01%, Mn 0.097%, P 0.01%, S 0.021%. The structure consisted of coarse ferrite crystals which increased in size towards the edge. Tests on rolled and forged bars and plates gave an average yield point of 21 kg., a breaking stress of 31 kg., an elongation of 47%, and a contraction of 80%, and compared favourably with tests made on copper bars. Impact tests on differently notched bars gave good results, and bending tests with plain and screwed bars were also satisfactory, the results being similar to those obtained with copper. Repeated bending tests gave a superior figure to similar tests on copper, while welded bars bent through 180° without fracture. Incorrect heat treatment or work developed a coarse crystalline structure resulting in brittleness, but this could be avoided or removed by reheating at temperatures not less than 920° C.—J. W. D.

Gases from iron made by various processes. E. Piowarsky. Stahl u. Eisen, 1920, 40, 1365–1366.

THE author has determined the percentage composition of the gases given off by Thomas pig iron, avoiding the experimental error of allowing access of air to the molten metal by the use of a special chill mould. A stream of nitrogen was passed through the top of the mould while it was filling with metal, thus preventing action between the carbon of the pig iron and the oxygen of the air. Samples of the gases were taken at the beginning, middle, and end of tapping, five being taken in each case at intervals of a minute. The composition of the gases at these three stages was: CO, 0.40–0.85, 0.45–0.74, 0.30–0.48; CO₂, 24.0–31.4, 36.1–41.9, 42.2–43.9; H₂, 30.8–49.6, 42.8–49.7;

44.1—48.3; and N_2 , 18.6—44.3, 9.8—19.8, 9.1—12.5% respectively. In the most favourable cases the sum of the hydrogen and carbon monoxide amounted to 88.5%, while in Munker's experiments (Stahl u. Eisen, 1904, 23) 70% was reached only in the case of spiegel. Very little gas was given off after the pig iron had solidified.—T. H. Bu.

Steel; Physical properties of arc-fused — H. S. Rawdon, E. C. Groesbeck, and L. Jordan. Chem. and Met. Eng., 1920, 23, 677—684.

TEST specimens were machined from blocks of arc-fused metal, built up by depositing the fused metal as a series of "beads," so arranged that they were parallel with the axis of the tension specimen which was cut later from the block. Two series, representative of a "pure" iron and a low-carbon steel, as used for welding electrodes, were examined. The general effect of the fusion was to render the two materials more nearly of the same composition, due chiefly to the loss of carbon and silicon by oxidation. A slight coating on the electrode before fusion had little influence in preventing this oxidation. The most noticeable change in composition was the increase in nitrogen content of the metal, following roughly the increase in current density. All the specimens contained a considerable number of cavities and oxide inclusions and no definite relation between "soundness" and current density within the range employed was shown. When stressed in tension, low ductility values were given; the elongation of the test specimen appeared to be a measure of the adhesion between the successive added layers, being due largely to the cumulative effect of numerous imperfections rather than to the real property of the metal itself, which, judged by its behaviour on bending, was inherently ductile. It is seemingly impossible to fuse the metal without serious imperfections, which are so gross that the changes in chemical composition which the fusion entails, together with the accompanying unusual features of micro-structure, are of minor importance in determining the strength, durability, and other properties of the arc weld.

—C. A. K.

Corrosion of iron and steel; Influence of copper, manganese, and chromium on the — E. A. and L. T. Richardson. Trans. Amer. Electrochem. Soc., 1920, 123—135. [Advance copy.]

COPPER and manganese have a mutual action in their effect on the atmospheric corrosion of iron. Copper alone reduces the corrosion of pure iron, and to a still greater extent that of steel containing manganese, the latter enhancing the effect of the copper. If manganese is replaced by chromium the effect is still more pronounced. The authors consider that in commercially pure iron the presence of copper produces an inter-grain film of oxide or other compound of iron, and that this film confers rust-resisting properties on the iron. Between 800° and 1000 C. this inter-grain material becomes weak and plastic, producing red-shortness in the iron. Additions of manganese and chromium result in a smaller grain size and consequently increase in resistance to corrosion. These elements also lower the red-short range of temperature, and if present in sufficient quantities this range occurs at a temperature below that at which the inter-grain film becomes weak or plastic.—J. W. D.

Metals; Solution of — in acids as related to corrosion. W. D. Richardson. Trans. Amer. Electrochem. Soc., 1920, 153—186. [Advance copy.] (Cf. J., 1920, 600 A.)

CORROSION and solution of metals in acids may be considered as actions tending to go forward at rates proportional to solution tension and hydrogen ion concentration, but subject to the accelerating and retarding influence of many substances acting as

catalysts. Cast metals are relatively less attacked in the presence of much oxygen than the purer rolled metals. They are also relatively less attacked by an oxidising acid, such as nitric acid, than by the non-oxidising sulphuric and hydrochloric acids. Silver, copper, and formaldehyde, as catalysts, all reverse this action and cause the rates of solution of the cast metals in nitric acid to be increased and the rates of solution of copper-bearing and pure open-hearth irons to be reduced. Platinum, while catalysing the rolled metals negatively in nitric acid, catalyses them positively in sulphuric and hydrochloric acids, and is without action on the cast metals in any acid. Nickel as a catalyst produces no effect on any of the metals in any of the acids except on aluminium in hydrochloric acid, in which case the rate of solution is greatly increased. Aluminium is not affected by any of the catalysts in nitric or sulphuric acids.—J. W. D.

Zinc retorts; Determination of the thermal condition of the charge in — O. Mühlhaeuser. Metall u. Erz, 1920, 17, 209—212.

DURING a zinc distillation the temperatures of the retort and combustion chamber were recorded at the end of each half-hour. On charging the retort the temperature of the combustion chamber falls rapidly at first, remains constant at about 1050° C. for a considerable time, and then rises slowly to the maximum (1300° C.). The temperature of the retort charge rises slowly to about 1000° C., where it remains constant for some hours due to the reduction of the zinc oxide to metal, which reaction absorbs 89,600 cal. The temperature then slowly rises while the zinc distils, to a maximum 100°—150° C. below that of the combustion chamber.—A. R. P.

Zinc distillation; Determination of the quantity of gas evolved during — O. Mühlhaeuser. Metall u. Erz, 1920, 17, 415—420.

TABLES and graphs are given showing the temperature and pressure in the retort and the volume and nitrogen content of the gases evolved during the progress of the distillation of zinc from zinc oxide (roasted blende) and anthracite. The rate of evolution of the gases is greatest at the commencement of the operation, when the temperature of the retort has reached 1000° C., and falls rapidly during the next two hours, after which it again rises with the rise in temperature to a second maximum, which, however, is much lower than the first. The rate then gradually decreases until the end of the distillation. The nitrogen content of the gases is at a maximum at the beginning, then slowly decreases until the middle of the distillation, after which it again rises to a maximum at the end of the operation.—A. R. P.

Aluminium casting alloys; Rapid method for the analysis of light — R. M. Berry. J. Ind. Eng. Chem., 1920, 12, 998—1000.

THE alloy (1 g.) is dissolved in 20 c.c. of 25% sodium hydroxide solution, the solution immediately diluted to 300 c.c., and filtered and the insoluble residue (containing Sn, Cu, Pb, Fe, Ni, Mg, part of the manganese, and traces of aluminium) washed at least ten times with 1% sodium hydroxide solution. The filtrate (containing about 99% of the aluminium and zinc, and the remainder of the manganese) is treated with formic acid to give an excess of 25 c.c., and then with hydrogen sulphide to precipitate the zinc as sulphide, which is dissolved in acid and the solution titrated with ferro-cyanide. The original alkali-insoluble portion is digested with nitric acid and the tin extracted from the residue by means of ammonium sulphide and chloride solution, and precipitated as sulphide, which is ignited to SnO_2 . The nitric acid solution is electrolysed to determine copper and lead, the

iron and aluminium separated from the exhausted electrolyte, and the iron determined by titration with permanganate. Nickel is determined in the filtrate from the iron and aluminium by means of dimethylglyoxime, and the magnesium determined in the filtrate from the latter operation by precipitation with ammonium phosphate. To determine manganese the alloy is treated with sodium hydroxide solution and then with nitric acid, and the solution treated with silver nitrate, oxidised by means of ammonium persulphate, and titrated with arsenite solution. Silicon is determined by dissolving the alloy in sulphuric and nitric acids, adding hydrochloric acid, evaporating, extracting the residue with hydrochloric acid, and filtering off and igniting the silica.—C. A. M.

Copper-aluminium alloys or hardeners; Manufacture of rich —. R. J. Anderson. Chem. and Met. Eng., 1920, 23, 617–621.

For the manufacture of rich copper-aluminium alloys containing 33, 50, or 67% Cu, the following method is recommended. About one-third of the aluminium is melted in an iron pot, cold pigs of aluminium are then thrown in to cool the metal, after which a quarter of the molten copper is poured in. The temperature rises owing to the heat of combination of the two metals; more cold aluminium is therefore added until some solid metal is seen floating in the molten alloy. Another quarter of the copper is then poured in and the process repeated until the whole of the metals have been added and thoroughly mixed, when the alloy is poured or ladled into pigs. The eutectic alloy (67% Al, 33% Cu) melts at 540° C., that containing 50% Cu at 575° C., and that containing 60% Cu at 658° C. All the alloys are brittle and may be readily broken up for use in hardening aluminium. —A. R. P.

Ternary alloys and coefficients of equivalence. L. Guillet and A. Portevin. Rev. Mét., 1920, 17, 561–567.

A THEORETICAL and mathematical consideration of the principles governing the influence of the addition of a third metal to a binary alloy such as brass. This addition of a third metal may have the effect of displacing the eutectic point in the diagram to the left or right, so that the resulting alloy under the microscope exhibits a structure corresponding, in the case of brass, to a copper content quite different from its real content of copper. The relation between the two may be expressed by the equation $A' = \frac{100A}{100+q(t-1)}$ where A'

and A are the apparent and real percentages of copper, q is the quantity of the third element added, and t is its coefficient of equivalence, i.e., the proportion of zinc which replaces 1% of the element in the alloy of apparent composition A' . In the triangular diagram of a ternary alloy it is possible to draw isomicrographic lines through points representing the composition of alloys having similar micrographic structures; the general equation of the lines is $\frac{A}{A'} + q \frac{(1-t)}{100} = 1$.—A. R. P.

Molybdenum, cobalt, and chromium alloys; Analysis of —. J. R. Camp and J. W. Marden. J. Ind. Eng. Chem., 1920, 12, 998.

A SMALL quantity of the alloy is dissolved in a few c.c. of *aqua regia*, 3 c.c. of sulphuric acid added, and the liquid evaporated until white fumes appear, diluted to 100 c.c., and transferred to a small pressure bottle. The solution is treated for 5–10 mins. with hydrogen sulphide, and the bottle closed and placed for an hour in boiling water. The

precipitated molybdenum sulphide is washed free from acid, redissolved, reprecipitated and again washed, and finally ignited for 2 hrs. and weighed as MoO_3 . The filtrate is boiled, treated with sodium peroxide, and again boiled, and the precipitated cobalt hydroxide washed with dilute hydrogen peroxide, the black cobaltic hydroxide ignited, and the residue of Co_2O_3 weighed. As a rule it is necessary to dissolve the precipitated hydroxide in dilute hydrochloric acid, and to reprecipitate it. The filtrate is acidified, boiled to remove hydrogen peroxide, and the chromate titrated with ferrous ammonium sulphate solution either by an electrometric method or with potassium ferrieyanide as outside indicator.—C. A. M.

Zinc, cadmium, and mercury sulphides. Tiede and Schleede. See VII.

Thermo-couples. Wilson and Epps. See XXIII.

Zirconium. Smith and James. See XXIII.

PATENTS.

Flotation separation of mineral substances. T. H. Palmer, H. V. Seale, and R. D. Nevett. E.P. 132,260, 3.9.19. Conv., 2.9.18.

ONE containing mixed sulphides is subjected to a flotation treatment in an aqueous medium containing elemental sulphur, added in proportion of 0.5% of the quantity of ore, whereby one of the sulphides is removed, the residue being subjected to further flotation in the same medium with the addition of an acid or a frothing agent or both and with the temperature raised.—J. W. D.

Furnace for the heat treatment of steel, iron, or the like. J. R. C. August. E.P. 151,323, 17.6.19.

A REMOVABLE muffle is supported concentrically within a furnace chamber by a number of firebrick partitions which form flues for the heating gas, the latter being generated by the burning of solid fuel in a fire-box at the rear and introduced by a port-hole into one of the longitudinal flues, or by the burning of liquid or gaseous fuel in a burner placed longitudinally below the muffle. The furnace is wound with electric coils so arranged that the iron or steel inside is located in the magnetic field produced by the current, the critical temperature, i.e., the magnetic change point, being indicated on a galvanometer.—J. W. D.

Tin scruff produced in the making of tin andterne plates; Treating —. G. H. Clegg. E.P. 151,374, 25.6.19.

THE scruff is crushed, agitated with cold water in a tank to dissolve the zinc chloride, and the lighter particles of sludge separated from the zinc chloride in the liquid drawn from the tank by means of a vacuum filter or filter-press. The sludge remaining in the tank is ground and agitated with cold water till pasty, the heavier particles of tin screened off, and the metallic oil soap or "tin soap," consisting of tin oxide, palmitin, and palmitic acid, separated into those constituents by filtration and treatment with hydrochloric acid.—J. W. D.

Sherardizing; Drums for —. S. O. Cowper-Coles. E.P. 151,426, 6.8.19.

A ROTATING drum is fitted with a central longitudinal tube, the annular space around it forming a chamber for the articles to be sherardised and the zinc dust. The tube is heated either by gas jets, electric heating coils, circulating hot oil, or superheated steam, the heat passing into heat-insulated chambers at either end and thence through a series of tubes extending longitudinally through the annular space and through an outer jacket on the drum.—J. W. D.

Sulphide ores; Treatment of argentiferous —. F. E. Elmore. E.P. 151,698, 23.6 and 18.12.19.

THE argentiferous lead-zinc sulphide residue obtained from the acid brine process (E.P. 6546 of 1917; J., 1919, 541 A) is heated with sodium chloride or other suitable halogen salt in the presence of air at a temperature sufficiently high to cause volatilisation of a substantial proportion of the lead as chloride or oxychloride. The silver and the remaining lead are then extracted from the residue by leaching with a strong solution of calcium or magnesium chloride containing hydrochloric acid, cupric chloride, or both, not greatly in excess of that required to convert into chloride the lead present.—J. W. D.

Electrode; Electrical-arc-welding — and process of making. J. Churchward, Assr. to Wilson Welder and Metals Co. U.S.P. 1,333,586, 9.3.20. Appl., 21.7.19.

A "sull finish" or rust coating is formed on a welding rod of iron or the like, which is then immersed in a hot bath of milk of lime and afterwards baked in order to fix the coating of lime.

Briquetting of ores. F. A. Vogel, Assr. to General Briquetting Co. U.S.P. 1,334,331, 23.3.20. Appl., 26.10.18.

IN making ore briquettes with coal as a binding agent the mixture of ore and coal is warmed and dried, mixed with a regulated quantity of a "stimulant," e.g., tar oil, subjected to the action of moisture, and the moistened material subjected to an intimate grinding and mixing operation until it is entirely converted into a "coherable mass possessing a cementing facility," after which the mass is formed into briquettes.

Ores; Process of treating [sintering] —. J. E. Greenawalt. U.S.P. 1,348,408, 3.8.20. Appl., 25.11.12.

FUEL and combustion-supporting gases are projected into a chamber above the charge of ore, the fuel is ignited, and the burning mixture is drawn downwards through the charge of ore.

Zinc-reduction furnace. J. D. Shipton. U.S.P. 1,353,362, 21.9.20. Appl., 10.6.19.

A SERIES of retort tubes extend across the furnace and are connected with a corresponding series of condensers. At the other end each tube is fitted with a door for charging. All the condensers are connected together to form a common condensing surface and means are provided for withdrawing the condensed metal.—J. W. D.

Alloys. F. Milliken, Assr. to F. Milliken, S. F. Weaver, and J. M. Repplier. U.S. Pats. (A) 1,354,988, (b) 1,354,989, and (c) 1,354,990, 5.10.20. Appl., 8.10.19.

THE alloys have the following compositions:—(A) Cu 50–60%, Ni 28–36%, Zn 4–8%, Fe 4–8%; (b) Cu 55–65%, Ni 6–11%, Zn 14–18%, Pb 10–14%; (c) Cu 31–38%, Ni 38–46%, Fe 16–20%, Cr 5–7%, Mn 0.25–0.75%.—J. W. D.

Bauxite; Process of smelting — for production of ferro-aluminium in an electric furnace. Rheinische Elektrowerke A.-G. G.P. 308,542, 7.3.18.

By the addition of calcium compounds, especially lime, to bauxite in the electric furnace, the vapour tension of the alumina is appreciably diminished owing to formation of calcium aluminate, and a slag rich in calcium carbide is obtained as a by-product.—W. J. W.

Aluminium; Production of a brown colour on —. Hirsch, Kupfer- und Messing-Werke A.-G., and L. von Grotthus. G.P. 324,619, 15.11.18.

THE metal is submitted to electrolytic treatment in an ammoniacal solution of ammonium molybdate, through which hydrogen sulphide has been passed until a deep-red coloration has formed. This preliminary treatment facilitates deposition of the molybdenum.—D. F. T.

Alloys; Metal —. C. B. Backer. E.P. 151,400, 10.7.19.

SEE U.S.P. 1,341,774 of 1920; J., 1920, 549 A.

Ores; Apparatus for roasting or sintering —. J. E. Greenawalt. U.S.P. 1,348,407, 3.8.20. Appl., 18.11.10.

SEE E.P. 14,805 of 1912; J., 1913, 239.

Wire cleaning; Method of — and apparatus therefor. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 151,399, 10.7.19.

Annealing, tempering, or otherwise modifying the physical characters of metal bars or other metal articles; [Quenching] apparatus for —. J. R. C. August. E.P. 152,083, 3 and 21.7.19.

Hardness of metals. E.P. 151,383. See XXIII.

XI.—ELECTRO-CHEMISTRY.

Lead accumulators in works practice. A. Langer. Chem.-Zeit., 1920, 44, 749–750.

CHARGING accumulators until the plates evolve gas freely is injurious, since energy is wasted, and the cathodes tend to disintegrate. Experiments showed that by careful charging the temperature of the acid may fall slightly. Changes of concentration of the acid in different parts of the cell may occur. Detailed directions for the use of an accumulator battery are given.—J. R. P.

Chlorate and perchlorate. Knibbs and Palfreeman. See VII.

Ilust-furnace gas. Hutchinson and Bury. See X.

PATENTS.

Electric accumulators. G. Marconi. E.P. 151,293, 5.4.18. Conv., 5.4.17.

AN electric accumulator of the zinc-chlorine type comprises a number of porous cylinders contained within a cylindrical steel sheath coated internally with zinc. Each porous cylinder contains a carbon rod and is packed with powdered carbon. The steel sheath is closed below by an insulated cover. The porous cylinders are immersed in an aqueous solution of zinc chloride and chloride of calcium or other alkaline earth or alkali. A layer of zinc-coated wire gauze or carbon grains rests upon the porous cylinders and the solution referred to is poured thereon. A circulating tube is introduced axially into the liquid electrolyte, and contains another tube enclosing a heating coil, which is so connected that during charging and discharging of the accumulator circulation of the electrolyte is effected owing to the heat generated in the coil. The various carbon rods are electrically connected with the insulated bottom cover. The space above the electrolyte is filled with air or hydrogen at a pressure of about 20 atm. The steel sheath and the insulated bottom cover serve as the electrodes of the accumulator.—J. S. G. T.

Carbohydrates [wood]; Process of procuring and securing products from —. Means actuated by alternating electric current for controlling or operating electric furnaces or other mechanisms. J. H. Reid, Assr. to International Nitrogen Co. U.S.P. (A) 1,327,737 and (B) 1,327,738, 13.1.20. Appl., 13.1 and 19.12.14.

(A) A MIXTURE of a carbohydrate (wood, sawdust) and a metal oxy-compound (lime, calcium carbonate) is exposed to successive stages of heat, induced therein by increments of resistance to a current of electricity so varying in character as to yield successively products of progressively increasing carbon content; 5% of coke is added to the mixture of wood or sawdust and lime to reduce the initial resistance. The volatile products (methyl alcohol, acetone, acetic acid, tar) are condensed, and the temperature is then raised so as to convert the residue into calcium carbide. (B) The apparatus comprises a receptacle and electrodes therein, with means for bringing the electrodes and receptacle into an electrical circuit. A non-magnetic armature is adapted to be acted upon by an alternating current for repelling it, and means, governed by the temperature, are provided for regulating the movement of the armature, the latter controlling the operative distance of the electrodes when in use; means are also provided for reversing the movement of the electrodes while communicating with the controlling member.—B. N.

Air and gases; Process for treating — in the electric arc. K. Friedrich. G.P. 300,722, 25.1.16.

In conjunction with a vertical, inverted electric arc, an annular slotted device is arranged horizontally, or in a slightly inclined position, at the lowest point, through which gases, after being heated by the arc, are drawn and thus produce an electric disc-shaped light.—W. J. W.

Electrodes and carbon rods; Furnace for baking —. F. K. Meiser. G.P. 323,797, 25.9.17.

A DEVICE for collecting tar vapours is placed on the saggars. If this receiver is put on the furnace walls instead of on the saggars leakages are unavoidable.—W. J. W.

Electrodes and similar materials; Tunnel furnace for —. F. K. Meiser. G.P. 323,798, 28.2.18.

Is a furnace for the manufacture of electrodes the containers for the electrodes and the cars carrying them are provided below with outlets for vapours evolved, which outlets can be connected with a condensing and collecting system.—W. J. W.

Electric furnaces; Induction —. British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 152,157, 8.8.19.

SEE U.S.P. 1,326,120 of 1919; J., 1920, 196 A.

Electrolytic apparatus. C. M. Walter. U.S.P. 1,353,194, 21.9.20. Appl., 12.6.19.

SEE E.P. 127,985 of 1918; J., 1919, 585 A.

Electrodes of electrolytic gas generators. I. H. Levin. E.P. 151,873, 31.12.19.

Welding electrode. U.S.P. 1,333,586. See X.

XII.—FATS; OILS; WAXES.

Fats; Catalytic reduction of unsaturated —. A. S. Koss. Przemysl Chem., 1920, 4, 39—48. Chem. Zentr., 1920, 91, IV., 442.

THE catalytic activity of the oxides of nickel in the hydrogenation of oils at 255°—260° C. (cf. Bedford and Erdmann, J., 1913, 611) depends on the method

used to prepare them. Addition of nickel monoxide to the sesquioxide does not always increase the activity of the latter (cf. loc. cit.). The particular combination most suitable for the treatment of any given oil must be ascertained by experiment. Nickel sesquioxide loses its activity on heating in the air, even at 115° C. Its activity is considerably increased by very small quantities of platinum, and this fact may account for some of the results obtained by Bedford and Erdmann. The resistance of drying oils to catalytic reduction is due to the position of the double linkages in the linolenic acid molecule. Those between the 9th and 10th and the 12th and 13th carbon atoms are readily reduced, whilst that between the 15th and 16th is only very slowly attacked. The partially reduced oil therefore remains liquid owing to the formation of a liquid oleic acid. Ordinary oleic acid is readily reduced and possesses no drying properties. The drying properties of linolic acid are due to the double linkage between the 12th and 13th carbon atoms. The further a double linkage is from the carboxyl group the more difficult is it to reduce and the greater is the tendency to polymerisation. These differences in the behaviour of double linkages are well shown by means of a weak catalyst like nickel sesquioxide.—J. H. L.

Fatty oils; Polymerisation of —. J. Marcussen. Z. angew. Chem., 1920, 33, 231—232, 234—235.

OILS thickened by blowing followed by electrical treatment (F.P. 440,843; J., 1910, 222) are sold under the name of "Voltol" oils. The apparatus consists of a horizontal drum which is rotated on an axis upon which is a series of alternate metal and glass discs spaced a few mm. apart. The metal discs are connected with a source of electricity, and a current of about 8 to 10 kw. is passed through the blown oil at a reduced pressure (0.1 atm. or less). Samples of rape and marine animal oils thickened by this process had the following characters:—

	Sp. gr. 15° C.	n_D^{15}	Viscosity at 100° C.	Acid value.	Iodine value.	Mole- cular weight.
Thickened rape oil	0.9740	1.485	83.6	11.7	52	1200
Thickened marine animal oil	0.9819	1.485	74.9	15.4	51	1000

The high viscosity is due to the formation of oxidation products and to intramolecular polymerisation. Polymerisation of oils may also be effected by heating with aluminium chloride or iron chloride. For example, 10 g. of linseed oil is dissolved in 40 c.c. of benzene, and the solution treated gradually with 3 g. of aluminium chloride. In one experiment the viscous product had acid value 15.6, saponif. value 185, and iodine value 120. Toluene, xylene, naphthalene, anthracene, anisol, etc. may be used in place of benzene. Polymerisation of sterols as well as of glycerides takes place in the treatment of oils with aluminium chloride. Oil separated from the yeast *Endomyces vernalis*, after drying in presence of sodium chloride, was found to be polymerised; it had the following characters:—Acid value, 50; saponif. value, 196; and iodine value, 92.—C. A. M.

Iodine value of fats; Comparison of methods for the determination of the —. W. Devrient. Ber. deuts. pharm. Ges., 1920, 30, 361—366.

THE iodine value of elaidic acid, as determined by different methods, was found to be as follows:—Hübl, 80.3; Waller, 80.0; Winkler, 80.8; Wijs, 80.8; Hanus, 82.5. The Hanus method is considered to be

the most rapid and simple, and the result obtained is nearer the theoretical value (90) than are those obtained by the other methods.—W. P. S.

Gelatin as emulsifying agent. Holmes and Child. See 11A.

PATENTS.

Catalysts used in hydrogenation; Revivifying spent —. H. Schlinck und Co. A.-G. G.P. 324,580, 22.1.16.

THE catalyst is boiled with sufficient or less than sufficient acid to dissolve the metal, an alkali or alkali carbonate is added to precipitate the dissolved metal, and the precipitate, after separation from the solution by filtration, is washed and reduced by heating in a current of hydrogen; the temperature is allowed to rise at a rate not greater than 150° C. per hr., so that in 4—5 hrs. it reaches 650° C., at which point the heating is stopped.

—L. A. C.

Yeast masses; Extraction of fat from —. Kriegsausschuss für pflanzliche u. tierische Öle und Fette, G.m.b.H. G.P. 309,266, 31.1.18.

BEFORE extraction with solvents the yeast masses are allowed to undergo autodigestion, whereby the fat globules within the cells are liberated.—J. H. L.

Aluminium hydroxide; Washing and cleaning by means of —. M. Buchner. G.P. 323,193, 21.4.15.

THE material to be washed is treated with water containing aluminium hydroxide in the form of a gel, produced by adding ammonia to solutions of aluminium salts of medium concentration, e.g., 1—5%. The precipitates so formed are highly dispersive, but can be readily filtered and washed.

—J. H. L.

Bile soaps and detergents; Production of —. C. H. Boehringer Sohn. G.P. 323,804, 22.11.18.

ALKALI salts of hydrolysed bile acids, or their components separately, are added to a soap. Apart from their cleansing action, the products soften the fibres of the materials to be washed, without injuring them, and lessen the injurious action of inferior detergents.—J. H. L.

Glycerin substitute [from lactates]. Chem. Fabr. vorm. Goldenberg Geromont und Co. G.P. 303,991, 8.2.16.

AQUEOUS solutions of alkali lactates may be used in place of glycerol for many purposes, e.g., lubrication of machinery, softening of leather, etc. An 80% solution of sodium lactate boils at 126° C., and loses no water by evaporation at the ordinary temperature; a 50% solution does not freeze at -60° C.—J. H. L.

Glycerol; Production of pure — from liquids containing glycerol or from crude glycerol. Elektro-Osmose A.-G. (Graf Schwerin Ges.). G.P. 323,666, 14.2.19.

THE impurities are caused to separate in a flocculent form by addition of soluble silicates, preferably water-glass, with the aid of heat if necessary. Heating may be continued until a "crystallisation skin" forms on the surface of the liquid. The products may afterwards be purified by means of milk of lime or barium carbonate.—J. H. L.

Hydrogenating fatty acids and their glycerides; Method of and apparatus for —. A. Radisson (Assr. to Soc. de Stearinerie et Savonnerie de Lyon) and P. Berthou. U.S.P. 1,353,419, 21.9.20. Appl. 21.6.17.

SEE E.P. 107,969 of 1917; J., 1918, 553A.

Colours soluble in oils. E.P. 151,657. See IV.

Fat from bones. G.P. 323,650. See XIXA.

Fatty acids. G.P. 324,663. See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

White lead; "Porcelain" —. E. Euston. J. Ind. Eng. Chem., 1920, 12, 974—975.

THE hardness of the porcelain-like crust formed in the stack process of corrosion for the manufacture of white lead is due, not to its being a definite compound, $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$, as commonly assumed, but to the cementing effect of the colloidal basic lead acetate present in the formation of the crust. If, in the precipitation process, the compound, $2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$, is dried without previous washing, the degree of hardness of the mass is proportional to the concentration of the basic lead acetate; if thoroughly washed before drying a friable mass is obtained.—C. A. M.

Linoleum; Maturing of —. F. Fritz. Kunststoffe, 1919, 9, 253. Chem.-Zeit., 1920, 44, Rep., 227.

THE relationship between the time, T, necessary for the maturing of linoleum and the thickness, d (in mm.) of the material, is given approximately by the formula: $T = k \cdot d \cdot (d + 2) / 8$; the coefficient, k, for Walton goods lies between 10 and 12. The formula holds good up to considerable thicknesses such as 6.25 mm., but its application is restricted to homogeneous "through to the back" linoleums. Thick materials are commonly removed too early from the maturing rooms. Penetration of air into the linoleum is not the only factor to be considered; the gaseous products formed must also have easy egress. The influence of moisture is also an important factor; the air of the maturing room must be capable of carrying off moisture, a condition which does not often obtain with warm moist air. A deep-seated change is brought about by addition of drier to the mix. Hardening substances, such as the oxides of calcium, barium, magnesium, and zinc, produce an apparently matured linoleum in a shorter time, the hardness subsequently increasing with time. The action of light has no essential influence.—A. de W.

Zinc sulphide. Tiede and Schleede. See VII.

Polymerisation of oils. Marcusson. See XII.

Aldehyde-resins. Hammarsten. See XX.

PATENTS.

Gold-coloured sulphurs and vermilions of antimony; Manufacture of —. P. Chaillaux. E.P. 151,422, 1.8.19.

NATURAL antimony sulphide is melted by heating in a closed vessel, and then treated with gaseous sulphur dioxide or hydrogen sulphide or a mixture of these gases; the trisulphide is thereby converted into golden or crimson tetrasulphide and pentasulphide, which are collected as powder in a settling chamber. Instead of introducing sulphur dioxide as such, air or oxygen in suitable quantity may be led into a molten mixture of the natural trisulphide and sulphur.—D. F. T.

Coating material; Water-resistant — and method of making it. G. W. Miles, Assr. to Ross Chemical Co. U.S.P. 1,354,575, 5.10.20. Appl., 10.1.19.

AN oxidised resin is saponified with ammonia and the concentration of the solution subsequently reduced with water at ordinary temperature.

—A. de W.

Ink tablets; Production of —. H. Rosenberg. G.P. 323,138, 21.9.17.

INK powders or powdered colours, pigments, or aniline dyes, are mixed with chemical substances which are very readily soluble or which develop gases on the addition of water. Thus mixtures generating carbon dioxide or hydrogen, or alkali percarbonates, may be used; basic colours may be converted into strongly acid salts or acid colours into strongly basic salts, and the products mixed with a salt of opposite reaction.—D. F. T.

Lithopone fast to light; Production of —. A. Eibner. G.P. 324,646, 19.2.18.

Zinc liquors free from ammonium salts are treated with ammonia until the precipitated zinc hydroxide is just dissolved; after boiling for a short time, granulated zinc is added and the solution kept at the ordinary temperature for 12 hrs. By this treatment any metals which could give rise to a coloured sulphide are removed; these metals are the cause of the lack of fastness of colour in lithopone which is accentuated by the presence of chlorine.—D. F. T.

Resin; Process of producing artificial —. H. N. Copthorne and A. L. Tannehill. U.S.P. 1,353,220, 21.9.20. Appl., 5.6.19.

A SOLUTION of artificial resin is obtained by adding to a solution of the lighter and heavier fractions of the light oil distillates of coal tar, in the absence of water, approximately 1½% of sulphuric acid and afterwards neutralising.—D. F. T.

Linseed oil varnish; Substitute for —. R. Koetschan, C. Harries, and E. Albrecht. G.P. 323,155, 25.3.17.

A SUBSTITUTE for linseed oil varnish is supplied by the ozonides of unsaturated hydrocarbons, particularly of petroleum, or of the tar products derived from lignite, peat, or shale. The ozonides can be used in inert solvents such as benzene or carbon tetrachloride, or in admixture with other varnishes or siccatives. The rate of drying can be increased by previous exposure in shallow vessels; by warming it is possible to cause partial decomposition of the ozonides with the formation of peroxides, thereby producing a varnish of different capacity for drying.—D. F. T.

Indurated compounds from lac or its derivatives; Process for producing —. W. W. Simonson and L. Van D. Blair. E.P. 127,825, 2.6.19. Conv., 1.6.18.

SEE U.S.P. 1,309,967 of 1919; J., 1919, 648 A. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 16,800 of 1904.)

Paint or varnish; Manufacture of —. Koppers Co., Assees. of M. Darrin. E.P. 129,264, 12.6.19. Conv., 29.6.18.

SEE U.S.P. 1,296,776 of 1919; J., 1919, 379 A.

Resin; Manufacture of —. Koppers Co., Assees. of M. Darrin. E.P. 132,229, 24.6.19. Conv., 6.9.18. SEE U.S.P. 1,297,328 of 1919; J., 1919, 428 A.

Coating compositions for parts of aircraft. P. R. Bradley. E.P. 151,396, 8.7.19.

SEE U.S.P. 1,321,708 of 1919; J., 1920, 13 A.

Paints; Manufacture of —. J. E. Schneider. E.P. 151,687, 19.6.19.

SEE U.S.P. 1,317,225 of 1919; J., 1919, 916 A. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 1432 of 1883, 15,363 of 1886, 9480 of 1905, and 25,266 of 1909.)

Paint or composition for covering the surface of metals and other materials. C. H. Iverson and G. S. Roberts. E.P. 151,666, 24.9.19.

Resinous product. U.S.P. 1,355,103. See 111.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Vulcanised rubber; Effect of certain accelerators upon the properties of —. G. D. Kratz and A. H. Flower. J. Ind. Eng. Chem., 1920, 12, 971–974.

As a result of vulcanisation tests of extracted and unextracted rubber in presence of light magnesia, it is concluded that the action of small amounts of magnesia in accelerating the vulcanisation of rubber is, to a large extent, of a secondary character, and depends upon its netting in conjunction with certain extraneous substances (probably nitrogenous) present in the rubber. The activity of the magnesia as accelerator depends upon the nature and quantities of these substances. (Cf. J., 1919, 781 A.)—C. A. M.

Rubber; Determination of factice in —. P. Dekker. Gummi-Zeit., 1920, 35, 52–55.

ON account of the resistance of rubber to penetration by alcoholic alkali hydroxide, the customary procedure for the estimation of fatty "rubber substitute" ("factice") tends to give low results. The following method is recommended:—2 g. of the rubber, after extraction with acetone, is steeped overnight in 20 c.c. of benzene; 40 c.c. of N/2 alcoholic potassium hydroxide is added, and the mixture is boiled under a reflux condenser for 4 hrs.; the liquid is filtered, the residue washed successively with boiling alcohol and boiling water, and the combined filtrates evaporated to a bulk of 3–5 c.c.; the fatty acids in this liquid are then estimated in the usual way. The presence of asphaltum in the rubber interferes with the accuracy of the factice determination, the results being too high. Oxidation of the rubber due to prolonged exposure of the sample between the acetone extraction and the subsequent hydrolysis also gives rise to inflated values for the factice content. In an editorial note it is stated that the addition of the alcoholic alkali to the mixture of rubber and benzene should be effected with both liquids at the boiling temperature.—D. F. T.

PATENTS.

Rubber; Treatment of raw — when freshly coagulated from the latex. S. C. Davidson. E.P. 151,344, 20.6.19.

FRESHLY obtained rubber coagulum ordinarily contains a large proportion of water partly enclosed mechanically. The sheets of coagulum are therefore finely perforated by pins mounted in a board; the perforations act as drainage tubes and facilitate the escape of inclusions of gas or water. The rubber is then rolled into thin sheets and finished in any desired way without the necessity of maceration.—D. F. T.

Vulcanised artificial rubbers; Process for increasing the elasticity of —. Farbenfabriken vorm. F. Bayer und Co. G.P. 301,757, 28.12.15.

PRIOR to vulcanisation, 10% or more of an oily substance, such as petroleum, linseed oil, castor oil, paraffin oil, hemp seed oil, safrole, aniline, the alkylanilines, xylydine, and dimethyl-*p*-toluidine, is introduced into the mixture.—D. F. T.

Rubbers, gutta-percha, balala, etc.; Improvement of the plasticity and adhesiveness of difficultly soluble natural —. Ungarische Gummiwaarenfabriks-A.-G. G.P. 323,732, 21.3.18. Conv., 4.5.17.

MATERIALS of the above type are heated with a

rubber solvent such as petroleum naphtha or benzene, phenol, aniline or their homologues, the solvent being subsequently removed. The products are easily soluble, plastic, adhesive, and capable of absorbing the usual compounding ingredients.

—D. F. T.

XV.—LEATHER; BONE; HORN; GLUE.

Chrome [tanning] liquors; Effect of neutral salts on the basicity of —. B. B. Dhavale and S. R. Das. *J. Soc. Leather Trades' Chem.*, 1920, 4, 225—230.

THE end point in the alkalimetric titration of chrome liquors for acidity by the Procter and McCandlish method was found to be influenced by the temperature at which the titration was done and the rate at which the alkali was added, but the dilution of the liquor under titration did not exert any influence. Additions of sodium chloride caused a marked increase in the acidity as determined by this method.—D. W.

Sulphide [in tannery lime liquors]; Estimation of —. Committee of the Soc. of Leather Trades' Chemists on Limeyard Control. III. J. E. Pickles. *J. Soc. Leather Trades' Chem.*, 1920, 4, 231—235.

SOLUTIONS of commercial sodium sulphide are precipitated with cadmium acetate solution; lime liquors containing sulphide are decomposed with acid and the hydrogen sulphide thus liberated distilled into cadmium acetate solution. In either case the cadmium sulphide is filtered off, treated with a known excess of N/20 iodine solution, and the excess titrated with N/20 sodium thiosulphate. 1 c.c. N/20 iodine is equivalent to 0.00085 g. H_2S or 0.00195 g. Na_2S .—D. W.

[Tannery] lime liquors; Caustic alkalinity of —. Committee on Limeyard Control. IV. W. R. and J. Atkin. *J. Soc. Leather Trades' Chem.*, 1920, 4, 236—243.

AT $pH=10.0$ only 24% of the ammonia present in a solution is neutralised. A sample of the lime liquor is titrated with standard acid to the value $pH=10$, using the comparator (J., 1920, 605 A) and thymolphthalein as indicator. This titration is a measure of the caustic alkalinity plus 24% of the ammonia but does not cause any decomposition of the sulphide present. The ammonia is determined by distillation, and the caustic alkalinity then calculated.—D. W.

Gelatins and glues; Jelly strength of —. S. E. Sheppard, S. S. Sweet, and J. W. Scott, jun. *J. Ind. Eng. Chem.*, 1920, 12, 1007—1011.

A TORSION dynamometer is described for determining the jelly-strength of glues and gelatins when moulded into cylindrical test pieces. The product of the breaking load multiplied by the percentage twist at breaking point, divided by the cross section of the test piece is taken as the jelly strength. No simple relationship could be established between the concentration of gelatin and the jelly strength, nor between the jelly strength at a given concentration and the tensile strength of a dry glue joint.

—C. A. M.

Gelatin as emulsifying agent. Holmes and Child. See 11A.

PATENTS.

Glue solutions; Apparatus for the concentration of —. Goslar-Apparetebau Samstag und Co. G.P. 323,845, 28.8.17.

THE glue solution trickles on to a whirling plate, which distributes the liquid in fine drops on to the

corrugated walls of the apparatus down which it flows, meeting an upward current of warm air.

—D. F. T.

Liming of hides [; Means for circulating the liquor in —]. H. Boston and Sons, Ltd., and J. L. Fairrie. E.P. 151,387, 3.7.19.

Adhesives. G.P. 323,665 and 324,928. See XVII.

XVI.—SOILS; FERTILISERS.

Plant analysis and manuring. F. Münter. *J. Landw.*, 1920, 68, 207—224.

EXPERIMENTS were made during five years upon the growth of barley and sugar beet in a loam soil fertilised with stable manure, nitrate, potash salts, and phosphate, separately and in combination. Results showed that the calcium and magnesium content of barley were little affected, but the silica of the straw was decreased by nitrogen and phosphate applications to the soil. The nitrogen content was raised by potash and phosphate. The weather factor had so much effect that no other definite deductions could be drawn. Analysis showed that the roots of sugar beets were little affected by manuring, whereas calcium and magnesium in the tops were increased by potash manuring and decreased by phosphate. In the case of beet also, weather variations influenced growth so strongly that no deductions could be drawn from plant analysis as to the needs of the soil for particular manures. For this last purpose, two differently manured plots must be compared, one fertilised with nitrogen, and the other with potash and phosphate. Under these conditions the following general statements may be made:—If, on the nitrogen plot, the ratio of nitrogen to potash is greater than unity, the soil is deficient in potash; if, on the same plot, the ratio of nitrogen to phosphate is greater than 100:20, the soil is deficient in phosphate; while if, on the phosphate and potash plot, the latter ratio is less than 100:35, the soil is deficient in nitrogen.—J. H. J.

Calcium and iron salts; Antagonistic action of — towards other salts as measured by ammonification and nitrification. J. E. Greaves. *Soil Sci.*, 1920, 10, 77—102.

EXPERIMENTS were made on the effect produced on the bacterial activity of a fertile loam soil by the addition of calcium and iron salts. The ammonifying and nitrifying powers of the soil were first reduced by the addition of various salts, then increasing amounts of the calcium salt or the iron salt were added, and the effect on ammonification and nitrification was observed. Calcium sulphate was found to neutralise the toxic effect on ammonification of sodium carbonate, nitrate, and sulphate, calcium chloride, and magnesium chloride and sulphate. It had no antagonism to sodium chloride. Similar results were obtained in the case of nitrification, except that there was no antagonism towards sodium sulphate and calcium chloride. Thus both the anion and cation take part in the antagonism. Ferrous sulphate was antagonistic to sodium sulphate, chloride, carbonate, and nitrate, and to calcium chloride; ferrous chloride to sodium chloride and nitrate, calcium chloride and magnesium sulphate; ferrous nitrate to magnesium chloride and sulphate, calcium chloride and sodium carbonate, sulphate, chloride, and nitrate; ferrous carbonate to sodium chloride and carbonate, calcium chloride, and magnesium sulphate. The greatest effect was produced by ferrous carbonate on magnesium sulphate. 75% of the toxic effect of the latter being neutralised. In the case of nitrification the action of the iron salts varied somewhat from that on ammonification, the greatest effect

being produced by ferrous nitrate on magnesium chlorido. The amount of iron required to produce the maximum effect varied with the different salts, but in no case exceeded 186 parts per million.

—J. H. J.

PATENTS.

Calcium cyanamide; Process of granulating — for use as a fertiliser. P. M. T. Savès. E.P. 135,847, 25.11.19. Conv., 26.11.18.

To produce calcium cyanamide in a granular condition it is treated with a solution of a silicate, preferably potassium silicate, during its hydration for removal of calcium carbide. The resulting paste is kneaded, dried, and crushed. Phosphorus-containing materials such as bone ash or slag may be incorporated with the product.—W. J. W.

Fertilisers. J. R. Partington and L. H. Parker. E.P. 151,024, 26.6.18.

AMMONIUM nitrate in the form of a dry powder is mixed with bone meal, and the mixture is finely ground. Suitable proportions are ammonium nitrate 1 pt., and bone meal 3 pts. Potassium salts or other fertilising materials may be added. The fertiliser is said to keep dry, and free from caking, on exposure to the atmosphere.—W. J. W.

Plants; [Use of radio-active composition in] growing —. E. Miller. U.S.P. 1,353,931, 28.9.20. Appl., 29.6.18. Renewed 12.8.20.

CARBE carnotite ore tailings is heated, ground, and mixed with inert material to form a radioactive composition for stimulating plant growth.—W. J. W.

Parasites; Process for destroying — and increasing the germinating power of seeds. A. Traube. G.P. 323,925, 3.18.19.

SRLPHUR, dissolved in pyridine or in substances containing it or its homologues, with or without addition of water, is effective for destroying animal or vegetable parasites and for stimulating the germination of seeds.—W. J. W.

Fertilisers; Process for the manufacture of —. A. C. Bohre; K. C. Bohre, extrix. U.S.P. 1,351,719, 5.10.20. Appl., 13.8.19.

SEE E.P. 132,496 of 1919; J., 1920, 311 A.

XVII.—SUGARS; STARCHES; GUMS.

Sucrose; Destruction of — during the boiling of low-grade massecuites. H. L. White. Int. Sugar J., 1920, 22, 588.

On boiling first massecuites at 140°–145° F. (60°–63° C.) figures were obtained leading the author to conclude that under the conditions obtaining generally in Hawaii no appreciable loss of sugar occurs; whereas at the higher temperature of 165°–172° F. (74°–78° C.) there may be a considerable loss.—J. P. O.

Sucrose; Loss of — by inversion during the heating of second massecuites and final molasses. R. C. Pitcairn. Int. Sugar J., 1920, 22, 589.

RESULTS were obtained indicating that there is a very decided danger of inversion while boiling the low-grade products if they are not kept nearly neutral. A massecuite having an acidity corresponding to 0.25% CaO may lose sugar by inversion during boiling, but in the case of molasses, owing to its lower purity, the risk is less. The diminution

of the sucrose content that may occur in this way is capable of reaching an important degree, and probably accounts for a proportion of the unknown losses.—J. P. O.

Decolorising blacks [for sugar refining]. C. F. Bardorf. Canad. Chem. J., 1920, 4, 207–211.

CHARRED sawdust, though destitute of decolorising power, greatly facilitates the filtration and handling of dusty and sooty chars when mixed therewith, and also increases their efficiency by acting as a dispersive medium and preventing agglomeration. Very satisfactory results were obtained by mixing it with prepared carbon from decomposed char dust or even with ground discarded refinery char. On these lines a new product, "Karhos," has been prepared as follows:—1 kg. of 100-mesh char dust is digested with 1250 c.c. of hydrochloric acid of 18° B. (sp. gr. 1.11) for 20 hrs. The pasty mass is mixed with 23 l. of water and 70 g. of charred sawdust, and the mixture is agitated and filtered rapidly; the filtrate, containing phosphates, may be used as a defecating agent or evaporated and sold as superphosphate. The resulting char is washed twice with 23 l. of water, the first washing being afterwards used to dilute a later batch of paste, whilst the second washing is discarded. The char thus obtained filters excellently, and can be partially dried by pumping warm air through the filter press. Addition of 30–50% of charred sawdust to "Eponite" was found to increase the decolorising efficiency of the latter in the mixture by about 25%.—J. H. L.

Sucrose; Determination of — in presence of both invert sugar and raffinose. W. Montgomery. Int. Sugar J., 1920, 22, 580–582.

TABLES are given by aid of which a "sucrose factor" and a "raffinose factor" can be calculated from the amount of reduced copper for use in estimating sucrose, invert sugar, and raffinose in mixtures by a combined method involving polarimetric and cupric-reducing determinations.

Carbohydrate economy of cacti. [Determination of pentose sugars.] H. A. Spoehr. Publ. No. 287, Carnegie Inst. of Washington, 36–37.

IN determining the pentose sugars in cacti extracts, in preference to employing Tollens' furfural method, the accompanying hexoses were eliminated by fermentation, the alcohol removed by distillation, and the cupric-reducing power of the residue determined (using the iodide volumetric method after separating the cuprous oxide by centrifuging), the absence of other non-fermentable sugars having been previously established.—J. P. O.

PATENTS.

Adhesives [gum substitutes]; Production of —. H. Wallasch. G.P. 323,665, 20.4.19.

By the condensation of formaldehyde with dicyanodiamide in the presence of sulphuric acid adhesive products are obtained; with 1 pt. of dicyanodiamide to 2 pts. of 30% formaldehyde solution a fluid product is obtained resembling gum arabic, whilst with a lower proportion of formaldehyde the product is more viscous.—D. F. T.

Adhesives (not sizes for paper); Production of — from lignite. H. S. Gerdes. G.P. 324,928, 27.3.18.

By treating finely divided lignite, which has been saturated with water, with caustic alkali a colloidal suspension and a thick deposit are obtainable. These can be dried separately or together and used for adhesive purposes.—D. F. T.

Nutrient yeast. G. P. 314,601. See XIX A.

XVIII.—FERMENTATION INDUSTRIES.

Diatase enzymes; Influence of neutral alkali salts on —. A. Hahn and K. Harpuder. *Z. Biol.*, 1920, 71, 287.

THE optimum hydrogen ion concentration for the activity of ptyalin is $p_H = 6.4-6.5$, for that of malt diastase $p_H 4.7$. = The enzymic activity is not only influenced by the hydrogen ion concentration of the buffer substances present, but by the concentration of the other ions contained in them. The combination of neutral alkali salts with the buffer substances produces either an accelerating or an inhibiting influence, according to the reaction and the concentration of the salts and buffer substances, on the activity of the enzymes. The optimum hydrogen ion concentration is also changed by the combined influence of the neutral salts and the buffer substances. Neutral alkali salts displace the isoelectric point of ptyalin and malt diastase in the acid direction. The influence of sodium salts is more marked than that of potassium salts. The mode of action of the above enzymes is independent of their electrical nature.—S. S. Z.

Maltase solutions from yeast. R. Willstätter, F. Oppenheimer, and W. Steibelt. *Z. physiol. Chem.*, 1920, 110, 232-241.

MALTASE can be obtained from fresh beer yeast previously killed by an antiseptic, such as chloroform or toluene, by extracting it with water in the presence of ammonia. The authors suggest that, owing to some enzymic process, acid is formed in the yeast cell, which is responsible for the destruction of the maltase when it is extracted from the undried cell, and that the neutralising effect of the ammonia makes it possible to obtain the enzyme also from the fresh yeast cell. The relation between the concentration of the enzyme and the decomposition of maltose, as well as the velocity of the reaction, was studied with maltase preparations from dried and fresh yeasts. The activity of maltase solution is impaired by treatment with kaolin.—S. S. Z.

Acetone and butyl alcohol fermentation of starch; Gas production during —. H. B. Speakman. *J. Biol. Chem.*, 1920, 43, 401-411.

THE following scheme is put forward to represent the course of the fermentation:—

- (1) $(C_6H_{10}O_5)_x + xH_2O = xC_6H_{12}O_6$.
(starch) (dextrose)
- (2) $C_6H_{12}O_6 = C_3H_7CO_2H + CH_3CO_2H + [O_2]$.
- (3) $2C_3H_7CO_2H + O_2 = 2CH_3CO_2CH_2COOH + 2H_2$.
- (4) $CH_3CO_2CH_2COOH = CH_3CO_2CH_3 + CO_2$.
- (5) $C_3H_7CO_2H + 2H_2 = CH_3(CH_2)_2OH + H_2O$.
- (6) $CH_3CO_2H + 2H_2 = CH_3CH_2OH + H_2O$.

This scheme satisfactorily explains the formation of the substances produced, but one or two points still require elucidation before the process is completely understood. (*Cf.* J.C.S., i., 797.)—J. C. D.

Jerusalem artichokes; Working of — in the distillery. K. Windisch. *Z. Spiritusind.*, 1920, 43, 292, 300.

IN laboratory experiments with artichokes gathered in the early part of the year, yields of alcohol ranging from 7 to 10 l. per 100 kg. of tubers were obtained by fermenting a mash of the pulped material with beer yeast (*cf.* Rüdiger, J., 1920, 608A). Preliminary steaming of the pulp at atmospheric or higher pressures reduced the yield of alcohol, and no advantage accrued from the addition of malt to the mash. For small distilleries the following procedure is recommended: The washed tubers are finely pulped, introduced into the fermenting vat, and treated with sufficient water to produce a somewhat fluid mash. Hot water may

be added at first to sterilise the pulp as far as possible, and then cold water. The mash is pitched with 0.5 kg. of pressed yeast per 100 kg. of artichokes, and the fermentation temperature should approach but not exceed 30° C.—J. H. L.

Iodoform reaction. Kunz. *See* XX.

PATENTS.

Malt; Reducing the amount of — used for the saccharification of amylaceous mashes. A. Deutschland. G.P. 323,653, 5.4.19.

THE addition of killed yeast, autolysed yeast, or yeast extract to amylaceous mashes enables saccharification to be effected with less than the usual quantity of diastase or malt. Yeast extract may be produced in the mash itself before the addition of the malt, by heating the mash to 80° C. and adding fresh yeast.—J. H. L.

Mashes and beer worts; Boiling of — under pressure. Nathan-Institut A.-G. G.P. 323,707, 30.3.17.

IN a process of boiling under pressure in which the vapours evolved are conducted away from the liquid, the intensity and duration of boiling are so increased as to remove all the volatile flavouring and aromatic substances and to render permanently soluble the constituents which have become soluble under pressure.—J. H. L.

Spanish moss. U.S.P. 1,327,873. *See* V.

Fat from yeast. G.P. 309,266. *See* XII.

Nutrient yeast. G.P. 314,601. *See* XIXA.

XIXA.—FOODS.

Histidine; Estimation of — in protein and protein-containing matter. M. T. Hanke and K. K. Koessler. *J. Biol. Chem.*, 1920, 43, 527-542.

THE diamino-acids are precipitated from the hydrolysed protein by means of phosphotungstic acid, and the histidine in this precipitate is estimated by means of the colorimetric method previously described (Koessler and Hanke, J., 1919, 962A). (*Cf.* J.C.S., Dec.)—J. C. D.

Histamine; Colorimetric method for estimation of — in protein and protein-containing matter. M. T. Hanke and K. K. Koessler. *J. Biol. Chem.*, 1920, 43, 543-556.

A DESCRIPTION of a long process which aims at isolating histamine in a more or less pure condition before applying the colour test previously described (Koessler and Hanke, J., 1919, 962A). The method is accurate in the estimation of minute quantities of the amine. Histamine was not found present in caseinogen or human blood serum. (*Cf.* J.C.S., Dec.)—J. C. D.

PATENTS.

Drying tea or like vegetable or other substances; Apparatus for —. H. A. Tempelaar. E.P. 151,461, 12.9.19.

IN a chamber through which an upward current of warm air is passed, a number of circular rotary drying surfaces are mounted one above the other on a central shaft and so geared that each rotates in a direction opposite to that of the next one. The drying surfaces consist of circular frames supporting segmental tray sections each of which is tilted about a radial axis, once in every revolution, by engaging a fixed projection from the casing. The material is first distributed over the top drying surface and, by the successive tilting of the sectional trays, discharged on to the surface next below, and so on.—J. H. L.

Drying [fish etc.]; Chambers for —. P. Fleury and D. L. Robertson. E.P. 151,488, 20.10.19.

PERFORATED trays to hold the material to be dried are arranged within a closed chamber through which heated air is drawn by fans carried by a rotating central shaft and arranged above and below the trays. A perforated distributing plate is provided at the bottom of the chamber, and below this is a chamber opening laterally to the atmosphere and containing a steam heating coil. A blower mounted at the upper end of the central shaft controls the passage of the heated air through the chamber.—J. S. G. T.

Food; Manufacture of animal — from kitchen waste and the like. R. L. D. Taylor, Assr. to Predigest Food Co., Ltd. U.S.P. 1,354,068, 28.9.20. Appl., 25.2.19.

GARBAGE is pulped, dried in motion so as to form nodules, cooked without breaking up the latter, and finally screened to separate the nodules from the coarser and finer fractions; or the garbage is mixed with a pulverulent absorbent vegetable filler, and the partly dried mixture is pulped and then dried in motion at a temperature above 300° F. (150° C.) so as to form nodules.—J. H. L.

Fire-gases from a coke or coal fire; Apparatus for obtaining — free from smell, soot, and dust, for drying vegetable and animal foodstuffs. O. Breidenbach. G.P. 319,777, 23.9.17.

The gas stream is divided into a number of horizontal streams to which air is admitted to ensure complete combustion of the gas. The velocity of the gas is increased towards the end of the horizontal course, the separate streams then unite, and the combined stream passes sharply downwards, whereby a portion of the fine dust settles out. The gas stream flows again in a horizontal course, and passes over means for precipitating the ash, turns sharply upwards, and the last traces of dust and soot are removed in a double filter-sieve against a counter-current of fresh air.—L. A. C.

Yeast; Production of nutrient — especially for use as nitrogenous fodder, from waste waters of sugar factories. A. Riedel. G.P. 314,601, 16.2.18.

TUZ diffusion process is carried out with ammoniacal "fall water" and condensed water from the evaporating plant, and the waste diffusion and press waters are mixed with yeast, without aeration, the solids being subsequently separated and dried.—J. H. L.

Potato products; Preparation of dried —. Chem. Ges. Rhenania m.b.H. G.P. 322,821, 17.9.18. Addn. to 312,614 (J., 1919, 842 A).

BEFORE being dried, the sliced potatoes are treated with an electrolysed sodium chloride solution containing 120 g. of active chlorine per 100 kg. of potatoes. No free chlorine remains in the potatoes after drying, and the latter retain their original colour.—J. H. L.

Bones; Utilisation of — with production of an edible fat and a valuable fodder. J. Brix. G.P. 323,650, 23.6.16.

FRESH bones are boiled, first under a low pressure, up to 2 atm., and then under a higher pressure. The liquors obtained from the two operations are separately freed from fat, and the liquor from the first boiling is then concentrated and mixed with the bone residues after the latter have been comminuted.—J. H. L.

Fodder from straw; Manufacture of —. E. Beckmann, and Veredelungsges. für Nahrungs- u. Futter-Mittel. E.P. 151,229, 15.8.19. Conv., 25.4.17.

SEE G.P. 305,641 of 1917; J., 1919, 789 A.

XIXB.—WATER PURIFICATION; SANITATION.

Ammonia nitrites, and nitrates [in water]; Colorimetric estimation of —. I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 1253—1264.

THE effects of time, temperature, quantity of reagent, presence of retarding substances, etc., on the colorimetric determinations in water analysis are examined, and several modifications suggested. In the ammonia determination, Winkler's reagent is found unsatisfactory, and nephelometric estimation by means of alkaline mercury-sodium chloride, which gives a white opalescence with 0.1 mg. NH_4 per l., is proposed instead. Modifications of the phenol-sulphuric acid method for determination of nitrate and of the Griess-Romijn method for nitrites are also put forward. (Cf. J.C.S., ii., 703.) —S. I. L.

X-ray fluorescence of certain organic [and inorganic] compounds. H. S. Newcomer. J. Amer. Chem. Soc., 1920, 42, 1997—2007.

SEVERAL inorganic salts, 308 organic compounds apart from dyes, and 157 dyes have been treated with powerful X-rays with the object of finding a substance, soluble in water, which will transform these rays into bactericidally active ultra-violet rays. A large number of the substances become fluorescent under the treatment, but only sodium bromide gives rays of the desired nature. (Cf. J.C.S., Dec.)—J. F. S.

PATENTS.

Purification of expired air; Process for —. M. Speter. G.P. 323,210, 1.7.19.

FOR the purification of vitiated air, use is made of alkali peroxides which are first fused, and then incorporated after solidification with inorganic compounds of high melting point which do not interact with them, such as chlorides, sulphates, carbonates, and phosphates. The efficiency of the product is increased by addition of catalysts such as manganese dioxide.—W. J. W.

Sulphite - waste - liquor preparation. U.S.P. 1,327,862. See V.

Destroying parasites. G.P. 323,925. See XVI.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Morphine in opium; Determination of —. A. Jernstad. Inaug. Dissert., Basle, 1920, 1—151. Chem.-Zeit., 1920, 44, Rep., 235.

THE only methods of practical importance for the estimation of morphine in opium are the water extraction and the lime extraction methods. The lime extraction methods of the American, English, French, Japanese, Dutch, and Spanish Pharmacopœias yield impure morphine and generally too high results. If, however, the morphine is freed from narcotine and calcium meconate by means of benzene and alcohol, the results coincide with those obtained by the Helfenberger water extraction method. Of the unofficial water methods only those of Dieterich (simplified Helfenberger method), Frey and Thoms are of value. The methods of Hauke and of Léger and Picard give too low, and that of Merck too high results. Of the unofficial lime extraction methods examined, only Beckurt's is usable, those of Asher, Carles, Debouredaux, Guérin, Hager, Maske, and Petit and Picard give impure morphine, some results being too high and others too low. In the Helfenberger method the use of ethyl acetate has no advantage over ether, but methyl red is preferable to iodoquin

indicator. For pharmacopœial use the following modification of the Helffenberger method is recommended:—6 g. of opium is rubbed to a smooth paste with 6 g. of water, and transferred to a weighed flask with water to a total weight of 54 g. After keeping with continual shaking for half an hour, the extract is filtered through a dry 10 cm. filter paper, and to 40 g. of the filtrate is added 2 g. of *N*/1 ammonia, and immediately after mixing the liquor is once more filtered. To 36 g. of the filtrate are added 7 g. of ether and 4 g. of *N*/1 ammonia and the mixture is vigorously shaken in a stoppered flask for 10 mins. A further 10 c.c. of ether is then added and after standing half an hour the ether layer is poured off as completely as possible through a small filter, the operation being repeated with another 10 c.c. of ether. The whole contents of the flask are then poured on to the same filter and the flask with the crystals remaining in it is rinsed out three times with 5 c.c. of water saturated with ether which is finally passed through the filter. The flask and filter paper are then dried at 100° C., and the contents of the latter transferred as completely as possible to the flask and dissolved in 25 c.c. of *N*/10 hydrochloric acid. The solution is then poured through the filter paper to dissolve any crystals remaining on it, and flask and filter are finally washed with 75 c.c. of water. The combined solution and washings are then titrated back with *N*/10 sodium hydroxide with methyl red as indicator. Between 8 and 11 c.c. should be required, corresponding to 10–12% of anhydrous morphine in the opium. (1 c.c. *N*/10 HCl=28.5 mg. of morphine.) For the preparation of *Extractum opii* the following shortened process may be employed:—10 parts of powdered opium is rubbed to a smooth paste with 10 parts of water, 40 parts of water added and after keeping an hour with frequent shaking, the extract is separated and the residue once more treated as above with 25 parts of water. The united extracts are filtered and evaporated to a dry extract.—G. F. M.

Cinchona preparations; Assay of — F. Lehmann. Arch. Pharm., 1920, 258, 85–90.

THE acid extraction of cinchona bark and its preparations indicates a lower alkaloidal content than the extraction from alkaline mixtures. When the alkaloidal extracts from the latter mixtures are subjected to a subsequent purification by being agitated with acid, a portion of the alkaloids is lost in addition to the neutral substances; the alkaloidal content as found by the two processes, however, becomes more nearly identical.—H. W.

Digitalis leaves; Activity of — from one and two year old plants. G. Joachimoglu. Arch. Pharm., 1920, 258, 33–55.

THE activity of the leaves of digitalis plants grown from seeds originating from various districts of Germany decreased rather than increased with the age of the plants, and the pharmacopœial requirement that the leaves should be gathered from two-year old plants appears to require revision. Leaves kept for a year after quickly drying at 60° C. showed no decrease in activity; when dried by the ordinary method a slight decrease was perceptible.—G. F. M.

Chelidonium alkaloids. II. Quantitative determination of dioxymethylene groups in alkaloids. J. Gadamer. Arch. Pharm., 1920, 258, 148–167.

A FULL description of the experimental evidence on which the formula of allocryptopine (Arch. Pharm., 1919, 257, 298) rests is given. Chelerythrine is closely related to homochelidonine. It contains two methoxyl and one dioxymethylene group. The dioxymethylene groups in alkaloids may be approximately determined in the following manner. Pure

phloroglucinol (0.3 g.) is dissolved in a warm mixture of water (15 c.c.) and concentrated sulphuric acid (15 c.c.) and a weighed amount of the alkaloid dissolved in water (5 c.c.) is added. The mixture is shaken until a clear solution results and concentrated sulphuric acid (10 c.c.) is added. The mixture is boiled gently until a precipitate begins to form, after which it is kept at 70°–80° C. for 3 hrs. After 24 hrs. the precipitate is collected in a Gooch crucible, washed with water (60 c.c.), dried at 100° C. for 4 hrs., and weighed. The weight of phloroglucide divided by 9.857 gives the amount of methylene, CH₂. The process should be repeated with varying amounts of alkaloids and occasional blank experiments must be performed. The results are to be regarded as minimal values but are, in general, sufficiently accurate to indicate the number of dioxymethylene groups present. (Cf. J.C.S., Dec.)—H. W.

Chelidonic acid; Occurrence of — E. Stransky. Arch. Pharm., 1920, 258, 56–69.

THE crystalline precipitate obtained by the fractional precipitation of the cold water extract of *Convallaria majalis* by means of lead acetate solution was identified as the lead salt of chelidonic acid, the occurrence of which has previously only been noticed in *Chelidonium majus*, *Veratrum album*, and *Buphone disticha*. As chelidonic acid is quantitatively converted by boiling milk of lime into 1 mol. of acetone and 2 mols. of oxalic acid, this reaction was utilised for the detection and quantitative estimation of the acid in *Convallaria* and numerous other plants. The largest amount found was 2.09–2.18% in the leaves of *Convallaria majalis*. A high percentage, 1.42–1.53, was found in an entirely new source, namely, *Sabadilla* seeds. *Chelidonium majus* contained from 0.14 to 0.74% according to the season and the particular part of the plant. In certain *Veratrum* sp. 0.04–0.39% was found, and traces were indicated in various other Liliaceous and Amaryllidaceous plants.—G. F. M.

Ephedrine and pseudoephedrine; Synthesis of inactive — A. Eberhard. Arch. Pharm., 1920, 258, 97–129.

IMPROVEMENTS in the preparation of α -methylamino-propiophenone from methylamine and α -bromopropiophenone (Eberhard, Arch. Pharm., 1915, 253, 62) and the substitution of hydrogen under pressure in the presence of palladinised charcoal for sodium amalgam in the reduction of the ketone have rendered possible the production of phenyl- α -methylaminoethyl carbinol, C₆H₅CH(OH)CH(NCH₃)₂CH₃, in considerable quantity. A fuller study of this base [B], m.p. 114° C., has revealed the existence of an isomeric base [A], m.p. 76° C., and a number of salts and derivatives of each are described. The inactive synthetic compounds, their salts and derivatives, show great similarity to the natural, optically active bases both in appearance and melting point; this is particularly noticeable in the case of the α -base and pseudoephedrine. The synthetic bases can be converted one into the other in a similar manner to ephedrine and pseudoephedrine; this can be partly effected by hydrochloric acid at the ordinary pressure, but more readily at higher temperature and under increased pressure, whilst the base A is completely converted into base B by acetylation but not by benzoylation. (Cf. J.C.S., Dec.)—H. W.

Sulphonal and trional; Identification of — W. Zimmermann. Apoth.-Zeit., 1920, 35, 27.

THE m. pt. of sulphonal is 125°–126° C., that of trional 76° C. Both substances yield an odour of mercaptan when fused with an equal weight of sodium salicylate; if the mixture is then heated

with alcohol and sulphuric acid an odour of methyl salicylate is observed. When 0.2 g. of sulphonal or trional is mixed with 0.04 g. of sodium salicylate and ignited in a porcelain basin, a violet coloured residue is obtained which yields a violet solution when treated with a drop of water; this colour changes rapidly to brown-red. If the residue is treated with a drop of dilute hydrochloric acid, a yellow solution containing a brown flocculent precipitate is obtained and sulphur dioxide is evolved. When treated similarly, acetanilide, antipyrine, and phenacetin give a black residue, veronal yields a bright yellow residue, and santonin a red residue.

—W. P. S.

Hypnotics; New class of —. *Dialkylhomophthalimides*. A. Lumière and F. Perrin. *Comptes rend.*, 1920, 171, 637–639.

DIETHYL-, ethylpropyl-, dipropyl-, and diallylhomophthalimides may readily be prepared by the action of the corresponding alkyl iodides on homophthalimide in the presence of sodium ethoxide. They are all hypnotics, and at the same time only very slightly toxic and free from any unfavourable secondary action. (*Cf.* J.C.S., i., 780.)—W. G.

Quinoline synthesis; Modification of Skraup's —. E. de B. Barnett. *Chem. News*, 1920, 121, 205–206.

By a modification of Skraup's quinoline synthesis, using the aniline and glycerol in approximately equimolecular proportions, and calcined ferric oxide instead of nitrobenzene or arsenic acid as oxidising agent, yields of 40–50% of the theoretical, calculated on both aniline and glycerol, were obtained. Increasing the proportion of glycerol did not materially improve the yield of quinoline, and it seems probable that the comparatively low yield is due more to the destruction of the aniline by the oxidising agent than to the decomposition of the glycerol. A 60% yield of quinoline was obtained by mixing 50 g. of aniline, 65 g. of glycerol, and 100 g. of ferric oxide, and adding the mixture rapidly to 150 c.c. of concentrated sulphuric acid in an open dish. After standing for half an hour the product is worked up in the usual way.—G. F. M.

Arsenic acids; Diazo synthesis of aromatic — and its theoretical significance in connexion with similar actions. A by-product containing arsenic. H. Schmidt. *Annalen*, 1920, 421, 159–174.

AROMATIC arsenic acids are prepared by the action of diazonium solutions on sodium arsenite (G.P. 250,261, 261,924; J., 1913, 1030). The most favourable conditions for the production of phenylarsinic acid and many of its nucleus substituted derivatives are secured when the alkalinity of the solution is so regulated that action can occur in accordance with the scheme: $\text{ArN}_2\text{Cl} + \text{AsO}_2\text{HK}_2 = \text{KCl} + \text{Ar.As}(\text{O},\text{HK}) + \text{N}_2$. In this manner, *o*-nitrodiazobenzene gives about 90% of the theoretical quantity of *o*-nitrophenylarsinic acid, but the amounts of arsenic-free by-products (azo-compounds etc.) are more considerable in the cases of many other arsenic acids. In more strongly alkaline solution the reducing action is more generally noticeable and the yields of arsenic acids are lower. Poorer yields are also observed with diazobenzene in acid solution, but an acid medium is favourable when strongly acidic substituents are present in the benzene nucleus. In addition to the well-known arsenic-free by-products, others containing arsenic can be isolated, the chief of which is phenylphenylenearsinic acid, $\text{C}_6\text{H}_5\cdot\text{C}_6\text{H}_4\cdot\text{AsO}_2\text{H}_2$. (*Cf.* J.C.S., Dec.)—H. W.

Civet. J. Nivière. *Bull. Soc. Chim.*, 1920, 27, 791–797.

A SAMPLE of civet was found to contain cholesterol and dextrose, and is thought to have been adul-

terated with wool fat and glucose syrup. Another sample was found to contain over 18% of talc powder.—W. G.

Antimony compounds; Aromatic —. I. Diazo-synthesis of aromatic stibinic acids and their polymeric constitution. *Aromatic derivatives of antimony pentachloride and its additive compounds*. H. Schmidt. *Annalen*, 1920, 421, 174–246.

THE action of diazo-compounds on antimony oxide is similar to that with arsenious acid (preceding abstract), nitrogen being copiously and spontaneously evolved and arylstibinic acids formed. The difficulty of retaining antimony oxide in solution with a little alkali can be surmounted by the use of potassium antimonyl tartrate and other soluble antimony salts or by the use of freshly precipitated hydrated antimony oxide which need not be completely dissolved. Reaction can frequently be effected in strongly alkaline solution, but when markedly acidic substituents are present in the benzene nucleus it is frequently preferable to operate in a neutral or acid medium. The arylstibinic acids are derivatives of polymeric antimonous acids (e.g., phenylstibinic acid $\text{O}(\text{H}.\text{SbC}_6\text{H}_5)_3(\text{O}.\text{O}.\text{SbC}_6\text{H}_5)_2(\text{O}.\text{O}.\text{H})$), which under the action of alkali and water yield the alkali salts of monomolecular arylstibinic acids, the latter, however, readily re-polymerising when liberated. They have pronouncedly colloidal properties. Antimony can frequently be estimated in arylstibinic oxides and triarylstibines which contain an amino group by direct titration with iodine in very dilute faintly acidic solution. If a group is not present which confers solubility in water, a suitable bicarbonate-alkaline solution can generally be obtained with the aid of tartaric acid and organic solvents. Alternatively about 1/1000 mol. of the substance is mixed with sodium chloride (0.2 g.) and sodium bisulphate (3 g.) in a Kjeldahl flask and boiled for 1 hr. with a mixture of nitric acid (sp. gr. 1.491, 1.5 c.c.) and concentrated sulphuric acid (10 c.c.). After cooling, ammonium sulphate (1 g.) is added and the solution is boiled for 30 mins. to expel nitric acid; it is subsequently diluted to about 300 c.c. and after addition of 5*N*-hydrochloric acid (20 c.c.) is reduced with sulphur dioxide and potassium bromide. The antimony is finally titrated with *N*/10 iodine in bicarbonate-alkaline solution (*Cf.* J.C.S., Dec.)—H. W.

Ichthyol oil and allied bituminous tar oils; Isolation of the active sulphur compounds of —. H. Scheibler. *Arch. Pharm.*, 1920, 258, 70–84. (*Cf.* J., 1920, 55a.)

THE typical sulphur compounds of ichthyol and other bituminous tar oils are derivatives of thiophene, which may be present in varying proportions up to as much as 50% in the case of ichthyol oil. From the purified oil (*loc. cit.*) by various reactions, particularly by treatment with less than the theoretical quantity of acetyl chloride and aluminium chloride, thiophene derivatives were isolated in a pure state.—G. F. M.

Cineol [eucalyptol]; Determination of — in essential oils. C. Kleber and W. von Rechenberg. *J. prakt. Chem.*, 1920, 101, 171–176.

THE solidifying point of the oil is determined in a jacketed vessel immersed in a mixture of ice and salt. Since the other constituents of eucalyptus oil are mainly hydrocarbons, the reference curves are obtained by observation of the solidifying temperatures of mixtures of cineol and rectified oil of turpentine; data are given over a range of 100% to 61% of cineol. In addition to hydrocarbons, the oil contains minor amounts of alcohols and aldehydes; the former, as exemplified by terpineol appears to raise the solidifying point, which is depressed by aldehydes as instanced by valeralde-

hyde. The results are accurate to within 1%. The process is not directly applicable to substances, such as cajuput oil, which contain less than 65% of cineol but may be utilised if a quantity of cineol equal to the weight of the oil is added.—H. W.

Acetone; Process for purifying —. J. Duclaux and A. Lanzenberg. *Bull. Soc. Chim.*, 1920, 27, 779–782.

ACETONE forms with carbon bisulphide a minimum boiling-point mixture. The acetone is mixed with 1.7 times its volume of carbon bisulphide and the mixture distilled, the product passing over between 35° and 40° C. being collected in three fractions. The acetone is extracted from these fractions by water and the aqueous extracts are distilled, the fraction boiling at 56.1°–56.3° C. being collected. If methyl alcohol is present in the acetone the minimum b.p. distillate of carbon bisulphide and acetone is left to stand over potassium carbonate before extraction with water, and copper sulphate is added to the subsequent aqueous extract before distillation. In this way most of the methyl alcohol is removed.—W. G.

Resin; Aldol condensation and formation of — by the action of dilute alkalis on acetaldehyde. H. Hammarsten. *Annalen*, 1920, 421, 293–315.

The action of the hydroxides of potassium, barium, and lead on dilute solutions of acetaldehyde at 0° C., 18° C., and 50° C. has been examined. The main reaction consists in the formation of aldols (the minimum quantity obtained being 40% of that theoretically possible), and this occurs even with very low hydroxyl-ion concentrations. Acetaldehyde, formed primarily, speedily passes into higher aldols which do not become converted into aldehyde resins as does the simpler substance. Aldehyde-resin consists of at least two substances, the red α - and β -acetaldehyde-resin described by Ekecrantz and a very pale yellow resin, m.p. 120°–130° C. after softening at 105° C. Acetaldehyde can be estimated in the presence of aldols by drawing purified air through the solution at 25°–30° C. and 25–30 mm. and then through a measured volume of N/5 iodine solution in the presence of N/1 potassium hydroxide; unused iodine is titrated with N/10 thiosulphate solution. Acetaldehyde (1 mol.) reacts with iodine (4 atoms) apparently in accordance with the scheme: $2\text{CH}_3\text{CHO} + 8\text{I} + 3\text{KOH} = 2\text{CHI}_3 + \text{H.CO.K} + \text{CH}_3\text{OH} + 2\text{KI} + 2\text{H}_2\text{O}$. Aldols are estimated, after removal of resin by filtration, by repeated extraction with ether, desiccation of the extracts with calcium chloride, removal of the solvent under diminished pressure, and weighing of the residue after exposure to phosphoric oxide at the ordinary pressure for 24 hrs. The aldols are subsequently volatilised at 120° C. and 10 mm., and any residual resin is weighed. (*Cf.* J.C.S., Dec.) —H. W.

Acetaldehyde; Determination of — in paraldehyde. W. Stüwe. *Apoth.-Zeit.*, 1920, 35, 153–154.

FIVE c.c. of the paraldehyde is added to a mixture of N/10 mercuric chloride solution 2 c.c., potassium iodide 2, 15% sodium hydroxide solution 20, and water 20 g., and, after 15 mins., the mixture is diluted to 100 c.c., and filtered; 50 c.c. of the filtrate is treated with gum arabic 0.5 g., sodium hydroxide solution 5, and formaldehyde solution 3 c.c., acidified, after 2 mins., with 15 c.c. of dilute acetic acid, cooled, and the separated mercury dissolved by the addition of 10 c.c. of N/10 iodine solution. The excess of iodine is then titrated with N/10 thiosulphate solution. Each c.c. of N/10 iodine solution = 0.0022 g. of acetaldehyde.—W. P. S.

Alcohols; Dehydrogenation of — by catalytic oxidation under reduced pressure. C. Moureu and G. Mignonac. *Comptes rend.*, 1920, 171, 652.

WHILST the method previously described (J., 1920, 247A) gives good results with alcohols of low mol. wt., it is found that with the higher alcohols it is preferable to pass the alcohol vapour and oxygen over the catalyst under a reduced pressure of 20–40 mm. and at 230°–300° C. Yields of 80–90% of the respective aldehydes may be obtained by this means from benzyl and cinnamic alcohols.—W. G.

Iodoform reaction [for detecting alcohol]; Sensitive modification of Lieben's —. R. Kunz. *Z. anal. Chem.*, 1920, 59, 302–303.

TEN c.c. of the dilute alcohol solution (e.g., 1 drop of alcohol in 100 c.c. of water) is treated with 2 c.c. of 10% sodium hydroxide solution, 0.15 g. of potassium iodide, and 0.2 g. of potassium persulphate, and heated at 50°–60° C. A turbidity, due to the formation of iodoform, develops in about 10 mins.—W. P. S.

PATENTS.

Chlorhydrins and bromhydrins of olefinic hydrocarbons; Manufacture of —. The Commercial Research Co., Assees. of B. T. Brooks. E.P. 128,578, 19.6.19. Conv., 20.6.18.

OLEFINIC hydrocarbons are converted into chlorhydrins or bromhydrins by subjecting them in the gaseous state, under a pressure of 5–6 atm., to the action of a weak aqueous solution of hypochlorous or hypobromous acid, respectively, of concentration preferably just under 0.15%. The aqueous medium is kept in continual circulation by means of a pump in a circuit comprising a pressure chamber in which the dilute acid solution sprayed in at the top meets and absorbs the olefinic gases forced in under pressure at the base, a storage tank into which the dilute chlorhydrin solution is discharged from the pressure chamber, and which communicates with a mixing vessel charged from time to time with sodium bicarbonate or other alkali salt of a weak acid, and finally a chlorinating or brominating chamber into which the alkaline solution from the mixing vessel overflows and reacts with halogen with the formation of sodium halide, carbon dioxide, and hypochlorous or hypobromous acid. Circulation is continued until a concentration of preferably 4.5%, but not exceeding 7%, of halogenhydrin is attained. The optimum temperature for an olefinic gas made by cracking petroleum products, and containing some 30–50% of ethylene and propylene, is about 15° C., and by regulating the pressure under which absorption takes place it is possible first almost quantitatively to convert the propylene from the gas into halogenhydrin without acting on the ethylene.—G. F. M.

Vanillylamine, vanillylacylamide, and production thereof. E. K. Nelson. U.S.P. 1,329,272, 27.1.20. Appl., 9.10.19.

VANILLYLAMINE, m.p. 131°–133° C., is prepared from vanillin by converting it into its oxime and reducing this with sodium amalgam and acetic acid. The corresponding acylamides, prepared from vanillylamine by the usual methods, are pungent substances which can be used in medicine in place of capsicum.

Di-(normal)-butylbarbituric acid. O. Kamm and E. H. Vollweiler, Assrs. to Abbott Laboratories. U.S.P. 1,331,712, 24.2.20. Appl., 23.1.19.

DI-N-BUTYLMALONIC ester, prepared by interaction of *n*-butyl bromide and malonic ester in presence of sodium ethoxide, is heated with sodium and alcohol and urea in an autoclave at 100°–110° C. After distilling off the alcohol from the reaction product, water is added, and di-*n*-butylbarbituric

acid precipitated by acidifying with hydrochloric acid. It forms white needles, m.p. 158° C., and has pronounced sedative properties together with low toxicity.

Ipecac alkaloids; Enteric preparation of —. J. K. Lilly, Assr. to The Eli Lilly and Co. U.S.P. 1,334,463, 23.3.20. Appl., 14.8.15.

CLAIM is made to a preparation containing the alkaloids of ipecacuanha combined with an adsorbent agent (Lloyd's reagent, hydrated aluminium and magnesium silicates; J., 1916, 651) capable of preventing the solution and absorption of alkaloids in the acid medium of the stomach, but releasing the alkaloids in the alkaline medium of the intestines.

Oils from the peel of citrus fruits; Process of extracting —. F. A. McDermott. U.S.P. 1,353,169, 21.9.20. Appl., 13.1.16.

THE peel is dried at a temperature below that at which the oil evaporates, and the oil is extracted from the residue, after grinding, by distillation under reduced pressure with a current of steam.
—L. A. C.

Acetone; Manufacture of — from acetic acid. Farbenfabriken vorm. F. Bayer und Co. G.P. 298,851, 8.3.16.

ACETONE may be produced by passing acetic acid vapour over a heated acetate or a base. If barium acetate is used, the reaction takes place at 320° C., or at a lower temperature under reduced pressure, and a quantitative yield results. Strontium carbonate, oxides of calcium and magnesium, or the corresponding acetates, may be substituted for barium acetate.—W. J. W.

Vapours [alcohol, ether] absorbed in sulphuric acid; Recovery of —. F. Herrmann. G.P. 300,733, 6.3.17.

THE solution in sulphuric acid, after dilution with water, is made to pass downwards through a series of chambers where it is submitted to direct or indirect heating by steam.—D. F. T.

Toxins; Production of Röntgen-, radium-, or thorium- —. E. Merck. G.P. 321,956, 30.9.17. Addn. to 257,473 (J., 1913, 507).

INTERNAL organs aseptically removed from animals are cooled by means of ice and subjected to Röntgen, radium, or thorium rays, and the decomposition products formed are dissolved in serum which has been similarly exposed to radiation. The serum is injected intravenously into living animals in increasing doses, and the animals are then treated according to the main patent. The tissues which have been exposed to radiation may alternatively be extracted with physiological salt solution containing radium emanation.—J. H. L.

Silver-protein preparations readily soluble in water; Production of stable —. Farbenfabr. vorm. F. Bayer und Co. G.P. 322,756, 12.5.16.

SILVER-protein compounds identical with or similar to those produced in accordance with G.P. 105,866, are mixed with urea or its derivatives (e.g., mono- or dimethylurea), with or without other suitable ingredients.—J. H. L.

Dihydroxyacetoxime; Preparation of —. C. H. Boehninger Sohn. G.P. 322,845, 1.12.17.

β -NITROPROPYLENEGLYCOL, or a salt thereof, is treated with reducing agents in presence of basic substances, such as hydroxides of alkalis or alkaline earths. Dihydroxyacetoxime may be almost quantitatively converted into glycerol.—W. J. W.

Yohimbine; Production of readily and perfectly soluble nucleic acid compounds of —. E. Weinert. G.P. 322,996, 26.7.17.

AMMONIA is added to a mixture of nucleic acid with yohimbé bases; or ammonium nucleate is allowed to interact with salts of yohimbine or of the total bases of yohimbé bark. Yohimbine nucleate, $C_{22}H_{33}O_5N_3 \cdot C_{10}H_{15}O_4N_3 \cdot P_1$, is a yellowish or greenish white amorphous powder with a slightly acid flavour and an almost neutral reaction. It is readily soluble in water, less soluble in alcohol, and insoluble in acetone or ether, and possesses therapeutic value.—J. H. L.

Arylurea; Preparation of an —. J. D. Riedel A.-G. G.P. 323,298, 20.12.17.

p-NITROPHENOL is treated with ethylenedichlorhydrin, and the amine formed by subsequent reduction is converted into the urea. *p*-Nitrophenol hydroxyethyl ether crystallises from aqueous solution as yellow needles, m.p. 94° – 95° C. The hydrochloride of *p*-aminophenol hydroxyethyl ether forms needles, m.p. 204° – 205° C., and its aqueous solution reacts with potassium cyanate to form the corresponding urea, m.p. 159° – 160° C.—W. J. W.

Glyoxal; Preparation of —. A. Wohl and K. Bräunig. G.P. 324,202, 30.6.16.

AIR or oxygen containing approximately 1% by vol. of ozone is passed through water and then mixed with $1\frac{1}{2}\%$ of acetylene in a large reaction space; a solution of glyoxal separates on the walls and bottom of the vessel.—D. F. T.

Hexamethylenetetramine; Preparation of derivatives of —. J. D. Riedel A.-G. G.P. 324,203, 11.6.14.

THE quaternary bases derived from hexamethylenetetramine, e.g., hexamethylenetetraminemethyl hydroxide and hexamethylenetetramine-ethyl hydroxide are combined with cholic acid; the resulting cholates are crystalline solids, soluble in water, and suited to the treatment of gall stones.—D. F. T.

Fatty acids and aldehydes; Method for the production of —. C. Harries, R. Koetschau, and E. Albrecht. G.P. 324,663, 29.2.16.

THE ozonides of unsaturated aliphatic oils derived from petroleum, or from the tar products of lignites, shales, peats, and asphalts, are decomposed, in solution if necessary, with formation of aldehydes or acids. A sample of gas oil from lignite tar yielded liquid acids, b.p. 80° – 220° C. under 10–12 mm. pressure, the fraction boiling at 180° – 200° C. at the same pressure containing solid fatty acids. The acids contained from seven to twelve atoms of carbon, and ranged from heptioic acid to lauric acid. The residual oil which resisted the action of ozone and of caustic alkali boiled at 260° – 330° C. and was of value for lubricating and for transformers.—D. F. T.

Glycerin substitute. G.P. 303,991. See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENT.

Colour photography and cinematography. W. V. D. Kelley. E.P. 129,638, 7.7.19. Conv., 8.7.18. See U.S.P. 1,337,775 of 1920; J., 1920, 468 A.

XXII.—EXPLOSIVES; MATCHES.

Detonation of explosives; Study of the causes governing the —. H. Kast. Z. ges. Schiess-u. Sprengstoffw., 1920, 15, 195–197.

THE phenomenon of detonation, induced by an initial impulse or shock, cannot be attributed to

the influence of air compressed in the interstices of the explosive. Experiments with pressed T.N.T. charges showed a diminution of the air content with rising density, and the velocity of detonation of T.N.T. filled into a tube was not appreciably different whether the explosion took place under normal atmospheric pressure or in an evacuated tube. If explosion were due to the effect of pressure in promoting the aggregation of the molecules, an increase of the explosion temperature with highly-compressed charges would be anticipated, but this is not the case. A probable interpretation of the problem is the disturbance of molecular stability, and this theory would account for the difficulty in effecting the detonation of dense charges having a rigid physical structure. Confirmatory evidence is found in the different initial impulses required for explosives of varying physical condition, though of similar chemical composition, and in the divergent velocities of detonation attained by them. The results obtained with explosives of various densities are tabulated. Nitrocellulose powders and gunpowder which, normally, achieve a velocity of ignition rather than of detonation, may nevertheless exert a disruptive effect if large quantities are involved.—W. J. W.

PATENTS.

Detonators; Blasting —. W. Eschbach. E.P. 151,572, 28.5.20.

ALUMINIUM is employed as the material for detonator shells, in which the detonating charge consists of lead azide, or lead azide mixtures, or tetryl with a top charge of lead azide, or other initiating composition which exerts no action on the aluminium. The advantages claimed for this metal, as compared with copper or brass, are the obviation of splitting when the shells are crimped on to the fuse, the freedom from oxidation, and the degree of hardness attainable which prevents distortion during pressing. The detonators are stated to have a greater coefficient of initiation, and to give a finer radiation, than those made with copper shells.—W. J. W.

Detonating agent; Initial —. W. O. Snelling. U.S.P. 1,353,805, 21.9.20. Appl., 6.5.20.

THE composition consists of lead azide prepared by the interaction of an alkali azide and a soluble lead salt in a solution of glue.—W. J. W.

Nitrating apparatus. Wülfling, Dahl und Co., A.-G. G.P. 300,079, 3.11.15.

THE interior cooler of a nitrating apparatus is provided with a flanged collar in which the pipe connexions are arranged, and which fits snugly between the flanges of the vessel and its lid when the apparatus is closed.—W. J. W.

Explosive; Gelatinised propellant — and process of preparing same. F. L. Nathan, W. Rintoul, and F. Baker (H. Baker, administrator), Assrs. to Nobel's Explosives Co. U.S.P. 1,338,691, 4.5.20. Appl., 26.2.14. Renewed 21.10.18.

SEE E.P. 4940—1 of 1913; J., 1914, 712.

Treating waste acids. G.P. 323,416. See VII.

XXIII.—ANALYSIS.

Thermocouples; Construction of — by electro-deposition. W. H. Wilson and T. D. Epps. Proc. Phys. Soc., 1920, 32, 326—340.

SMALL thermocouples suitable for the construction of batteries containing a large number of junctions may be constructed from a continuous coil of one of the metals which is coated, electrolytically, in suitable places with the other metal. If the elec-

trical conductivity of the latter element is considerably greater than that of the former, and a fairly thick sheath is deposited, a thermocouple is produced which is not appreciably impaired in efficiency by the short-circuiting effect of the core. Constantan wires coated with either copper or silver sheaths are suitable for most purposes.—J. F. S.

Gases; Apparatus for measuring the rate of flow of —. J. Erlich. Ann. Chim. Analyt., 1920, 2, 289—297.

THE pressure produced by the passage of the gas through a capillary connecting the two limbs of a U-shaped differential manometer is registered by the change in level of the liquid in the manometer, the apparatus being calibrated by actual measurement of the gas passed through the apparatus under definite conditions of pressure and time.—W. P. S.

Ultrafiltration; Use of — in toxicological analysis. C. Mannich and G. Wipperling. Ber. deuts. pharm. Ges., 1920, 30, 348—361.

ALTHOUGH substances such as oxalic acid, potassium chlorate, etc., may be separated by ultrafiltration (filtration through collodion membrane), this method cannot be recommended for general use. Small quantities of alkaloids and heavy metals, particularly mercury, are adsorbed by the membrane, and these quantities may be important where traces only of the substances are present. The adsorption of arsenic and antimony by the membrane is, however, practically negligible.

—W. P. S.

Refractometer; Analysis of aqueous solutions with the aid of the —. M. de Crinis. Z. physiol. Chem., 1920, 110, 254—266.

THE refractive index of salt solutions which do not manifest the phenomenon of molecular attraction is in direct linear proportion to the percentage concentration of the salt in the solution, and it is possible to calculate the percentage concentration by means of the formula

$$Y = (nD_x \% - 1.33320) / b,$$

where $nD_x \%$ = refractive index of $x \%$ solution, b = refractive index of 1% solution of the same salt, 1.33320 = refractive index of distilled water. The refractive index of a mixture of equal parts of various salt solutions is equal to the sum of the refractive indices of the separate solutions. If by mixing these salts some substance is precipitated the refractive index of the precipitated salt can be obtained by subtracting the refractive index of the remaining solution from the sum of the refractive indices of the component solutions. The ion content of the aqueous solution can therefore be calculated as follows:—

$$P = (nDR_x \% - nDO_x \%)/K,$$

where P = percentage, $nDR_x \%$ = refractive index of $x \%$ solution obtained by calculation, $nDO_x \%$ = refractive index observed after precipitation, $K = nDR_1 \% - nDO_1 \%$ of a 1% solution.—S. S. Z.

Volumetric analysis; Application of a new physico-chemical method of —. R. Dubrisay. Comptes rend., 1920, 171, 670—672.

THE method previously described (*cf.* J., 1918, 609 A; 1919, 103 A; 1920, 542 A) when applied to the neutralisation of sulphuric acid and sodium hydroxide, shows two points of inflexion on the curve, one corresponding to the formation of sodium hydrogen sulphate and the other to the formation of the normal sulphate. With mixtures of solutions of sodium sulphate and sulphuric acid the maximum difference between the observed and calculated temperatures is reached when the solutions are mixed in equimolecular proportions. The same is true for solutions of sodium sulphate and hydrochloric acid.—W. G.

Potassium; Determination of — as perchlorate, and its separation from sodium, etc. R. L. Morris. Analyst, 1920, 45, 349—368.

THE following procedure is recommended as the result of an exhaustive examination of the perchlorate method. After the potassium salt solution has been evaporated three times with 20% perchloric acid, the practically dry residue is stirred for 15 mins. with 10 c.c. of wash liquid (100 c.c. of 98% by vol. alcohol and 1 c.c. of 20% perchloric acid) and the liquid then decanted through a weighed Gooch crucible containing a layer of asbestos. If potassium only is present, the precipitate is rinsed directly on to the filter with a measured volume of the wash liquid. When sodium is present, the precipitate is heated to expel alcohol, dissolved in the minimum amount of boiling water, the solution evaporated to dryness, the treatment with 10 c.c. of wash liquid and the decantation repeated, and the precipitate rinsed on to the filter with 10 c.c. of the filtrate. The precipitate is washed on the filter with a measured volume of wash liquid, then dried at 130°—150° C. for 1 hr., cooled in a desiccator for 1 hr., and weighed; the weight is taken as being constant when, on washing with 3 c.c. of wash liquid, the difference between successive weighings does not exceed 0.2 mg. If the solution under examination contains phosphate, it should not be evaporated quite to dryness with perchloric acid, and the moist pasty residue should be treated with 15 c.c. of 98% alcohol previous to the treatment with the wash liquid. Calcium, iron, aluminium, and barium salts do not interfere. If magnesium is present, the evaporation must be stopped while the mass is still moist. Citric and tartaric acids tend to char during the evaporation with perchloric acid and a preliminary separation of the potassium as cobaltinitrite appears to be necessary. To determine potassium in potassium sulphate, the sulphuric acid is first precipitated with barium chloride, but it is unnecessary to remove the small excess of barium chloride used. At 15° C., 100 c.c. of the wash liquor dissolves 3.9 mg. of potassium perchlorate.—W. P. S.

Potassium; Determination of — as perchlorate. III. G. P. Baxter and F. E. Rupert. J. Amer. Chem. Soc., 1920, 42, 2046—2049. (Cf. J., 1920, 530 A.)

ETHYL alcohol denaturated with 5% methyl alcohol may safely be substituted for ethyl alcohol in washing potassium perchlorate. The temperature at which the washing takes place is unimportant if the washing liquor employed after the initial extraction is previously saturated with potassium perchlorate. (Cf. J.C.S., Dec.)—J. F. S.

Magnesium; Detection of —. F. Eisenlohr. Ber., 1920, 53, 1476—1477.

FIVE c.c. of a solution of alkannin in alcohol (96%) is treated with a drop of 2N-ammonium carbonate solution, which does not cause any change in colour, and then with a drop of the neutral salt solution to be tested; the presence of magnesium, strontium, or manganese is denoted by the development of a bluish-violet coloration, which becomes pale red after acidification with one or at most two drops of 2N-hydrochloric acid; subsequent addition of the same number of drops of 2N-ammonium carbonate solution restores the bluish-violet colour only if magnesium is present. If the test is to be applied to suspected magnesium ammonium phosphate, the salt is dissolved in 2N-hydrochloric acid and a drop of this solution is added to the alkannin solution; further addition of 1—2 drops of ammonium carbonate causes the appearance of the bluish-violet colour if magnesium is actually present, whilst

otherwise the original colour of the tincture is restored. It is essential that the alcoholic solution should not become diluted with water, since in this case a blue coloration is invariably produced which is due to ammonia formed by hydrolysis of the ammonium carbonate.—H. W.

Magnesium ammonium phosphate; Microchemical "two-phase reaction" for —. H. Kunz-Krause. Ber., 1920, 53, 1672—1673.

THE reaction is specially suited to the detection of magnesium ammonium phosphate in urinary sediments, but may be applied to the detection of phosphates generally. The solution of the sediment in acetic acid is neutralised with ammonia. On addition of a drop of silver nitrate solution, an egg-yellow, cheese-like deposit of silver phosphate is formed which disappears on addition of a drop of ammonia. At the same time the magnesium ammonium phosphate is deposited as colourless, shining prisms united in the form of rosettes. The reaction may be applied to the detection of phosphates as cellular enclosures in plant tissues.

—E. H. R.

Acidimetric determination of heavy metals in their salts. I. M. Kolthoff. Z. anorg. Chem., 1920, 112, 172—186.

EXPERIMENTS were made to determine with what degree of accuracy the salts of heavy metals which form insoluble hydroxides can be determined by titration with alkali hydroxides, either by the conductometric method or by the use of indicators. Magnesium sulphate gave satisfactory results by the conductometric method with barium hydroxide, and also by the indicator method using phenolphthalein. Zinc and copper sulphates gave unsatisfactory results by both methods. Mercuric chloride was determined satisfactorily by the conductometric method by titrating sodium hydroxide solution not greater than N/100 in strength with the mercury solution. In the case of aluminium salts, when these were titrated with sodium or barium hydroxide, a sharp break in the conductivity curve was not obtained at the neutral point, but there was a very sharp break at the point where the formation of aluminate was complete. Aluminium chloride or alum was successfully titrated with sodium hydroxide solution at the boil in presence of excess of barium nitrate, using phenolphthalein as indicator. Slight excess of alkali was run in and the excess titrated with acid. (Cf. J.C.S., Nov.)—E. H. R.

Iron; Separation of — from aluminium by precipitation as Prussian blue. H. Hale and G. O. Burr. J. Amer. Chem. Soc., 1920, 42, 2056—2058.

THE quantitative separation of iron from aluminium as Prussian blue cannot be effected, since the aluminium reacts with the excess of potassium ferrocyanide forming a jelly which cannot be handled. The precipitate of Prussian blue cannot be successfully filtered even after apparently perfect coagulation.—J. F. S.

Iron, mercury, and vanadium; Volumetric determination of — in the same solution. G. Hinard. Ann. Chim. Analyt., 1920, 2, 297—299.

To determine ferric, mercuric, and vanadic sulphates in sulphuric acid solution a portion of the latter is treated with hydrogen sulphide, the mercuric sulphide collected, oxidised with bromine, the solution then rendered alkaline with potassium hydroxide, and a measured excess of standardised potassium cyanide solution added; a few drops of potassium iodide solution are added as indi-

cator, and the excess of cyanide is titrated with silver nitrate solution. $\text{HgO} + 4\text{KCN} + \text{H}_2\text{O} = \text{K}_2\text{Hg}(\text{CN})_4 + 2\text{KOH}$. The filtrate from the mercuric sulphide is boiled to expel hydrogen sulphide, oxidised with bromine, and the iron precipitated as ferric hydroxide by treating the hot solution with potassium hydroxide; the ferric hydroxide is dissolved in hydrochloric acid, again precipitated (to remove remaining traces of vanadium), then dissolved in sulphuric acid and determined iodometrically. $\text{Fe}_2\text{O}_3 + 2\text{HI} = 2\text{FeO} + \text{H}_2\text{O} + \text{I}_2$. The iron and vanadium are then determined together iodometrically in another portion of the original solution; $\text{V}_2\text{O}_5 + 2\text{HI} = \text{V}_2\text{O}_4 + \text{H}_2\text{O} + \text{I}_2$. Slight modifications of the method are required when mercurous, ferrous, and vanadyl salts are also present.

—W. P. S.

Calcium group and magnesium; Precipitation of the —. I. M. Kolthoff. Pharm. Weekblad, 1920, 57, 1229—1234.

THE usual method of precipitating group IV. metals by means of ammonium carbonate is shown to be relatively insensitive, and the use of sodium carbonate in presence of sodium hydroxide is proposed. This throws down the metals of group IV. completely, together with magnesium, the former being separated from the precipitated carbonates by the chromate method, and reprecipitated by means of ammonium carbonate. (*Cf.* J.C.S., Nov.)

—S. I. L.

Nickel (cobalt); Electro-analytical separation of — from arsenic. N. H. Furman. J. Amer. Chem. Soc., 1920, 42, 1789—1793.

NICKEL may be quantitatively separated from salts of arsenic acid in ammoniacal solution by the electric current. Cobalt on deposition carries down arsenic, whereas the nickel deposit is free from arsenic. If the two metals are deposited simultaneously arsenic may or may not be deposited, according to the conditions.—J. R. P.

Zirconium; Determination of —. M. M. Smith and C. James. J. Amer. Chem. Soc., 1920, 42, 1764—1770.

ZIRCONIUM is precipitated by selenious acid from a boiling solution acidified with hydrochloric acid. The basic selenite on ignition leaves zirconia. This method gives satisfactory results in the determination of zirconium in a pure salt, in the separation of zirconium from aluminium and rare earths, and from iron when the amount of ferric oxide in the combined oxides of iron and zirconium does not exceed 10%. Titanium is precipitated together with zirconium, and a correction must be applied in this case or the precipitation carried out in the presence of excess of hydrogen peroxide, when zirconium alone is precipitated. If phosphates are present the zirconium phosphate in the ammonia precipitate is insoluble in hydrochloric acid, and must be fused with sodium carbonate, boiled with water, and the residue dissolved in hydrochloric acid and added to the main solution. For the determination of zirconium in zirconia ore, about 1.25 g. is fused with 12—15 g. of potassium hydrogen fluoride, the cooled melt is treated with 50 c.c. of sulphuric acid (1:1), heated gently at first and then more strongly until abundant fumes are evolved, cooled, boiled with water, and diluted to 250 c.c. 100 c.c. of the solution is diluted and precipitated with ammonia in presence of ammonium chloride. The precipitate is washed, dissolved by boiling with 36 c.c. of concentrated hydrochloric acid and 40 c.c. of water, diluted to 700 c.c., and precipitated with a 12.5% solution of selenious acid. The precipitate is collected, washed with 3% hydrochloric acid, dried, and ignited.—J. R. P.

Carbon monoxide; Determination of — in air contaminated with motor exhaust gas. M. C. Teague. J. Ind. Eng. Chem., 1920, 12, 964—968.

UNDER controlled conditions the apparatus devised by Graham (J., 1919, 10r) is capable of determining carbon monoxide in air with an accuracy of 0.005%, but in the case of air contaminated with motor exhaust gas, hydrocarbons must be removed before oxidising the carbon monoxide with iodine pentoxide. In a new portable apparatus described this is effected by passing the gas through U-tubes immersed in liquid air, which condenses the water, carbon dioxide, gasoline, and traces of other saturated and unsaturated hydrocarbons. The hydrogen, methane, and carbon dioxide pass into iodine pentoxide tubes, which are heated in an oil bath to 100°—175° C. Only the carbon monoxide is oxidised, the concentration of the hydrogen being too low to affect the results. The liberated iodine is absorbed by potassium iodide solution and titrated with sodium thiosulphate solution. A determination takes 15 mins., and should be accurate to 0.003—0.005% of carbon monoxide.

—C. A. M.

Hydrocarbons; Absorbent for heavy — [in gas analysis]. A. Piechota. Chem.-Zeit., 1920, 44, 797.

HEAVY hydrocarbons may be removed completely from a gas by passing the latter into an ordinary absorption pipette containing a saturated solution of potassium bichromate in concentrated sulphuric acid. About 10 mins.' contact is required, but it is better to pass the gas in and out of the pipette about twenty times.—W. P. S.

Fatty acids; Influence of electrolytic dissociation on the distillation in steam of the volatile —. J. Reilly and W. J. Hickinbottom. Sci. Prec. Roy. Dublin Soc., 1920, 16, 120—130.

THE deviations in the distillation constants of dilute solutions of volatile fatty acids (*cf.* J., 1919, 913 A) are explained on the basis of electrolytic dissociation. A mathematical expression to represent this and to correct the constants is given, and it is thus shown that the distillation constant is practically independent of the dilution. The effect of certain salts and acids in increasing the distillation constant of acetic acid is dependent on the nature of the salt added. (*Cf.* J.C.S., Dec.)—W. G.

See also pages (A) 741, *Iodine value of mineral oils* (Röderer), *Paraffin wax in ozokerite* (Koss); 746, *Carbon dioxide* (Hartmann), *Soda-lime* (Wilson); 747, *Cyanic acid* (Fosse); 748, *Radium* (Deaigies), *Naturally-occurring gases* (Henrich); 752, *Aluminium alloys* (Berry); 753, *Magnesium, cobalt, and chromium alloys* (Camp and Marden); 755, *Iodine value of fats* (Devrient); 757, *Factice in rubber* (Dekker); 758, *Sulphide in lime liquors* (Pickles), *Alkalinity of lime liquors* (Atkin); 759, *Sucrose, invert sugar, and raffinose* (Montgomery), *Pentose sugars* (Spoehr); 760, *Histidine* (Hanke and Koessler), *Histamine* (Hanke and Koessler); 761, *Ammonia etc. in water* (Kolthoff), *Morphine in opium* (Jernstad); 762, *Cinchona preparations* (Lehmann), *Dioxymethylene groups in alkaloids* (Gadamer), *Sulphonal and trional* (Zimmermann); 763, *Aromatic antimony compounds* (Schmidt), *Cineol* (Kleber and von Rechenberg); 764, *Acetaldehyde and aldols* (Hammarsten), *Acetaldehyde* (Stüwe), *Iodoform reaction* (Kunz).

PATENTS.

Hardness of ferrous metals; Testing —. L. W. Wild. E.P. 151,383, 30.6.19.

THE specimen is introduced within a standard magnetising coil, through which current is passed for a period somewhat less than a second. The speci-

men is removed and introduced into a ballistie coil connected with a fluxmeter, preferably of the Grassot type. The specimen is quickly withdrawn from the coil, and the resulting throw of the fluxmeter serves, after calibration of the instrument, to indicate the physical hardness of the specimen.

—J. S. G. T.

Thermo-elements; Protective covering for —.

Siemens und Halske A.-G. G.P. 322,919, 6.7.18.

As incombustible, insulating material, of a pulverulent nature, e.g., sand, fireclay, or magnesia, is packed into the protecting tube of the thermocouple.—W. J. W.

Colorimeter. F. G. Spindler, Assr. to A. Traeger.

U.S.P. 1,353,500, 21.9.20. Appl. 4.8.19.

The claims relate to means for supporting the materials to be tested in such a way that they can be moved away from or towards the eyepiece of the instrument.

Gas-colorimeter. W. G. Laird, Assr. to H. L. Doherty. U.S.P. 1,351,568, 5.10.20. Appl. 20.12.18.

A CURRENT of water is passed through a heat-absorption chamber in which the gas is burnt, and flows thence to a gas-reservoir, from which it displaces its own volume of gas, forcing it to the gas burner. Means are provided for measuring the temperature of the water passing through the heat-absorption chamber.

Removing carbon monoxide from gases. G.P. 303,931. See II A.

Soda-lime. U.S.P. 1,333,521. See VII.

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Boynton and Webster. Producing interactions between gas and liquid. 30,351. Oct. 27. (U.S., 20.11.19.)

Capro. Filters. 30,588. Oct. 29.

Drake and Roydhouse. Crushing and pulverising apparatus. 30,429. Oct. 27.

Duckham, Kent, and Woodall. Duckham, and Jones. Tunnel kilns. 30,653. Oct. 29.

Dunsford. Atomising liquids. 30,261. Oct. 26.

Emmens. Apparatus for distilling or boiling liquids. 30,387. Oct. 27.

Fothergill. Evaporators. 30,120. Oct. 27.

Grant and Jones. Apparatus for separating substances from gases etc. 30,951. Nov. 2.

Harris. Dehydrator. 31,100. Nov. 3.

Hatfield, and Imperial Trust. Means of separating substances. 31,242. Nov. 4.

Hauenschild. Shaft furnaces. 30,142. Oct. 25.

Hilgendorff and Kousnetzoff. Apparatus for utilising heat of outlet gases. 30,291. Oct. 26.

Kerr and O'Connell. 31,201. See XIX.

Kurth and Mogel. Furnaces. 31,458. Nov. 6. (Ger., 27.2.20.)

MacLachlan. 30,661-2. See XIX.

Morgan, and Thermal Industrial and Chemical Research Co. Process of heating substances for producing chemical change. 30,975. Nov. 2.

Perry and Co., and Rallings. Rotary mullers or furnaces. 31,400. Nov. 6.

COMPLETE SPECIFICATIONS ACCEPTED.

14,816 (1919). Whitfield. Drying-apparatus. (152,712.) Nov. 3.

15,845 (1919). Smallwood. Furnaces. (153,012.) Nov. 10.

18,311 (1919). British Thomson-Houston Co. (General Electric Co.). Boilers particularly for vaporising mercury or other fluid of high boiling point. (152,785.) Nov. 3.

18,531 (1919). Siegwart and Siegwart. Operation of furnaces. (130,596.) Nov. 3.

23,859 (1919). Zwermann. Kilns. (140,740.) Nov. 3.

32,294 (1919). Thoens. Refrigerating. (152,898.) Nov. 3.

6819 (1920). Killby and Allen. Drying apparatus. (152,922.) Nov. 3.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Aymard. Manufacture of gas. 30,435. Oct. 27.

Black. Retorts for distilling oil-bearing shales etc. 30,724. Oct. 30.

Chown. Treatment of peat. 31,097. Nov. 3.

Clifton and Steele. Fuel for internal-combustion engines. 30,510. Oct. 28.

Crowden. Drying peat. 30,524. Oct. 28.

Davidson. Preparation of peat fuel. 30,866. Nov. 1.

Dootson and McClelland. 30,127. See III.

Edser. Fuel. 30,655-6. Oct. 29.

Frewen. Treatment of peat. 31,267. Nov. 4.

General Electric Co., and Goucher. Drawn-wire filaments. 30,552. Oct. 28.

Gewerkschaft Enischer Lippe, and Heyn. Coking plants. 30,947. Nov. 2. (Ger., 3.11.19.)

Greenstreet. Producing and transporting fuel. 30,792. Nov. 1.

Haynes and Williams. Production of carbon. 31,327. Nov. 5.

Klärding. Purification of blast-furnace and generator gases. 30,525. Oct. 28. (Ger., 31.10.19.)

Lamplough, and Oil Extractors, Ltd. Retorts for low-temperature distillation. 31,200. Nov. 4.

Leadbeater. Manufacture of metallurgical coke. 30,925. Nov. 2.

Lewis. Protective progressive distillation and gasification of solid carbonaceous matters. 30,537. Oct. 28.

Lockett and Sinnatt. Manufacture of combustible material from coals, peats, etc., and sewage and trade waste sludges. 30,471. Oct. 28.

Maclaren. Utilisation of wet powdered fuel. 30,749. Oct. 30.

Marriott. Liquid for reducing fuel consumption in internal-combustion engines. 31,073. Nov. 3.

Sauer. 30,285. *See* XVII.

Soc. d'Exploit. Brev. Arnould. *See* III.

Wells. Gas-producers. 31,082. Nov. 3.

COMPLETE SPECIFICATIONS ACCEPTED.

16,311 (1919). Meade. Manufacture of water gas. (153,051.) Nov. 10.

17,686 (1919). British Thomson-Houston Co. (General Electric Co.). *See* X.

17,728 (1919). Poore. Destructive distillation of wood, woody fibre, etc. (152,741.) Nov. 3.

17,912 (1919). Hickman. Apparatus for washing and drying gas from blast furnaces, gas-generating plants, etc. (153,053.) Nov. 10.

18,405 (1919). Allan. Apparatus for continuous distillation and fractionation of petroleum or other hydrocarbons or mixtures of volatile liquids. (152,791.) Nov. 3.

19,478 (1919). Wells. Tar extractors and scrubbers for gas. (153,109.) Nov. 10.

21,383 (1919). West, Wild, and West's Gas Improvement Co. Heating vertical retorts. (153,139.) Nov. 10.

25,317 (1919). Allgem. Ges. f. Chem. Ind. Continuous treatment of hydrocarbons with liquid sulphur dioxide. (133,962.) Nov. 10.

28,326 (1919). White (Soc. Franco-Belge de Fours à Coke). Recovery of ammonia from coke-oven gases. (153,177.) Nov. 10.

28,360 (1919). Soc. Anon. d'Oughrée-Marihaye. Vertical gas retorts. (135,214.) Nov. 3.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

Baddiley, Payman, Wignall, and British Dyestuffs Corp. Manufacture of ortho-sulphonic acids of aromatic amines. 31,140. Nov. 3.

Dootson and McClelland. Production of light oils, lubricating oils, and gas by cracking pitch and heavy oils. 30,127. Oct. 25.

Norsk Hydro-Elektrisk Kvaelfstafaktieselskab. Production of dinitrophenol. 30,558. Oct. 28. (Norway, 29.10.19.)

Soc. d'Exploit. des Brevets C. Arnould. Obtaining pitch from tar etc. oils. 31,265. Nov. 4. (Fr., 13.8.20.)

Wilson. Distillation of tar. 30,696. Oct. 30.

COMPLETE SPECIFICATION ACCEPTED.

10,285 (1918). Ransford (Cassella u. Co.). Manufacture of anthraquinone derivatives. (153,055.) Nov. 10.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Denocl. Apparatus for testing sizing of paper. 31,396. Nov. 6. (Belg., 6.11.19.)

Dreaper. Manufacture of artificial filaments etc. 31,394. Nov. 6.

Kämpf. Manufacture of nozzles for producing artificial silk etc. 31,116. Nov. 3. (Ger., 12.3.20.)

Russell. Treatment of paper-making materials and liquors and by-products obtained therefrom. 30,872. Nov. 1.

Thornton (Feculose Co.). Method of sizing paper. 31,367. Nov. 5.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Ashmore, Cochrane, and Calico Printers' Assoc. Production of colour effects on fabrics. 30,140. Oct. 25.

Bangle and others. Dyeing-machines. 30,644. Oct. 29.

Edser and Reynard. 31,456. *See* IX.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Camus, Criquebœuf, and Duchemin. Manufacture of acetic acid and its homologues. 30,311. Oct. 26.

Chaillanx. 30,268. *See* XIII.

Dykes and Maconochie. Furnaces for and manufacture of zinc oxide. 31,077. Nov. 3.

Schantz. Production of chloride of mercury. 31,350. Nov. 5.

Wohl. 31,195. *See* XX.

Wolcott. Production of aluminium chloride. 30,793. Nov. 1.

COMPLETE SPECIFICATIONS ACCEPTED.

17,929 (1919). Petersson, and Metals Extraction Corp. Treatment of zinc solutions. (152,752.) Nov. 3.

18,058 (1919). Lessing. Manufacture of sulphate of ammonia. (152,766.) Nov. 3.

19,601 (1919). Wade (Pennsylvania Salt Manuf. Co.). Manufacture of basic sulphate of zirconium. (153,113.) Nov. 10.

19,969 (1919). Beakbane and Arnot. *See* XXII.

21,039 (1919). Deuts. Molybdän Werke. *See* X.

28,326 (1919). White. *See* II.

29,982 (1919). Toniolo. Oxidation of ammonia. (137,036.) Nov. 10.

30,977 (1919). Pederson. *See* X.

489 (1920). Fredrikstad Elektrokem. Fabr. Manufacture of sodium perborate (139,753.) Nov. 3.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Bailey. Tunnel ovens etc. for firing pottery, tiles, etc. 30,577. Oct. 29.

Kaufman, Nicols, and Scholes. Removing striæ from melted glass. 30,797. Nov. 1.

Lamarque. Obtaining agglomerates capable of being cut and polished. 30,636. Oct. 29.

Marks (Buffalo Refractory Corp.). Refractory compositions. 30,964—5. Nov. 2.

Wade (Titanium Pigment Co.). Manufacture of glassware. 30,657. Oct. 29.

COMPLETE SPECIFICATIONS ACCEPTED.

18,247 (1919). Crossley. Manufacture of non-frangible glass. (152,780.) Nov. 3.

14,969 (1920). Frink. Drawing glass. (144,629.) Nov. 10.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Edser and Reynard. Protecting surfaces and waterproofing articles. 31,456. Nov. 6.

Hartner. Manufacture of mortar-forming material from anhydrite etc. 31,358. Nov. 5. (Ger., 25.11.19.)

Nielsen. Producing or treating cement objects. 30,277. Oct. 26.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Aitchison and Dyson. Purification of ores or residues containing metallic oxides. 30,507. Oct. 28.

Aitchison and Dyson. Purification of tungsten ores or residues containing tungsten oxide. 30,508. Oct. 28.

British Thomson-Houston Co. (General Electric Co.). Alloys. 30,390—1. Oct. 27.

General Electric Co., and Goucher. 30,552. See II.

Haworth. Alloy for repairing defective castings. 30,785. Nov. 1.

Klärding. 30,525. See II.

Leadbeater. 30,925. See II.

Melamid. Manufacture of oil for foundry cores. 30,635. Oct. 29.

Perry and Co., and Rallings. 31,400. See I.

Schalenbach. Melting furnaces. 31,347. Nov. 5.

Soc. Anon. de Commentry, Fourchambault et Decazeville. Alloys. 31,378—31,380. Nov. 5. (Fr., 26—28.2.20.)

Turton. Apparatus for electrodeposition of metals. 31,052. Nov. 3.

COMPLETE SPECIFICATIONS ACCEPTED.

5227 (1918). Schmidt. Smelting furnace heated by oil or gas. (152,697.) Nov. 3.

18,311 (1919). British Thomson-Houston Co. See I.

21,039 (1919). Deuts. Molybdän Werke. Extraction of molybdenum compounds from yellow lead ores. (131,897.) Nov. 10.

21,827 (1919). Smith. Deposition of copper on non-conducting surfaces. (152,835.) Nov. 3.

22,388 (1919). Marks (Union Carbide Co.). Alloy particularly for treating molten iron and steel. (152,840.) Nov. 3.

24,744 (1919). Electrolytic Zinc Co. Roasting zinc ores preparatory to leaching. (134,825.) Nov. 10.

30,977 (1919). Pederson. Extraction of sulphur from sulphide ores. (152,887.) Nov. 3.

180 (1920). Cachemaillo (Westinghouse Lamp Co.). Wire-drawing lubricants. (137,329.) Nov. 3.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Chlorido Electrical Storage Co., and Dean. Secondary electric batteries. 30,267. Oct. 26.

Turton. 31,052. See X.

COMPLETE SPECIFICATIONS ACCEPTED.

19,895 (1919). Fuller, Fuller, and Fuller. Galvanic batteries. (152,818.) Nov. 3.

21,827 (1919). Smith. See X.

28,845 (1919). Ashcroft. Electrolytic apparatus. (152,879.) Nov. 3.

811 (1920). Wade (Booth Electric Furnace Co.). Electric furnaces. (152,904.) Nov. 3.

2657 (1920). Norskø Akt. for Elektrokemisk Industri. Suspension of electrodes in electric furnaces. (138,354.) Nov. 10.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Bickel. Hydraulic oil press. 30,438. Oct. 27.

Jackson (American Cotton Oil Co.). Hydrogenation of oils and liquid fats. 31,145. Nov. 3.

Parodi. Extraction and refining of oils etc. 31,436. Nov. 6. (Fr., 20.3.18.)

Sauer. 30,285. See XVII.

Tonkin. Extracting edible fats from seeds, nuts, etc. 31,231. Nov. 4.

COMPLETE SPECIFICATIONS ACCEPTED.

17,709 (1919). Lessing. Applying nickel carbonyl in hydrogenation processes. (152,740.) Nov. 3.

22,455 (1919). Willis. Apparatus for extracting and purifying beeswax. (152,841.) Nov. 3.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Chaillaux. Manufacture of gold-coloured sulphurs and vermilion of antimony. 30,268. Oct. 26.

Dykes and Maconochie. 31,077. See VII.

Ellis. Production of artificial gums. 30,203. Oct. 26.

Fraymouth, Nagle, and Kestner. Evaporator and Engineering Co. Separating impurities from sticklac to obtain pure lac resin. 31,199. Nov. 4.

COMPLETE SPECIFICATIONS ACCEPTED.

18,726 (1919). Hutchison. Non-inflammable and waterproof paint. (153,081.) Nov. 10.

4576 (1920). Guimet and Guillochin. Manufacture of ultramarine. (152,916.) Nov. 3.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Etty. Tanning. 30,532. Oct. 28.

Johnson (Badische Anilin u. Soda Fabr.). Tanning. 30,276. Oct. 26.

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Ges. f. Landwirtsch. Bedarf. Manufacture of artificial manure. 30,160. Oct. 25. (Ger., 24.2.19.)

Kowalski. 31,354. See XVII.

COMPLETE SPECIFICATION ACCEPTED.

18,234 (1919). Molassine Co., and Whalley. Treating peat and production of a preparation for use in horticulture and agriculture. (155,779.) Nov. 3.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

Ellis. 30,203. See XIII.

Gilmour. 31,257. See XX.

Kowalski. Purification of raw juices, waste waters, etc., manufacture of manures and food for animals etc. 31,354. Nov. 5.

Sauer. Neutral and sterile decolorising carbon. 30,285. Oct. 26.

XVIII.—FERMENTATION INDUSTRIES.

COMPLETE SPECIFICATION ACCEPTED.

18,428 (1919). Takamine and Takamine. Preparation of enzyme extracts. (152,792.) Nov. 3.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Bendixen. Making liquid milk from dried milk, and artificial milk from casein. 30,714. Oct. 30.

Kerr and O'Connell. Internally heated or cooled rollers for drying, heating, or cooling milk etc. 31,204. Nov. 4. (Australia, 3.12.19.)

Kowalski. 31,354. See XVII.

Lockett and Sinnatt. 30,471. See II.

Maclachlan. Deodorising gases. 30,661. Oct. 29. (U.S., 28.7.20.)

Maclachlan. Continuously treating waste matter. 30,662. Oct. 29. (U.S., 28.7.20.)

Morison. Water purifiers. 30,809. Nov. 1.

Raeburn, Simpson, and Simon, Ltd. Parboiling and gelatinising etc. rice. 30,904. Nov. 2.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Camus and others. 30,311. See VII.

Drew and Morgan. Organic selenium and tellurium compounds. 31,139. Nov. 3.

Fabr. de Prod. Chim. de Thann et de Mulhouse. Manufacture of borneol. 30,873. Nov. 1. (Fr., 4.2.20.)

Gilmour. Production and utilisation of compounds of sugars and boric acid. 31,257. Nov. 4.

Nitrum A.-G., and Schellenberg. Converting lime-nitrogen into urea. 30,116. Oct. 25. (Switz., 7.11.19.)

Ransford (Cassella u. Co.). Process for rendering stable preparations of serum, lymph, etc. 31,261. Nov. 4.

Wohl. Production of aldehyde and acetic acid. 31,195. Nov. 4. (Ger., 24.11.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

4827 (1920). Chem. Fabr. vorm. Sandoz. Selective isolation of alkaloids. (153,219.) Nov. 10.

8208 (1920). Commercial Research Co. Manufacture of benzoic acid. (141,333.) Nov. 10.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Colour Photography, Ltd., and Shepherd. Colour photography. 30,748. Oct. 30.

Dawson and Howard. Natural-colour photography. 31,113. Nov. 3.

Taylor. Photographic plates, films, etc. 31,461. Nov. 6.

XXII.—EXPLOSIVES; MATCHES.

COMPLETE SPECIFICATION ACCEPTED.

19,969 (1919). Beakbane and Arnot. Removing amatol from shells etc. to recover the ammonium nitrate. (153,123.) Nov. 10.

XXIII.—ANALYSIS.

APPLICATION.

Siemens u. Halske A.-G. Photometers. 30,637. Oct. 29. (Ger., 5.11.19.)

I.—GENERAL ; PLANT ; MACHINERY.

Catalysis; Promoter action in —, R. N. Pease and H. S. Taylor. *J. Phys. Chem.*, 1920, 24, 241–265.

PROMOTER action in catalysis is understood as including all those cases in which a mixture of two or more substances is capable of producing a greater catalytic effect than can be accounted for on the supposition that each acts independently and in proportion to the amount present. An example is furnished by the use of iron-molybdenum in the synthesis of ammonia. Iron and molybdenum are both catalysts for the reaction, but a mixture of equal parts of the two is much superior as a catalyst to either alone. A distinction is made between this type of activation (co-activation) and cases (activation) in which a relatively inert substance increases the activity of a catalyst. Neutral salt action is an example of the second type. Examples of promoter action in heterogeneous and homogeneous catalysis and enzyme action are given.

—J. R. P.

Vaporisation formula; Clausius' — and a comparison of the vapour pressure curves of two substances. F. A. Henglein. Z. Elektrochem., 1920, 26, 431—436.

A FORMULA has been derived which holds even to the highest temperatures, viz.,

in which T_1 and δ_1 are the boiling points of a substance at two different pressures, and T_2 and δ_2 the boiling points of another substance at the same two pressures. The constant, k , is approximately equal to the ratio of the molecular heats of vaporisation of the two substances at the same pressure. Another formula, $\log T_1 = a \log T_2 + b$, which holds up to the critical point, has also been developed. In this formula T_1 and T_2 are the temperatures at which both substances have the same vapour pressure, a and b are constants. It is possible by means of this formula to determine the vapour pressure curve of a substance from that of water.—J. F. S.

Gases from fire extinguishers. Fieldner and others.
See XIX_B.

PATENTS.

Dehydrating, reducing, calcining or roasting minerals and other pulverulent material; Apparatus for —. J. Réol. E.P. 130,978, 26.7.19. Conv., 9.8.18.

THE material is fed by a distributor at the top of a tower containing annular hearths arranged as steps in flights from the centre to the periphery and from the periphery to the centre. The material is pushed from step to step by rakes actuated by a central shaft and falls through a current of hot gas admitted at either end of the tower and exhausted at the other through a dust catcher.—B. M. V.

Extracting liquid from fresh peat, beet mass, and similar watery materials; Apparatus for —.
N. T. Thäberg. E.P. 151,907, 15.4.20.

A PRESS filter is formed of a number of flat elastic metal plates arranged edgewise side by side. The plates may be initially slightly curved or corrugated so that the spaces between them may be varied by moving the plates to or from one another. The plates are mounted on links to form an endless chain driven by sprocket wheels or rollers, and the chain passes between two converging plates to exercise pressure on the material and press the liquid through the filter. The converging plates may be

fixed or one may be an endless belt travelling in the same direction as the filter chain.—W. F. F.

Separating crystals from mother liquors containing more than one salt. J. T. Windram. E.P. 152,041, 13.8.18.

The mother liquor is centrifuged at an appropriate temperature and the crystal mass remaining in the centrifuge immediately treated with a fine spray of water or wet steam so that no substantial alteration of temperature of the mass occurs. The centrifuge continues to run during this treatment and during subsequent discharge of the crystal mass.

—J. S. G. T.

—J. S. G. T.

Distilling or recovering volatile inflammable liquid solvents; Process of —, W. K. Lewis and W. Green. E.P. 152,374, 31.1.19.

To remove benzol or gasoline from fabrics etc. the volatile solvent is evaporated by clean flue gas, being afterwards recovered by condensation or by washing with oil or other absorbent medium. The flue gas may be re-heated and used again.

—B. M. V.

Kilns; Continuous regenerative gas-fired — for burning ore briquettes and high temperature refractory materials. C. F. Priest. E.P. 152,509, 6.10.19.

TWENTY or more chambers are arranged on either side of a central main flue and each is provided with a controlled supply of gas. Through the dividing walls between adjacent chambers are passages providing communication either between adjacent chambers only or, in addition, by means of man-holes with the air or by means of a damper-controlled outlet with the main flue. The chambers are preferably worked in groups of seven, air being admitted (through the appropriate manholes) to the first chamber and gas to the middle chamber; in the first three chambers the material is cooled and the air heated, and in the last three chambers the material is pre-heated and the waste gases cooled. When the material in the fourth chamber is properly burnt the first chamber is cut out, and the eighth brought into operation.—B. M. V.

Mixer. J. Johnson. U.S.P. 1,354,489, 5.10.20.
Appl., 7.6.16. Renewed 21.2.19.

A ROTATING mixing wheel in a casing receives fluid to be mixed through central openings on opposite sides. The fluid is given a radial motion by centrifugal force and is directed to the side of the casing opposite to that of its entry.—W. F. F.

Grinding machine. J. Hereche. U.S.P. 1,355,093,
5.10.20. Appl., 25.1.17

A GRINDING roller is provided with longitudinal helical furrows separated by grinding ribs. A series of separate grinding bars are arranged parallel to the axis of the roller and at one side only, the faces of the grinding bars being corrugated or roughened to give a texture of increasing fineness from the upper to the lower bar. The bars are arranged with their upper longitudinal edges spaced further from the roller than the lower edges, so that a space of tapering cross-section is provided for the passage of the material.—W. P. F.

Tunnel dryer. Internat. Ges. für Trockenanlagen
m.b.H. G.P. (a) 322,783, 29.11.14, and (b)
322,784, 11.11.15.

(A) The goods are carried through the dryer on a series of wagons. The front wagon of the series acts as a connecting wagon and can be pushed into the blast-chamber, leaving the next one free to be taken away without difficulty. The connecting

wagon is then again connected with the train until another wagon is ready for removal. The connecting wagon is permanently attached by a flexible connexion to the mouth of the blast-chamber. (B) The dryer consists of a series of wagons divided into compartments and pushed against each other. Each wagon carries a part of the air-pipe and the last wagon, which is without divisions, is closed, so that it diverts the air back along the other wagons through a return pipe. By this means the air can be circulated backwards and forwards from the air-chamber over the material to be dried.—A. R. P.

Drying trough with fixed drum and revolving mixing and stirring shovels. W. Wurl. G.P. 323,462, 7.5.14.

The drying medium, e.g., hot air or gases, passes through a branched tube arranged along one side of the drying chamber, which is fitted with revolving discs carrying shovels, over the material to be dried, and out through a similar tube on the other side of the chamber. The revolution of the shovels through the slimy mass (e.g., sewage sludge), for the drying of which the furnace is especially designed, causes an intimate mixing of the hot gases with the material to be dried and also heats the stirring gear, so that the operation is carried out efficiently and quickly.—A. R. P.

Filtering membranes; Process for permanently glazing —. E. de Haën, Chem. Fabr. "List" G.m.b.H. G.P. 323,185, 29.8.17.

The moist discs of prepared filtering-membrane stretched on a frame are dipped for a short time in hot water or treated with steam, whereby a surface is produced which is so smooth that a completely air-tight connexion between the membrane and its support can be obtained. The membrane may be dried without any alteration of the physical properties of the surface.—A. R. P.

Washing and rectifying columns; Hood exits for —. H. Frischer. G.P. 323,295, 17.1.18.

The hood is provided with two, three, or more series of outlet openings so arranged that they are not in alignment in adjacent series. Between each series a partition is constructed with an opening at the foot so that an uninterrupted delivery of liquid and gas or vapour may be obtained.—A. R. P.

Distillation apparatus; Electrically heated —. Allgem. Elektrizitäts-Ges. G.P. 323,414, 26.10.18.

The liquid is heated by utilising its resistance according to the electrode principle in a special heater with which is combined a storage tank through which a condenser worm passes so that the condensation of the vapours from the heater serves to preheat a fresh quantity of liquid. By working in this manner it is possible to distil liquids by simply passing a current through them, and the heat economy obtained is much greater than in any previous apparatus.—A. R. P.

Perforated plate for apparatus for the mutual interaction of liquids and gases or vapours. H. Pauling. G.P. 323,296, 17.1.18.

The holes of the perforated plate are made conical from each side towards the middle, so that the plate may be used from either side. This has the advantage of requiring a smaller pressure for the same rate of flow of the same volume of gas through an equal number of holes; also the conical form ensures that during the passage of the liquid through the holes they become filled with liquid, which remains there by capillary attraction.—A. R. P.

Gases and vapours; Apparatus for treating — with liquids. H. Frischer and M. Drees. G.P. 323,474, 30.10.17.

The reaction chamber is subdivided by perpen-

dicular walls into separate compartments for the reacting liquids; each separate compartment is divided by horizontal walls into a series of superimposed chambers which are connected with one another in such a manner that the gases and vapours pass through the streams of liquid successively in each horizontal group in an up and down direction. While the gases are passing through the perpendicular chambers they come in contact with a number of different absorption liquids. It is possible in this apparatus to separate one and the same component from a number of different liquids without the residual liquids becoming mixed, and to rectify or purify gases and vapours.—A. R. P.

Ball or tube mills. N. L. Hall. E.P. 152,398, 8.7.19.

SEE U.S.P. 1,315,770 of 1919; J., 1919, 805 A.

Shaft-furnaces and such like; Blast-proof closing of discharging devices for —. H. Stebmann. E.P. 148,818, 10.7.20. Conv., 4.5.17.

Furnaces; [Burners for] oil-fired —. E. L. Woolley. E.P. 152,920, 2.3.20.

Drying apparatus. E.P. 152,505. See VIII.

Catalysts. U.S.P. 1,329,322-3. See XII.

IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Determination of the calorific value of the volatile matter of — as a measure of its suitability for gas manufacture. R. Mezger and M. Müller. J. Gasbeleucht., 1920, 63, 669-673.

The disadvantages of the methods of analysis described by Strache (J., 1911, 1245) and by Hiller (J. Gasbeleucht., 1916, 129) are pointed out, and the method of the authors is claimed to be simpler and more easily performed. The apparatus consists of a quartz tube of 12 cm. length and 6 mm. diam., which is sealed at one end. The other end is connected by capillary tubing with a Hempel gas burette. 0.2-0.5 g. of coal is weighed into the quartz tube, which is placed in a furnace heated by two Teclu burners, the temperature being maintained at 1000° C., using a Le Chatelier pyrometer for control. The tar is deposited on asbestos wool, filled into the free space in the quartz tube, this portion of the tube being cooled by water. The gas is cooled to atmospheric temperature, measured, and analysed, and from the analysis its calorific value can be calculated. With all coals studied, the calorific value of the volatile products was lower than that calculated by subtracting the calorific value of the coke from that of the original coal, whence it must be concluded that loss of heat took place due to exothermic reactions during gasification.—A. G.

Producer-gas and coal-dust firing; Comparative observations on the utilisation of heat in —. E. Terres. J. Gasbeleucht., 1920, 63, 673-675.

Tests were made with a lignite containing: moisture 53.63%, C 27.04%, H 1.72%, O 11.0%, N 0.39%, S 0.79%, and ash 5.43%. The calorific value of the raw lignite was 2500 Cals. per kg. For dust firing the lignite was dried to 15% moisture content and ground. The limiting temperature obtained by this method was 1850° to 2050° C., according to the amount of excess air added for combustion. In the second process "compound gas" was produced by carbonisation and subsequent gasification in lignite producers. The distillation gas produced from 100 kg. of lignite amounted to 20 cb.m. and contained 22.0% CO,,

1.0% heavy hydrocarbons, 11.0% CO , 48.0% H_2 , 10.0% CH_4 , and 5.0% N_2 , whilst the producer gas averaged 7% CO_2 , 25% CO , 10% H_2 , and 58% N_2 , and amounted to 119 cb.m. The temperature of combustion of the mixed gas is given as 1480° — 1625° C. By preheating the air supply to 850° C., this was increased to 1810° — 1960° C., whilst if the gas was also preheated to 850° C. before combustion, temperatures of 2080° C. (1 vol. of gas to 3 vols. of air) to 2250° C. (1 vol. of gas to 1 vol. of air) were attained. The author gives the thermal efficiencies of lignite-blast and gas-firing as 75.73 and 53.29 respectively, the difference representing the heat required to convert the lignite into gas.—A. G.

Flame; Propagation of — in mixtures of methane and air. *Part II. Vertical propagation. Part III. Propagation in currents of the mixtures.* W. Mason and R. V. Wheeler. *Chem. Soc. Trans.*, 1920, 117, 1227—1240.

WHEN mixtures of methane and air are ignited at the open end of a vertical tube closed at the other end, the first phase in the propagation of flame is the uniform movement. The speeds at which the flames travelled downwards with different mixtures were uniform over a considerable distance from the point of ignition, and were about two-thirds of those found for horizontal propagation in the same mixtures; transference of heat by convection presumably plays a considerable part in aiding the propagation of flame during the horizontal uniform movement. With upward propagation the flames sometimes began to vibrate within 50 or 60 cm. of the open end of the tube. The speeds over the whole range of mixtures were 3—5 cm. per second less when the flames travelled upwards than when they travelled horizontally. This slight difference probably arises from the difference in the shape of the flame front. Flames travelling upwards in mixtures containing 9—11% CH_4 were very sensitive to the effect of resonance of the tube, and could acquire either of two speeds, but not speeds intermediate between the two, the lower speed in the one case being shown, photographically, to be accompanied by, and probably due to, the setting up of resonance. The shape of the flame front was different when resonance was set up. If mixtures are ignited at one end of a vertical tube open at both ends, the tube acts as a chimney, and greatly enhanced speeds are obtained with flames travelling upwards owing to the effect of the draught and the turbulence it sets up in the mixture. Flame does not travel downwards if the mixture is ignited at the top of the tube, but continues to burn at the mouth of the tube. When the mixture is ignited at the closed end of a tube open at the other end the position of the tube does not affect the results. The lower limit of inflammability of methane, horizontal propagation, is 5.4% in a still mixture. With the mixture moving between 35 and 65 cm. per second along the tube, a flame passed through a mixture containing only 5.02% CH_4 , at the speed of the current. Comparatively gentle movement of the mixture along a tube resulted in greatly increased speed of flame, the major effect of the initial movement given to the mixture being evidently due to turbulence.—W. P.

Paraffin wax candles; Manufacture of —. Lach. Seifenfabr., 1920, 40, 325. *Chem.-Zeit.*, 1920, 44, Rep., 244.

THE wax is purified by treatment with heavy petroleum spirit, and with sulphuric acid (66° B., sp. gr. 1.84) at 120° C., and is decolorised by means of animal charcoal etc. Water is eliminated by melting the wax in jacketed vessels. For crystalline candles the melted wax is cooled to about 20° — 30° C. above its m.p., whilst for stearine-like candles it is stirred while it cools until it begins to solidify. Composite candles contain less than 5% of stearine.

Candles containing 2—3% of stearine and 5% of alcohol have a good appearance, but gutter when burned. The addition of a benzylnaphthol compound in place of alcohol improves the appearance, but not the burning qualities of the candles. Wicks with 24 threads are the most suitable for paraffin wax candles; wicks for night-lights must be coated with wax material.—C. A. M.

Hardwoods. Dore. *See* V.

Ammonium carbonate solutions. Terres and Weiser. *See* VII.

Carbon black. Neal. *See* XIII.

PATENTS.

Briquetting coal and other powdered material; Process for —. Rütgerswerke A.G., and H. Teichmann. G.P. 325,072, 18.4.19.

THE product obtained by heating coal with heavy coal-tar oils above 300° C., with subsequent removal of a portion of the oil, is employed as a binder in manufacturing briquettes.—L. A. C.

Gas retorts; Gas-producer furnaces for use in heating —. P. Litwin. E.P. 152,369, 21.1.18.

A GAS-PRODUCER furnace using coke is provided with a series of longitudinal water tanks below the stationary grate, each tank being covered by a section of the grate having small nozzle-like openings in the firebars. An injector nozzle is provided in the front of the furnace for each tank, so that a mixture of air and steam may be blown over the surface of the water below the grate-bars and then through the grate into the fuel. The furnace is suitable for using coarse coke and coko ash as it comes from the retort.—W. F. F.

Vertical retorts for carbonising coal. Woodall, Duckham, and Jones, Ltd., and W. T. Gardner. E.P. 152,548, 23.12.19.

TO secure a more uniform withdrawal of volatile products from vertical retorts of oblong cross-section, the mouthpiece is provided with transverse partitions in line with the sides of the hopper to confine the coal on its way from the hopper to the top of the retort. These partitions have openings in their upper parts to provide for the passage of gas from one side of the retort through the openings and above the coal in the mouthpiece to the outlet at the other side of the oblong section.

—W. F. F.

Gas producer. O. E. Yeo. E.P. 151,445, 21.8.19.

FUEL is supplied to the top of a producer which is conical at its upper end and cylindrical at its lower end. Air is blown in at the bottom and producer gas is drawn off through openings, arranged around the circumference about midway of the height, into an annular chamber surrounding the upper half of the producer and thence to the gas main. Another gas outlet is provided at the top of the conical part of the producer. A central refractory pipe, open at the top, projects upwards through the bottom of the producer to a level above the circumferential gas outlets, and steam may be supplied to the bottom of this pipe to generate water-gas. This water-gas may be drawn off from the top of the producer simultaneously with producer gas from the circumferential outlets. If the latter are closed the producer gas mixes with the water-gas. If the retort is charged with coal and the producer gas is burnt in the annular chamber, coal gas will be generated and mix with the water-gas. The producer may also be worked with downward draught.

—W. F. F.

Suction gas plants. F. D. Dilley. E.P. 152,380, 15.5.19.

FUEL is admitted into the top of a vertical producer,

and air for primary combustion is admitted horizontally at the side near the top. A gas outlet for heavy hydrocarbons is arranged immediately opposite to the air inlet, and the gas is led downwards through an external pipe to the bottom of the producer. Steam and air are also admitted at the bottom of the producer in controlled amounts for secondary combustion, and a ring of gas outlets is provided about midway of the height of the producer. The gas is led through a jacketed pipe to a washer, and steam or hot water from the jacket is led to the base of the producer. The apparatus is suitable for use with bituminous material, coconut shells, rubber seed husks, and sawdust.—W. F. F.

Mineral oils which have been treated with [sulphuric] acid; Neutralising —. G. Grisard. E.P. 136,169, 4.12.19. Conv., 4.12.18.

The oil is agitated at 50°–70° C. with anhydrous sodium or potassium carbonate and calcium chloride.—L. A. C.

Lubricant from naphthene acids; Manufacture of a —. C. Francke. G.P. 324,227, 5.10.17.

NAPHTHENE acids are purified by means of a mixture of fullers' earth and sulphur dioxide, and then separated by filtration. The filtrate is heated to 200° C., and again heated after addition of nickel formate, and subjected to the action of hydrogen until the temperature reaches 300° C.—W. J. W.

Peat; Manufacture of fuel from —. N. de Long. E.P. 152,515, 11.10.19.

SEE U.S.P. 1,312,521 of 1919; J., 1919, 709 A.

Gas producers; Feeding mechanism for —. The Wellman-Seaver-Morgan Co., Assees. of J. F. Rogers and J. S. McClimon. E.P. 137,299, 29.12.19. Conv., 30.12.18.

Purifying hydrocarbons. G.P. 325,230. See III.

Hydrogen sulphide and ammonia from gases. G.P. 325,652. See VII.

Building materials. G.P. 324,637. See IX.

Ore-flotation agent. U.S.P. 1,337,542. See X.

IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Gas; Disappearance of — in the electric discharge. Research Staff of the General Electric Co., Ltd., London (N. R. Campbell and J. W. H. Ryde). Phil. Mag., 1920, 40, 585–611.

THE disappearance of gas in vessels under the influence of an electric discharge is closely connected with the appearance of a glow in the vessel. This glow appears sharply at a definite potential difference between the electrodes. The glow potential is of great importance for interpreting the rate of disappearance of the gas. The experimental results are explained by supposing that the glow causes a reversible chemical change to occur in the gas. (Cf. J.C.S., Dec.)—J. F. S.

Photometer. Eder. See XXI.

PATENTS.

Retorts [for distillation of solid materials]. J. E. Kennedy. E.P. 141,028, 20.2.20. Conv., 31.3.19.

A HORIZONTAL rotating retort tapers from the outlet towards the inlet end and carries trunnion members at each end which rotate against stationary feed and discharge conduits of the same diameter. The retort is built up of a number of sections having in-turned flanges which are screwed together, and internal longitudinal ribs are provided to transmit the heat to the material. Raw material is fed into

the smaller end of the retort from a hopper by means of a reciprocating plunger, and the solid residue is removed at the other end by a number of radial blades which lift it on to a guide cone, having its apex pointing towards the discharge trunnion. The residue passes into the fixed discharge conduit and thence to a water seal. A perforated pipe projects longitudinally into the inlet end of the retort to withdraw gaseous products, and steam may be admitted at the discharge end of the retort.

—W. F. F.

Destructive distillation of wood in suction gas plants; Process for the extraction and recovery of by-products formed by the —. J. C. Roberts. E.P. 152,420, 14.7.19.

To obtain acetic acid, methyl alcohol, and acetone when wood is distilled in suction gas plant, the gas is washed in a scrubber through which the same water is passed time after time until it contains sufficient products to be worth recovering. The volatile products are drawn off under partial vacuum and the acid is neutralised with chalk or barium carbonate after each passage of the wash liquor through the scrubber.—W. F. F.

Charcoal; Method for increasing the absorptive capacity of —. Farbenfabr. vorm. F. Bayer und Co. G.P. 307,761, 19.6.17.

By heating charcoal prepared by the process described in G.P. 290,656 (F.P. 471,295; J., 1915, 216) to 400° C. its absorptive capacity is increased and is retained, e.g., in absorption of vapours from air, when the charcoal has regained its original moisture content.—W. J. W.

Tungsten incandescence lamps [; Reducing dissipation of the filament in —]. Deutsche Gasglühlicht A.-G. E.P. 139,161, 4.2.20. Conv., 17.2.19.

DISSIPATION of the metallic filament is reduced, with consequent diminution of the blackening effect upon the bulb, by applying to the finished filament or its support a thin layer of a borate, particularly potassium borate, preferably mixed with a little amorphous phosphorus, the layer being vaporised by passing an electric current through the filament after sealing off and evacuating the bulb.

—J. S. G. T.

Electric lamps; Manufacture of incandescence —. F. Harrison. E.P. 152,379, 13.5.19.

SEE U.S.P. 1,353,654 of 1920; J., 1920, 743 A.

III.—TAR AND TAR PRODUCTS.

Nitro-derivatives of toluene; Synthesis of some —. O. L. Brady and P. N. Williams. Chem. Soc. Trans., 1920, 117, 1137–1140.

3,4-DINITROTOLUENE was prepared from o-toluidine by nitrating to 4-nitro-o-toluidine by Noelling's method (Ber., 1884, 17, 265), acetylating, and further nitrating with fuming nitric acid and sulphuric acid to a mixture of 3,4- and 4,5-dinitro-aceto-o-toluidines, which gave the corresponding dinitrotoluidines on hydrolysis. Up to this stage the yields were good, but on removing the amino-group with nitrous acid only 2 g. of pure 3,4-dinitrotoluene was obtained from 9 g. of the mixed dinitrotoluidines. Although separation of the two isomerides is not necessary for the preparation of 3,4-dinitrotoluene, they are readily isolated by fractional precipitation with water from the solution in 50% sulphuric acid resulting from the hydrolysis of the acetyl derivatives, and if individually treated with Caro's acid, and finally nitrated with fuming nitric acid give good yields of 2,4,5- and 2,3,4-trinitrotoluenes respectively.—G. F. M.

Nitro compounds; Analysis of aromatic — by means of titanium trichloride. F. L. English. *J. Ind. Eng. Chem.*, 1920, 12, 994—997.

MONONITRO-HYDROCARBONS are very resistant towards reduction by titanium trichloride, but the presence of positive or negative substituents, with the exception of chlorine, in the nucleus facilitates the reduction of the nitro groups. The position of the substituents with regard to the nitro group has no appreciable effect; *m*- and *p*-nitroaniline are reduced with equal ease, as are *o*- and *p*-nitrophenols, *o*- and *m*-nitro-*p*-toluidines, and 5-nitro- and 3-nitro-salicylic acid, whilst *o*- and *p*-nitrochlorobenzenes are about equally refractory. (Cf., Callan, Henderson, and Strafford, *J.*, 1920, 86 r.)—W. P. S.

Nitrohydrocarbons; Action of aluminium chloride on solutions of — in aromatic hydrocarbons. A. Kliegl and H. Huber. *Ber.*, 1920, 53, 1646—1655.

THE action of anhydrous aluminium chloride on a boiling mixture of toluene and nitrobenzene leads to the formation of much resinous matter and small amounts of *p*-aminophenyl-*p*-tolyl, m.p. 99° C., b.p. 190° C. at 18 mm., and phenyl-*p*-toluidine, m.p. 88°—89° C. (Cf. *J.C.S.*, Dec.)—H. W.

PATENTS.

Benzol or the like; Purification of —. South Metropolitan Gas Co., E. V. Evans, and H. Hollings. E.P. 152,470, 16.8.19.

THTOPHENS are removed from benzol by treating the benzol with 60—85% sulphuric acid at 30°—90° C., or by passing benzol vapour through 73—82% sulphuric acid.—L. A. C.

Hydrocarbons; Continuous purification and neutralisation of liquid —. H. Heinemann and A. Hellmann. G.P. 325,230, 27.9.17.

THE hydrocarbon, e.g., benzol, is passed continuously through vessels containing successively sulphuric acid and sodium hydroxide solution, and the purified hydrocarbon is withdrawn through jackets surrounding the vessels in order to allow of regulation of the heat of reaction.—L. A. C.

Acid sludge produced in the purification of benzol; Process and apparatus for dealing with the —, and for recovering benzol and its homologues therefrom. G. Stephenson. E.P. 152,054, 2.5.19.

THE acid sludge is heated in a wide, shallow pan by steam introduced through a perforated lead pipe at the bottom of the pan. The benzene vapour as it passes into an outlet pipe at the top of the vessel is scrubbed by a spray of ammonia gas or of a solution of ammonium carbonate, whereby benzenesulphonic acid is converted into benzene and ammonium sulphate. The vapour then passes through a scrubber containing dilute sulphuric acid to remove any excess of ammonia, and thence to a condenser.—L. A. C.

Thionaphthene; Process of separating — from coal-tar [naphthalene]. Ges. für Teerverwertung m.b.H. G.P. 325,712, 8.7.19.

THE sulphonic acid produced as by-product in the technical purification of naphthalene, i.e., by treatment of the crude naphthalene with sufficient sulphuric acid to sulphonate only a fraction of the material, is hydrolysed by steam distillation at 145° C. The product contains about 12% of thionaphthene, and by further partial sulphonation and subsequent hydrolysis an oily product, containing about 36% of thionaphthene, is obtained, which on treatment with the calculated amount of mercury acetate in boiling methyl alcohol yields a crystalline double compound of thionaphthene and mercury acetate. The crystalline product is separated from

the solution, washed, and decomposed to yield pure thionaphthene. (Cf. *J.*, 1920, 716 a.)—L. A. C.

Soap. E.P. 131,881. See X11.

Lacquer paints. G.P. 324,722. See X111.

Resin from wood tar. G.P. 324,876. See X111.

IV.—COLOURING MATTERS AND DYES.

Azopyrazolones; Some new — and allied compounds. K. H. Saunders. *Chem. Soc. Trans.*, 1920, 117, 1261—1272.

A COMPLETE series of compounds has been prepared in which the anthraquinone nucleus is introduced either into the 1-position of the pyrazolone ring, or as the first component of an azo-pyrazolone compound, or in both positions together. 1-β-Anthraquinonyl-3-methyl-5-pyrazolone was obtained by condensing ethyl acetoacetate with β-anthraquinonylhydrazine, dehydrating the resulting hydrazone by boiling with acetic anhydride, and finally removing the acetyl group by hydrolysis with hydrochloric acid. It is a highly insoluble orange powder which couples with diazonium salts to give stable arylazopyrazolones, the benzene-azo-compound being yellow, and the α- and β-naphthalene-azo-compounds vermilion and red respectively. 4-β-Anthraquinone-azo-anthraquinonylmethylpyrazolone forms yellow needles from nitrobenzene; it is practically insoluble in all other solvents. All the anthraquinone-azo-pyrazolones, whether unsubstituted in the 1-position or substituted by phenyl, naphthyl, or anthraquinonyl residues as in the above-mentioned compound, are similarly sparingly soluble. They dissolve, however, in warm alkali hydroxide to deep purple solutions. The 1-phenyl- and 1-β-naphthyl-compounds can exist in a yellow unstable, and a red stable modification, both of which give the same absorption spectrum. On the other hand the 1-anthraquinonyl- and the 1-unsubstituted compounds exist only in the yellow forms.—G. F. M.

Catechin; Constitution of —. II. M. Nierenstein. *Chem. Soc. Trans.*, 1920, 117, 1151—1156. (Cf. *J.*, 1920, 685 a.)

3,4,2',4',6'-PENTAMETHOXY-α-α-DIPHENYLPROPANE, which was previously found to be identical with the methylated reduction product of Kostanecki and Lampe (*loc. cit.*), has been synthesised from 3,4,2',4',6'-pentamethoxydiphenylacetyl chloride by converting this, by treatment with diazomethane, into the corresponding pentamethoxydiphenylmethyl chloromethyl ketone, and reducing this latter substance with sodium and alcohol.—G. F. M.

Pyrrole Blacks. VIII. A. Angeli and C. Lutri. *Atti R. Accad. Lincei*, 1920, 29, i., 420—423.

THE black substance formed from pyrrole and quinone is obtained also when these compounds react in acetic acid solution and hence probably represents an individual compound and not a mixture (cf. *J.*, 1920, 329 a). Pyrrole and *p*-nitrosophenol react in cold acetic acid solution giving an amorphous, deep violet-black powder of the formula C₂₂H₁₂O₃N₂ or C₂₂H₁₂O₃N₂ (cf. Angeli and Cusmano, *J.*, 1917, 923). Intensely coloured products are formed also by the action of pyrrole on *p*-quinone-imino and *p*-quinonedimine.—T. H. P.

Carbocyanines. Mills and Pope. See XX1.

PATENTS.

Azo dyestuffs; Manufacture of —. P. A. Newton. From Farbenfabr. vorm. F. Bayer und Co. E.P. 3557, 11.2.14.

YELLOW azo dyestuffs, which can be rendered very

fast to washing by after-treatment on the fibre with formaldehyde, are obtained by coupling with aminopyrazolones the tetrazo compounds prepared from the aminobenzoylamino compounds described in F.P. 426,201 and E.P. 18,205 of 1911 (J., 1911, 948; 1912, 678), and the monodiazoo compounds of the sulphonic acids of aminobenzoylaminothiozole compounds (G.P. 163,040; J., 1906, 216). The dyes can be developed on fibre and yield pure whites when discharged with hydrosulphite.

Disazo dyestuffs able to be chromated. E. Reber, Assrs. to Soc. of Chem. Ind. in Basle. U.S.P. 1,338,397, 27.4.20. Appl., 24.8.18.

Disazo dyestuffs are prepared from aromatic diazoazo compounds and hydroxycarboxyarylpiazolones. They are orange to brown powders dissolving in water to form orange to brownish-red solutions and in concentrated sulphuric acid to red to blue solutions. They dye wool in an acid bath clear red to orange and brownish-red tints, which when chromed become fast to fulling and to potting.

Disazo dyestuffs dyeing mordanted fibres. C. Jagerspacher and M. Isler. Assrs. to Soc. of Chem. Ind. in Basle. U.S.P. 1,338,506, 27.4.20. Appl., 17.8.18.

SEE E.P. 141,401 of 1918; J., 1920, 443 A.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Hardwoods; Proximate analysis of —. Analysis of *Quercus agrifolia*. W. H. Dore. J. Ind. Eng. Chem., 1920, 12, 984—987.

SLIGHT modifications of methods used previously for coniferous woods (J., 1920, 400 A, 513 A) were applied to the analysis of hardwoods. The purification treatment employed for coniferous woods (successive extraction with benzene and alcohol) was supplemented by digestion with cold water and with cold 5% sodium hydroxide solution; these treatments removed all adventitious matter without injuring the cellulose or lignin. The latter was determined by König and Becker's hydrogen chloride method (J., 1919, 530 A). Oak (*Q. agrifolia*) wood yielded the following results:—Loss on drying, 4.20; benzene extract, 0.50; alcohol extract, 4.33; soluble in cold water, 3.66; soluble in cold 5% NaOH solution, 18.71; cellulose, 45.48; lignin, 20.25; pentosans not otherwise accounted for, 1.89; mannan (residual), none; galactan (residual), 1.49; total, 100.51%.—W. P. S.

Lignin and reactions of lignin. P. Klason. Ber., 1920, 53, 1862—1863. (Cf. J., 1919, 570 A; 1920, 482 A.)

YELLOW cyclic naphthylamine salts, similar to those obtained from pine, have been prepared from fir, beech, birch, oak, linden, alder, and aspen. The corresponding salts obtained from herbaceous plants have a different composition.—H. W.

Pine lignin; Constitution of —. P. Klason. Ber., 1920, 53, 1864—1873. (Cf. *supra*.)

THE lignin which contains an acrolein group and, in the form of its sulphonic acid, yields cyclic arylammonium salts and a calcium salt precipitable by calcium chloride, is termed acrolein-lignin or α -lignin, whilst the second variety, which does not exhibit this behaviour but which contains a free carboxy group, is named carboxylignin, or β -lignin. The two varieties can be quantitatively separated by β -naphthylamine hydrochloride; a complete proximate analysis of pine wood can therefore now be effected giving carbohydrates (68%), lignin (30%), and other substances (2%), 63% of the lignin being present as the α variety and 37% as the β form.

The formulæ, $C_{22}H_{22}O_8$, and $C_{13}H_{10}O_8$, are advanced for α -lignin and β -lignin respectively, and reasons are given for considering the former to be a derivative of flavone. (Cf. J.C.S., Dec.)—H. W.

Straw lignin. F. Paschke. Wochenbl. Papierfabr., 1920, 51, 1139—1141, 2322—2323. Chem. Zentr., 1920, 91, IV., 53, 485.

TREATMENT of straw with sodium carbonate produces good yields of straw pulp if attention is paid to control of temperature and concentration. About 50% is obtainable from rye straw. From the alkaline extract a granular, reddish-brown powder, which decomposes at 195° C., and has the formula $C_{22}H_{21}O_8$, was isolated. It has a higher content of oxygen than wood-lignin. It forms a colloidal solution with water and is precipitated by solutions of barium chloride, calcium chloride, and other metallic salts. Waste liquors from the treatment of straw with sodium carbonate contain no combined ammonia, as is the case when sodium hydroxide is used, which indicates that the protein of the straw is not decomposed.—W. J. W.

PATENTS.

Vegetable fibres; Process for obtaining — for textile purposes. Deutsche Faserstoff-Ges. Fulda m.b.H. G.P. 324,333, 11.1.19.

FIBROUS parts of plants are heated with an alkaline solution of sodium silicate to which an electrolyte is added. The gelatinous silicic acid formed effects separation of the fibres from incrustants.—W. J. W.

Nettle fibres; Treatment of — for textile purposes. Nessel-Anbau-Ges.m.b.H. G.P. 324,519, 10.9.18.

NETTLE fibres may be made crisp and stronger by treatment with strong alkali liquors or other alkaline solutions.—W. J. W.

Fibres; Production of — from plants. Nessel-Anbau-Ges.m.b.H. G.P. 324,520, 15.9.18.

PLANTS such as nettle, sedge, broom, ramie, reha, etc., are treated with a neutral alkali aluminate, which has no deleterious action on the fibres.—W. J. W.

Plants; Process for separating fibres from —. Nessel-Anbau-Ges.m.b.H., and A. Hoermann. G.P. (A) 325,887, 10.4.18 and (B) 325,888, 15.9.18.

(A) ALBUMINOUS and starchy constituents are removed from the fibres by the action of diastatic enzymes before the material is boiled. (B) The appearance and pliability of the fibre are improved by the addition of glycerin or a lactic acid ester to the water or alkaline solution in which the material is boiled.—L. A. C.

Artificial silk solutions; Process for precipitation and purification of —. A. Thilmany. G.P. 324,334, 14.9.19.

ACIDIFIED water or a weak salt solution is sprayed into a bath of the usual precipitating solutions through nozzles, which are fixed between two electrodes. The precipitated, or superficially coagulated fibres are thus hardened and purified.—W. J. W.

Viscose; Bath for spinning threads from —. J. C. Hartogs. G.P. 324,433, 1.3.14.

THE bath consists of a dilute solution of sulphuric acid containing not less than 7% H_2SO_4 , to which two or more soluble sulphates are added in such quantities that not less than 5 equivs. of SO_4 ions are present for every 3 equivs. of H ions. A suitable solution for viscose which has been kept for 7—8

days consists of Na_2SO_4 , 16 parts; $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 30 parts; H_2SO_4 , 9 parts; and water, 45 parts.

—W. J. W.

Hemicellulose; Manufacture of — from wood by the soda process. T. and E. Kuttelsen. G.P. 324,053, 4.3.19. Conv., 20.1.19.

Wood is reduced to small fragments and boiled with clarified "black liquor," after which it is boiled under pressure with a mixture of fresh liquor and "black liquor." It is then washed, ground, and subjected to further treatment.—W. J. W.

Cellulose; Process of obtaining — in two stages. C. S. Fuchs. G.P. 324,894, 2.5.18.

VEGETABLE matter, especially straw, is boiled with alkaline lyes, at the ordinary or increased pressure, until a solution rich in lignic acid is obtained, and the boiling process is then completed with fresh lye. The lignic acid precipitated from the first solution can be used as a resin substitute etc.—C. A. M.

Cellulose esters; Preparation of lacquers, and solutions or masses of —. Chem. Fabr. von Heyden A.-G. G.P. 324,786, 29.8.17.

ESTERS of acyl-hydroxy-fatty acids are used in the preparation of lacquers etc. The ethyl ester of acetylhydroxyacetic acid yields flexible films with cellulose acetate, and nitrocellulose forms very elastic sheets on treatment with the amyl ester of acetylglucolic acid.—C. A. M.

Cellulose waste lyes; Decolorisation of —. A. Wipfler. G.P. 324,787, 1.4.19.

CELLULOSE waste lyes are treated with metals or their compounds (stannous chloride, metallic chromium and hydrochloric acid, chromic acid with aluminium bisulphate) capable of forming insoluble colour-lakes with the colouring matter.

—C. A. M.

Paper; Manufacture of dense strong —. C. G. Schwalbe. G.P. 323,745, 21.2.18. Addn. to 303,498.

PAPER stock, or cellulose waste, is treated with acid, after which it is subjected to mechanical treatment in edge runner mills or disintegrators, and the gummy material so obtained is added to paper pulp.

—W. J. W.

Paper; Manufacture of waterproof —. H. Wandrowsky. G.P. 323,816, 13.6.19.

CELLULOSE esters, insoluble in water, are added to paper pulp, and the paper, after drying, is treated with a soluble solvent for gelatinising the cellulose esters. In the case of paper textiles the solvent is not added till after the weaving process.—W. J. W.

Sizing paper; Material for — obtained from the waste liquors in the Mitscherlich cellulose process. A. Michl. G.P. 323,865, 16.1.19.

WASTE liquors from the Mitscherlich process are exposed to air for a short time, and then centrifuged, a concentrated emulsion of the resinous constituents of any required density being thus obtained. The sizing material is fixed by means of alum.—W. J. W.

Drying paper or other material in sheet form; Method of and apparatus for —. O. Minton. E.P. 152,928, 26.3.20.

Acetates. U.S.P. 1,339,040. See VII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Washing or scouring wool; Process and apparatus for —. R. M. Poole and H. F. Davis. E.P. 152,513, 10.10.19.

Wool is scoured with a solution containing, e.g., 2–5 oz. of potassium carbonate, 2–5 oz. of sodium sulphate, 0.1 oz. of potassium chloride, and 0.25 oz. of sodium chloride per gall., and is subsequently washed with water. The solution, after it has been used for scouring, is concentrated, and the residue is dried and calcined. The product is dissolved in water, and the solution, after filtration and addition of fresh potassium carbonate, is employed for scouring more wool. Any fat rising to the surface during scouring or evaporation of the solution is recovered.—L. A. C.

Fulling process. C. H. Boehringer Sohn Chem. Fabr. G.P. 324,575, 10.5.19.

SALTS of bile acids (e.g., cholic and taurocholic acids) are used for fulling, whereby the amount of soap required is reduced, and the fibres are protected from the action of alkalis. Suitable solutions contain sodium carbonate, 3%, and sodium cholate, 0.1%, or ammonium carbonate, 2%, and potassium taurocholate, 0.2%.—W. J. W.

Stk; Method of loading —. Deutsche Gasglühlicht A.-G. (Auerger.). G.P. 324,562, 30.10.17. Addn. to 320,783 (see E.P. 116,102; J., 1920, 17a).

TREATMENT of the fibres with alkaline gases is followed immediately by treatment with phosphates or silicates, after removing excess of alkali by suction or ventilation.—W. J. W.

Broom corn; Treating [dyeing] —. H. J. Broderson and S. W. Parr. U.S.P. 1,337,306, 20.4.20. Appl., 19.7.16.

BROOM corn is treated with a hot aqueous solution containing a substance, such as sodium sulphite or bisulphite, which facilitates the solution of the objectionable red colouring matter. A reagent, such as aluminium or chromium sulphate or a copper, nickel, tin, or uranium salt, is then added to form an insoluble precipitate with the dissolved colouring matter, and a protective colloid, e.g., glue or casein, is also added to prevent too rapid hydrolysis of the precipitating salt. After removing the fibre from the solution and precipitate, it is dried and then dyed.

Textile fabrics; Sizing of —. A. Poulson. E.P. 152,096, 4.7.19.

CHINA clay (3–4 pts. by measure) is added to a solution of 3 pts. of neutral sodium silicate in 3–4 pts. of water, the mixture is agitated, and, after settling, is sifted to remove lumps. The composition may be used direct, or may be dried and ground, and subsequently made into a paste with water as required.—L. A. C.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric and nitric acids; Distillation of mixtures of —. J. Baumann. Chem.-Zeit., 1920, 44, 805.

A MIXTURE of nitric acid and sulphuric acid (sp. gr. 1.84) cannot be completely separated into its components by distillation. Recovery of the nitric acid without loss of sulphuric acid can only be effected by dilution and re-distillation of the mixed acid residue from the first distillation, which is not a

practicable operation. In two experimental distillations of diluted mixed acid prepared from sulphuric acid of sp. gr. 1·84 and nitric acid of sp. gr. 1·40 (64·5% HNO_3) and 1·30 (48% HNO_3), respectively, in the first case 94·8% of the nitric acid was recovered as acid of 92% strength and 5% was retained by the waste sulphuric acid, whilst in the second 88·6% of the nitric acid was recovered as concentrated acid, and 11·3% was left in the residue. Decrease in concentration of the nitric acid therefore necessitates the use of a disproportionately larger amount of sulphuric acid as water-absorbing medium, and also results in an appreciably larger percentage of nitric acid retained by the sulphuric acid. Dilute nitric acid, such as that derived from the ammonia oxidation process, e.g., of 34°–40° B. (sp. gr. 1·30–1·38) should be concentrated to at least 42° B. (sp. gr. 1·44) in a dephlegmating column, and the waste sulphuric acid should be denitrated. Some loss of nitric acid may arise from the action of reducing substances, such as arsenic, selenium, and organic matter, in the sulphuric acid.—W. J. W.

Distillation of a ternary mixture [e.g., sulphuric acid, nitric acid, and water], one constituent of which is not volatile. P. Pascal. Bull. Soc. Chim., 1920, 27, 814–820.

A MATHEMATICAL discussion of work already published (J., 1917, 1232). The results are applied to the concentration, by distillation with sulphuric acid, of nitric acids of different strength.—W. G.

Atmospheric nitrogen; Fixation of — as cyanide. T. Thorssell. Z. angew. Chem., 1920, 33, 239–240, 245–247, 251–254.

In the fixation of nitrogen as cyanide by its interaction with a mixture of carbon and carbonate of barium or sodium, intimate admixture of the materials is essential, and the addition of manganese, nickel, or iron has been recommended; the use of the last-named metal, in the case of sodium carbonate, admits of a working temperature of 930° instead of 1000°–1100° C., and thus reduces loss of cyanide. The effect of impurities in the carbon, such as silica, alumina, magnesia, or lime, is deleterious and cumulative. Increase of temperature and of pressure correspondingly accelerate the reaction. A preponderance of cyanide over carbonate has a retarding action, but this is not realised in a continuous process, as the partial pressure of the nitrogen is greatest at that part where most cyanide is present. The most important condition governing the process is the relative proportion of nitrogen and carbon monoxide, and in order to avoid accumulation of the latter it must be removed by a stream of nitrogen. A cheap

monoxide formed in the cyanide process or by water-gas, but as the limits within which reduction takes place, without separation of free carbon or formation of carbon dioxide which would contaminate the nitrogen, are restricted, a proportion of carbon dioxide must be added (to prevent separation of free carbon which would subsequently be oxidised to carbon dioxide) when water-gas is employed as reducing agent. The reactions which take place in the furnace and the energy consumption are compared in the two cases where barium carbonate and sodium carbonate respectively are used. As regards advantages offered by these two carbonates, the former is less liable to cause overheating, and gives a very stable basic cyanide, $\text{BaO} \cdot \text{Ba}(\text{CN})_2$. On the other hand, the sodium cyanide formed by use of sodium carbonate is more serviceable as the starting-point for the manufacture of many other compounds, such as ferrocyanides, formic acid, oxalic acid, etc. In comparison with other nitrogen-fixation processes, the cyanide process has the advantages of cheapness and reliability. It has the drawback of requiring a slightly higher temperature; in this connexion some experimental investigations appear to indicate that the temperature required is proportional to the melting point, not of the metallic salts as has been stated by some, but of the metals, and that it is inversely proportional to their atomic weights. It may therefore be conjectured that with salts of rubidium and caesium, a working temperature of 600° C. might be practicable.—W. J. W.

Ammonium carbonate; Determination of the partial and total pressures of solutions of — in water, with special reference to ammonia scrubbing and concentration of ammoniacal liquor. E. Terres and H. Weiser. J. Gasbeleucht., 1920, 63, 705–712.

Gas liquor from horizontal retorts contained 20·45% NH_3 , of which 67·82% was present as ammonium carbonate, whilst liquor from vertical retorts contained 16·39% NH_3 , of which 78·83% was present as carbonate. Liquors containing 85–88% of their ammonia as ammonium carbonate are also met with. The partial pressures of water vapour, ammonia, and carbon dioxide in aqueous solutions containing ammonia and carbon dioxide in proportion corresponding to $(\text{NH}_4)_2\text{CO}_3$, were determined by a transpiration method at 20° C., 40° C., 60·8° C., and 80° C., and the values for the respective total pressures deduced from such partial pressures were confirmed at these temperatures by a manometric method, which was likewise applied to determine the total pressure in the solutions at 90° C. and 95° C.

The following results were obtained:—

Concentration of solution. ($\text{NH}_4)_2\text{CO}_3$	Partial pressure (mm. Hg)											
	H_2O				NH_3				CO_2			
	20°	40°	60·8°	80°	20°	40°	60·8°	80°	20°	40°	60·8°	80°
2·82	16·8	55·6	155·0	350·0	1·8	4·8	15·3	38·8	2·7	9·6	53·5	106·0
5·65	16·3	55·0	153·2	348·0	3·6	9·7	27·0	75·0	3·7	17·6	82·2	156·6
8·46	15·6	54·2	153·5	345·0	5·1	14·5	38·9	112·3	4·4	22·0	100·5	200·5
11·28	15·4	53·1	152·2	—	7·1	19·4	51·0	—	4·9	24·0	118·1	—
14·10	14·8	52·0	151·0	—	8·8	24·4	62·8	—	5·2	25·3	129·5	—
16·92	14·2	51·0	150·0	—	10·6	29·5	72·5	—	5·4	26·6	137·2	—
19·74	13·8	50·2	149·0	—	12·3	34·6	84·0	—	5·2	27·4	152·0	—
22·56	13·3	49·3	148·0	—	14·2	39·5	102·0	—	5·5	28·0	163·1	—

method of separating atmospheric nitrogen is by the use of spongy iron as an oxygen-absorbent. To avoid choking caused by pulverisation and to promote activity the iron is impregnated with sodium hydroxide and briquetted. The ferric oxide formed may be subsequently reduced either by the carbon

At higher temperatures and also at 80° C. with the 14·1% solution it was not possible to determine the partial pressures, but the total pressures were 854 mm. for the 14·10% solution at 80° C., 695 mm. and 801 mm. for the 2·82% solution at 90° and at 95° C., 842 mm. and 978 mm. for the 5·65% solu-

tion at 90° and 95° C., and 1076 mm. for the 8.46% solution at 90° C. The application of the results to the operations of gas scrubbing and the concentration of ammoniacal liquor is briefly discussed.

—J. S. G. T.

Ammonium salts; Microchemical test for —. C. van Zijp. Pharm. Weekblad, 1920, 57, 1345—1348.

FORMALDEHYDE combines directly with ammonium salts, yielding salts of hexamethylenetetramine, which can be detected by means of iodine-potassium iodide solution. This solution affords an excellent microchemical reagent for ammonium salts after addition of formalin. (Cf. J.C.S., Dec.)—S. I. L.

Iodic acid; Action of very concentrated solutions of — on ammonia gas. G. Denigès and J. Barlot. Bull. Soc. Chim., 1920, 27, 824—825. (Cf. J., 1920, 595 A.)

If a drop of 50% iodic acid is exposed to an atmosphere containing a trace of ammonia gas, it becomes coated with a thin white film, and if this is then well mixed with the acid, rhombic crystals of ammonium tri-iodate can be observed under the microscope. If a drop of 50% iodic acid and a drop of strong ammonia are placed a few mm. apart on a microscope slide, the successive formation of the neutral iodate, the di-iodate, and the tri-iodate may be observed.—W. G.

Cyanic acid; Microchemical detection of —. R. Fosse. Comptes rend., 1920, 171, 722—723.

CYANIC acid precipitated as silver cyanate may be detected by three tests. (1) The silver cyanate when crystallised from hot water is obtained in a definite and characteristic microscopic form. (2) When silver cyanate, potassium chloride, and cobalt acetate are ground together, blue potassium cobaltcyanate is produced; the colour is destroyed by water and restored by alcohol. The cobaltcyanate may be distinguished from the sulphocobaltcyanate in that the latter gives a colour with amyl alcohol or ferric chloride, while the former does not, and the cobaltcyanate is not destroyed nor decolorised by dilute acids. (3) If silver cyanate is ground with hydroxylamine hydrochloride, and the product moistened with a drop of ferric chloride solution, a violet-blue colour is obtained.—W. G.

Sulphur dioxide; Oxidising properties of —. I. Iron chlorides. W. Wardlaw and F. H. Clews. Chem. Soc. Trans., 1920, 117, 1093—1103.

THE oxidation of ferrous chloride by sulphur dioxide is represented by the equation $4\text{FeCl}_2 + \text{SO}_2 + 4\text{HCl} = 4\text{FeCl}_3 + 2\text{H}_2\text{O} + \text{S}$, and as sulphur is able to reduce ferric chloride to a small extent the reaction is apparently reversible. The degree of oxidation by sulphur dioxide is independent of the initial concentration of iron. The optimum temperature in presence of 33% hydrochloric acid is 95° C., and no oxidation at all occurs at this temperature if the concentration of free hydrogen chloride is less than 165 g. per l. The highest percentage of ferric iron obtained in any of the flask experiments recorded was 8.8. This result was obtained by treating ferrous chloride solution in 33% hydrochloric acid at 115° C. with a 50% mixture of sulphur dioxide and hydrogen chloride under special conditions. Sealed tube experiments gave a maximum yield of 9.5% of ferric iron. Iron solutions containing 10—18.3% of ferric iron in 33% hydrochloric acid at 115° C. showed no evidence of oxidation or reduction by sulphur dioxide under the special conditions mentioned above, but when the ferric iron exceeded 18.3% a slow reduction occurred.—G. F. M.

Sulphur dioxide; Oxidising properties of —. II. Iron phosphates. W. Wardlaw, S. R. Carter, and F. H. Clews. Chem. Soc. Trans., 1920, 117, 1241—1247.

THE reaction between sulphur dioxide and iron phosphates in concentrated phosphoric acid solution is solely one of oxidation: $4\text{Fe}(\text{H}_2\text{PO}_4)_2 + 4\text{H}_2\text{PO}_4 + \text{SO}_2 = 4\text{Fe}(\text{H}_2\text{PO}_4)_3 + 2\text{H}_2\text{O} + \text{S}$. The reaction is probably reversible, similarly to that of sulphur dioxide with ferrous chloride (cf. *supra*), but it is considerably modified by the formation of the complex compound which ferric phosphate forms with phosphoric acid. This may account for the much higher yield of ferric salt from a pure ferrous phosphate solution than from a ferrous chloride solution, and may also explain the non-reducibility of ferric phosphate in phosphoric acid solution by sulphur. The limiting concentration of phosphoric acid below which oxidation by sulphur dioxide does not take place is about 332 g. of "free" acid per l. at 100° C. The authors seek to correlate the oxidising and reducing action of sulphur dioxide in strong and dilute acid media respectively by an explanation on an ionic basis.—G. F. M.

Phosphates or arsenates; Sensitive colour test for —. G. Denigès. Comptes rend., 1920, 171, 802—804.

THE reagent used is a mixture of equal volumes of a 10% solution of ammonium molybdate and concentrated sulphuric acid. From 3 to 10 drops of the reagent are added to the solution to be tested, the mixture is shaken, and one or two drops of a freshly prepared solution of stannous chloride are added. The solution develops a blue colour if phosphates or arsenates are present. Fluorides tend to inhibit the reaction, but this can be overcome by the preliminary addition of boric acid if they are present. The reaction is given in the presence of organic matter.—W. G.

Thermochemical analysis of solutions. [Detecting formation of double salts.] Chauvenet, P. Job, and G. Urbain. Comptes rend., 1920, 171, 855—857.

THE method used is similar to that of Dubrisay (J., 1920, 542 A) and by its use the existence of the following double salts is indicated: $2\text{KCl} \cdot \text{CaH}_2$, $2\text{CuCl}_2 \cdot 3\text{MgCl}_2$, $3\text{CuCl}_2 \cdot 2\text{MgCl}_2$, $2\text{CuCl}_2 \cdot 3\text{CaCl}_2$, $\text{CuCl}_2 \cdot \text{CaCl}_2$, $\text{CuCl}_2 \cdot 2\text{AlCl}_3$, and $2\text{CuCl}_2 \cdot \text{AlCl}_3$.—W. G.

Hydrous oxides. I. Hydrous ferric oxide. II. Hydrous aluminium oxide. H. B. Weiser. J. Phys. Chem., 1920, 42, 277—328, 505—538.

THE existence of a definite basic ferric salt has not been established with certainty, and only one definite crystalline hydrate of ferric oxide has been prepared. It is probable that the different ferric iron colloids are not composed of basic salts of varying composition or of hydrous hydrated oxides, but are hydrous ferric oxides. The difference in properties of colloidal hydrous ferric oxides prepared by different methods and of different ages is due to a difference in the degree of hydration and in the size of the colloidal particles. Certain electrolytes precipitate the Péan de St. Gilles colloid (Comptes rend., 1855, 40, 568, 1243) in a gelatinous form whilst others precipitate it in a granular form. The most gelatinous precipitate is obtained when there is very rapid aggregation of the hydrous particles throughout the entire solution and when the electrolyte present exerts no solvent action; the most granular precipitate results when the agglomeration proceeds slowly and when the electrolyte possesses a solvent action. Hydrochloric acid has two precipitating values for the Péan de St. Gilles colloid, one above the other with a zone of non-precipitation between, in which

the colloid is less stable than the original colloid. The degree of reversibility of the precipitation of hydrous ferric oxide is determined mostly by the specific adsorbability of the precipitating ion, although the physical character of the precipitate has an influence. It is possible to make a transition from brown to yellow hydrous ferric oxide by increasing the size of the particles, e.g., by heating water to which ferric chloride has been added. The yellow colloidal hydrous oxide is not readily dehydrated at 100° C., and probably contains adsorbed ferric salt. A yellow colloidal solution was prepared by the Péan de St. Gilles method by allowing the acetate solution to stand for a few days before diluting and boiling. The slow hydrolysis of the acetate favours the formation of yellow hydrous oxide stabilised by adsorbed ferric salt. When the solution is boiled, a stable yellow colloid is formed instead of the usual brick-red one.

Aluminium oxide forms only one definite hydrate, the trihydrate, precipitated from solutions of the oxide in alkali. The so-called hydrates formed by precipitating with ammonia are hydrous oxides. Any number of hydrous oxides, similar to those of ferric oxide, may be prepared, differing in size of particles and amount of water. The larger and less hydrous the particles, the less soluble are they in acids and alkalis. There is no definite temperature of inversion from soluble oxide to insoluble meta-oxide. The colloidal oxide prepared by prolonged digestion of aluminium acetate at high temperature is insoluble in acids and alkalis, and has no mordanting action; the colloid prepared by peptising gelatinous alumina with aluminium chloride and dialysing in the cold is soluble in acids and alkalis, and possesses a mordanting action. Colloids with intermediate properties may be obtained. Colloidal alumina is a positive colloid, and is stabilised by preferential adsorption of cations. The relative peptising power at 100° C. was found to be in the order: nitric acid > hydrochloric acid > ferric chloride > aluminium chloride > acetic acid. Strong acids with univalent anions and their salts precipitate colloidal alumina only at high concentrations and the reaction is reversible. Acids with multivalent anions and their salts precipitate the colloid at low concentrations and irreversibly. Gelatinous alumina dissolves in sodium or potassium hydroxide with the production of an unstable aluminate, from which colloidal hydrated alumina separates in a granular, difficultly soluble form. Observation of the effect of the nature of the coagulating agent on the physical character of the precipitated oxide confirmed the conclusions reached with ferric oxide.—J. R. P.

Occlusion of hydrogen by palladium; Influence of hydrogen sulphide on the —. II. E. B. Maxted. Chem. Soc. Trans., 1920, 117, 1280—1288.

The inhibitive effect of hydrogen sulphide on the occlusive power of palladium for hydrogen is a linear function of the concentration of hydrogen sulphide, the critical value for complete inhibition being about 8.5 c.c. of hydrogen sulphide per g. of palladium. The primary absorption of hydrogen is followed in all cases by a slow secondary occlusion which gradually increases in velocity and finally slows down again as the limit of total occlusion is approached. This secondary occlusion appears to be due to the slow spontaneous dissociation of the absorbed hydrogen sulphide, a change which has already been shown to take place instantaneously at 100° C., the sulphur being retained by the palladium as a Pd₂S complex, which is itself incapable of occlusion, but leaves the residual palladium with its normal occlusive power for hydrogen. The hydrogen liberated by this dissociation is equal in volume to the hydrogen sulphide dis-

sociated, and must be added to the observed volume of hydrogen absorbed to arrive at the true occlusive power of the preparation. Confirmation of the above theory is found in the fact that the total volume of hydrogen thus found to be contained in samples of palladium previously treated with varying amounts of hydrogen sulphide falls on the known poisoning curve, calculated on the basis of the presence of the Pd₂S complex in amount equivalent to the sulphur contained in the hydrogen sulphide occluded.—G. F. M.

Promoter action in catalysis. Pease and Taylor. See I.

Antimony sulphide. Van Rossem and Dekker. See XIII.

Carbon bisulphide and ammonium carbonate. Gilfillan. See XX.

PATENTS.

Sulphur dioxide in gases from roasting low-grade ores; Process for increasing the concentration of —. H. Petersen. G.P. 310,073, 15.3.18.

DURING the roasting of ores, poor in sulphur, by means of furnace gases, only so much air, which may be previously heated, is introduced into the furnace as is required to attain the desired temperature.—W. J. W.

Sulphuric acid from copper matte; Process for obtaining —. Metallbank und Metallurgische Ges. A.-G. G.P. 310,621, 10.4.18.

FUSED copper matte is passed in a continuous stream into a converter, which is provided with means for collecting the sulphur dioxide evolved and conducting it to a sulphuric acid plant. The oxidised copper matte and slag are removed continuously at the same rate as fresh matte is introduced.—W. J. W.

Nitrogen oxides; Apparatus for the catalytic preparation of — from ammonia and air. S. Barth. G.P. (A) 298,951, 31.7.15, and (B) 310,352, 21.11.15.

(A) THE apparatus consists of a revolving drum with cylindrical sieve-like walls which act as the catalyst, arranged in a closed receptacle fitted with two tubes through which the gas enters and leaves the apparatus. The former tube contains a valve and terminates in a perforated porcelain tube arranged along the axis of the drum above the other tube, which is set in the floor. (B) The catalyst is built up between a fine network of aluminium wires which act as a cooling medium and prevent decomposition of the gases either before or after passing through the catalyst.—A. R. P.

Fixation of nitrogen; Process and apparatus for the electrical — from air and the products resulting therefrom. J. E. Bloom. U.S.P. 1,334,590, 23.3.20. Appl., 21.8.18.

IN the fixation of atmospheric nitrogen by means of the electric arc, an earthing electrode is provided in the path of the gases near the arc and is connected with the earth through an insulated ground line and an insulated "interrupter-selector" adapted to pass to the earth only one kind of charge, electron, or the like. The gases leaving the arc are passed rapidly through successive alternating current fields beyond the arc furnace, then through absorption towers or chambers containing masses of comminuted solid absorption material (corundum, silica, etc.) at progressively reduced temperatures down to 50° C. The gases are finally passed through absorption towers supplied with sprays of water or alkaline solution and provided

with electrodes instead of the usual plates. Alternating current fields are created in the absorption tubes or chambers and in the absorption towers.

—B. N.

Combustion of nitrogen; Process for —. *Gewerkschaft des Steinkohlenbergwerks "Lothringen,"* and M. Kelting. G.P. 324,261, 23.7.19.

IN the combustion of nitrogen in a hydrogen or carbon monoxide flame, the yield of nitrogen compounds is appreciably increased by introducing 1—2% of solid carbon or carbon compounds of high melting point, so as to produce finely-divided incandescent carbon in the flame.—W. J. W.

Alkali percarbonates; Manufacture of —. H. Wade. From Deutsche Gold- und Silber-Scheideanstalt vorm. Roessler. E.P. 152,366, 20.8.17.

CALCINED alkali carbonates are treated with hydrogen peroxide solution to which common salt has been added, preferably in presence of anti-catalysts, such as sodium silicate, magnesium chloride, or magnesium-sodium silicate. A yield of 90% of solid alkali percarbonate is obtainable.—W. J. W.

Magnesium chloride or double chlorides of magnesium; Production of anhydrous —. E. A. Ashcroft. E.P. 152,401, 9.7.19.

MAGNESIUM chloride in the form of needle crystals is subjected to the action of a current of heated air by which it becomes partially dehydrated, and its m.p. is raised to about 250° C., without the salt undergoing decomposition. It is then transferred to another apparatus in which, by the action of a current of hydrogen chloride the remaining water of crystallisation is removed, the temperature being slowly raised to 650° C. during the process. The product contains at least 98% of pure anhydrous magnesium chloride, and the hydrogen chloride is recovered by condensation as a 33% solution. Double salts of magnesium chloride may be similarly treated, with slight modifications of temperature.—W. J. W.

Arsenious acid; Process for refining —. M. Uchino. E.P. 152,526, 27.10.19

ARSENIOUS acid may be refined by adding to it copper oxide, or a mixture of oxides of copper and iron, and then distilling.—W. J. W.

Acetates, etc.; Production of — [from carbohydrates]. J. W. H. Randall, Assr. to Industrial Chemical Co. U.S.P. 1,338,040, 27.4.20. Appl., 27.6.17.

CARBOHYDRATES, such as sugar, starch, cellulose, gum, etc., are dissolved in, or mixed with, caustic alkali solution, the mixture is concentrated, mixed with sufficient quicklime to form a dry, porous mass, and heated to 250°–300° C. with exclusion of air.

Titanium and vanadium compounds; Process for recovering —, or either, from minerals containing them together with iron. B. P. F. Kjellberg. G.P. 324,581, 17.10.19. Conv., 24.2.19.

THE mineral is dissolved in a suitable solvent, the solution evaporated to dryness, and, if titanium is present, the residue is heated without decomposition, treated with water, and the solution heated. The precipitated titanium compound is separated, the solution evaporated, and the residue heated to convert the iron into insoluble oxide. If vanadium is present the product is then dissolved in a suitable solvent to extract the soluble vanadium compounds. Special methods are given for treatment of ores containing only iron and vanadium, or, in addition, very small amounts of titanium.—W. J. W.

Carbon dioxide; Process of recovering — from gases. Deutsche Oxydrie A.-G. G.P. 324,868, 3.6.19.

CARBON dioxide is removed from gases containing it by absorption in water under pressure, from which it is recovered by passing the water into a vacuum chamber. The exhaust water is used first to cool the compressor employed in the first operation, then to wash further quantities of gases before compression.—A. R. P.

Furnace for the production of sulphates from bisulphates. Verein Chem. Fabr. in Mannheim. G.P. 325,314, 18.6.16.

THE iron muffle, in which the bisulphate is heated with a reducing agent or with rock salt, is charged by means of a worm screw so arranged that the charge is preheated and thoroughly mixed.

—L. A. C.

Ammonia and hydrogen sulphide; Removing — from gases or vapours containing less hydrogen sulphide than ammonia. W. Feld, Gasabteilung, G.m.b.H. G.P. 325,652, 9.8.18.

THE gas is washed with a thionate solution which is used continuously without regeneration, but to which a little regenerated solution is added as required. Hydrogen sulphide only is thus removed, and the gas is then passed through sulphuric acid to recover the ammonia as ammonium sulphate.

—L. A. C.

Hydrogen; Process for production of —. C. Toniolo, and Officine Elettrochim. Rossi. E.P. 152,554, 5.1.20. Conv., 29.10.19.

OXIDES of iron, or of alkali or alkaline-earth metals, or of mixtures of these, are reduced by means of gases from smelting furnaces, or from blast furnaces fed with oxygen. The free metals or their lower oxides are then treated with water vapour, by which means the original oxides are reproduced and hydrogen is formed.—W. J. W.

Supersaturated solutions of oxygen; Manufacture of — from per-salts and catalysts. M. Sarason. G.P. 325,155, 21.5.16.

SOLUTIONS of perphosphates, e.g., pyrophosphates, are used, such solutions being considerably more stable than those of perborates.—L. A. C.

Sulphur; Process for separation of — from gases obtained in the roasting and smelting of sulphur-bearing ores. C. G. Collins. E.P. 152,447, 22.7.19.

SEE U.S.P. 1,314,631 of 1919; J., 1919, 861A. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 1974 of 1881 and 6567 of 1892.)

Acid-proof alloys. E.P. 121,730. See X.

Magnesium and chlorine. E.P. 152,402. See X.

Magnesium chloride. E.P. 152,403. See X.

VIII.—GLASS ; CERAMICS.

Glasses; Density of soda-magnesia —. C. J. Peddle. J. Soc. Glass Tech., 1920, 4, 281–283.

IN glasses of the type $x\text{Na}_2\text{O} \cdot y\text{MgO} \cdot 5\text{SiO}_2$, the density falls proportionately as the Na_2O is replaced molecularly by MgO . English and Turner (J., 1920, 627A) have found that in glasses of a similar type but with 6SiO_2 , the same change occurs. As the latter glasses have a lower density for the same content of soda and magnesia, the addition of silica must lower the density. When the silica remains constant, the replacement of 1 mol. Na_2O by 1 mol.

CaO increases the density, whilst the substitution of 1 mol. MgO for 1 mol. Na₂O decreases it, so that 1 mol. Na₂O added to a glass of this type has a greater effect than the addition of 1 mol. MgO, but a smaller effect than 1 mol. CaO.—A. B. S.

Glass; Surface effects on — probably produced by reheating in the glory-hole. M. W. Travers. J. Soc. Glass Tech., 1920, 4, 284—286.

SPECIMENS of microscope slides and photographic plates were scratched on one surface and apparently pitted on the other, the apparent pitting being in the form of minute bubbles, 0.001 mm. diameter, immediately below the surface. The author suggests that the pitting was caused by the evolution of gas when the ball was reheated in the glory-hole before blowing the cylinder from which the slides and plates were made, and that the scratches were formed on the surface which was inside the cylinder by the wooden block used in the flattening process. The "sulphuring" of lead glass may be due to a similar cause.—A. B. S.

Glass; Expansion of — at high temperatures. W. B. Pietenpol. Chem. and Met. Eng., 1920, 23, 876—877.

THE EXPANSION of various kinds of glass was measured from ordinary temperatures up to 750° C. and the results plotted. The rate of expansion of annealed glass is nearly linear up to 450°—550° C.; it then increases by 4—6 times for the next 60°—100° C., after which it again becomes linear until the softening point is reached, when it gradually decreases until the glass loses its form. With unannealed glass the rate of expansion at first decreases due to the relieving of the internal strain, then the glass behaves as annealed glass except that on cooling shrinkage takes place in amount depending on the strain which has been relieved.—A. R. P.

Plastic substances [glass]. Le Chatelier. See X.

PATENTS.

Glass capillary tubes; Process for manufacturing — of a prescribed interior form. K. Küppers. G.P. 306,004, 25.7.16.

A TUBE of approximately the correct size is pulled over a metal wire of the exact size required, then heated sufficiently, but without melting, so that it acquires the correct size. After cooling, the wire is partly dissolved by forcing acids or, in the case of nickel, carbon monoxide between the wire and the glass until the wire is so thin that it can readily be pulled out.—A. R. P.

Drying apparatus [for bricks etc.]. O. Imray. From F. A. Secord, C. H. Peters, and A. P. Paterson. E.P. 152,505, 1.10.19.

THE BRICKS or other plastic material to be dried are placed on hollow supports which are perforated for the passage of drying gases and supported on a truck, or other means of conveyance, in such a manner that any support (above the lowest) may be swung aside to permit access to the bricks on the next lower support without allowing the escape of drying fluid. The drying fluid is preferably supplied through a conduit supplying several supports, to which it is connected by detachable headers, and the supply may be cut off from any support at will.—B. M. V.

Gas fired ring furnaces; Process and apparatus for firing at high temperatures in —. J. Schmelz. G.P. 319,698, 13.4.19.

THE FURNACE consists of two series of parallel chambers joined to each other and heated one after the other in the same direction by a common regenerator, while the preheating chamber is fired with gas and preheated air from the cooling

chamber. In this way a temperature sufficiently high to fire refractory materials, such as magnesite, Dinas brick, etc., is rapidly and economically obtained.—A. R. P.

Bricks; Process for preparing dry-pressed — from clay. Chem. Lab. für Tonind. und Tonind.-Zeit. H. Seger und E. Kramer G.m.b.H. G.P. 324,189, 26.10.15. Addn. to 321,930 (J., 1920, 690 A).

THE BRICKS are made from clay, part of which has been heated above the usual drying temperature to render it non-plastic and the remainder of which is raw clay. The mixture still has a certain plasticity.—A. R. P.

Lithographic stones; Manufacture of artificial —. J. Kiermayer, and Vercin. Chem. Fabr. zu Leopoldshall A.-G. G.P. 324,222, 26.6.19.

ARTIFICIAL lithographic stones are prepared from mixtures of magnesium oxide obtained by heating magnesium chloride to redness, and magnesium chloride solution of 15°—35° B. (sp. gr. 1.116—1.320).—W. J. W.

Plastic masses from fine powders and colloidal solutions; Process for producing —. E. Podszus. G.P. 325,307, 4.2.14.

PRECIPITANTS such as tannic acid, gelatin, or the like, or oppositely charged colloids are added, in quantity nearly sufficient to produce precipitation, to solutions of colloids, such as silicic acid, which are precipitated with difficulty by means of electrolytes. When moulds other than plaster moulds are used for shaping the mixture of colloidal solution and fine powder, they are first impregnated with salts capable of precipitating the colloid. By these means precipitation of the colloid at the surface of the mould is facilitated.—L. A. C.

Kilns. C. H. Zwermann. E.P. 140,740, 29.9.19. Conv., 27.3.19.

SEE U.S.P. 1,308,973 of 1919; J., 1919, 636 A.

Glass; Methods of and apparatus for feeding molten —. Hartford-Fairmont Co., Assees. of K. E. Peiler. E.P. 142,786, 15.1.20. Conv., 5.5.19.

Kilns. E.P. 152,509. See I.

Filtering compound. U.S.P. 1,336,591. See XIXb.

IX.—BUILDING MATERIALS.

Cement; Influence of additions of calcium sulphate or chloride on the shrinkage of —. A. Guttmann. Zement, 1920, 9, 310—313, 429—432. Chem. Zentr., 1920, 91, IV., 527—528.

ADDITION of raw gypsum or calcium chloride to Portland, iron-Portland, or blast-furnace cement causes them to swell so that the natural shrinkage is counteracted or only takes place after a long time. Cements so treated are especially adapted for use as mortars where it is desired that the joint should be nearly invisible.—A. R. P.

Wood preservation. Moll. See XIXb.

PATENTS.

Porous material [from slag]; Manufacture of —. Det Norske Aktieselskab for Elektrokemisk Ind. Norsk Ind.-Hypotekbank. E.P. 136,818, 15.12.19. Conv., 16.12.18.

MATERIAL of uniform porosity suitable for building or insulating purposes is produced by passing water, with or without the addition of steam or other gases, into slag at about 1400° C., stirring until a foam is formed, and drawing off the foam into moulds to cool.—L. A. C.

Cement; Manufacture of — from slag. R. Boveroulle. E.P. 149,988, 9.8.20. Conv., 7.8.19.

In the manufacture of slag cement by the cold process, lime cinder or dross is used instead of fat (or white) lime or hydraulic lime in stone form.

—L. A. C.

Cement; Process for burning — in shaft furnaces. Fellner und Ziegler. G.P. 320,731, 30.11.18.

WATER is sprayed into the discharging hopper of the furnace so as to precipitate dust that would otherwise be blown out, and to quench any incompletely sintered material. In this way a certain amount of steam is produced which, together with air, is drawn into the furnace and exerts a beneficial effect on the calcining operation.—A. R. P.

Light building materials; Utilisation of the residues from burning bituminous shales in the preparation of —. Rekord-Zement-Ind. G.m.b.H. G.P. 324,637, 25.9.17.

THE finely powdered or slimed residues are mixed with a light filling material, and the mixture is moulded and burnt, with or without the addition of clay. Alternatively, the finely powdered material may be mixed with the necessary binding and hardening substances and the moulded articles prepared from this mixture in a known manner.—A. R. P.

Rough sand for industrial uses; Manufacture of —. H. Stoltzenberg. G.P. 325,613, 1.7.19.

SAND for use, e.g., for admixture with clay, lime, cement, tar, or the like, is treated with hydrogen fluoride gas or with salts of hydrofluoric acid, such as ammonium fluoride, which dissociate when vaporized. The treatment is carried out, e.g., in heated drums, and has the effect of pitting the surface of the sand.—L. A. C.

Colouring wood grey or black; Process for —. R. E. Slade. E.P. 152,427, 14.7.19.

WOOD is impregnated separately under a pressure of 60–300 lb. per sq. in. with solutions of an iron salt and tannin and/or logwood extract, and subsequently with a solution of lime water or ammonia. Before treatment and after each impregnation the wood is dried under reduced pressure with or without the aid of heat.—L. A. C.

Wood; Process for artificial seasoning of —. M. Kleinstück. G.P. 323,973, 30.10.14.

WOOD is first dried in a vacuum, and then treated warm with a mixture of oxygen, ozone, or air, and catalysts such as ammonia, methylamine, or dimethylamine, or the products obtained by interaction of ammonia and formaldehyde.—W. J. W.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Blast furnace; Effect of temperature, pressure, and moisture of the air upon the working of the —. A. Wagner. Stahl u. Eisen, 1920, 40, 1397–1403.

THE theoretical effects of temperature, pressure, and moisture of atmospheric air on its weight were calculated according to the mechanical theory of heat, utilising the average highest and lowest values of these variables issued for a period of twelve months from a meteorological station. Graphical representations of the working results from a Rhenish blast furnace plant indicate that for given blowing power, the lowest blast pressure and the greatest production were attained during the month of May, in which the air weight per cb. m. was below the average value. Coke consumption was not affected by the moisture of the air and for practical purposes varied inversely with

the yield of iron from the ore. Further work is necessary to determine whether blast regulation according to the weight of the air or according to pressure exerts the better influence on output.

—J. W. D.

Steel manufacture; New deoxidisers for —. J. R. Cain. Chem. and Met. Eng., 1920, 23, 879–882.

WITH the idea of finding a new alloy of two or three of the metals manganese, silicon, titanium, aluminium, which, when used as a deoxidiser for steel would give a very fusible slag, the melting points in the systems, manganese oxide—silica, manganese oxide—titanium oxide, manganese oxide—silica—alumina, manganese oxide—silica—titanium oxide, and manganese oxide—alumina—titanium oxide were determined. The results indicate that the alloys which may be expected to give the most fusible slags are: 75 Mn, 24 Si, oxides melt at 1228° C.; 41% Mn, 59 Ti, oxides melt at 1160° C.; 49% Mn, 18% Ti, 33 Si, oxides melt at 1130° C.; 13% Al, 52% Mn, 35% Si, oxides melt at 1121° C.; 12% Al, 47% Mn, 41 Ti, oxides melt at 1320° C.—A. R. P.

Steel; Woody fibrous fracture in —. E. H. Schulz and J. Goebel. Stahl u. Eisen, 1920, 44, 1479–1485.

SEVERAL different type fractures of rolled and pressed steel showing woody fibre structure were investigated, and it was found that this phenomenon may be due to widely different causes. The fractures may be divided into two principal groups. The first type is due to separation of material in the unbroken rods, which may be due to piping, inclusion of extraneous metal which has got into the mould without dissolving in the fluid steel, and blowholes, or to slag inclusions and segregation which produce regions of low tenacity. The cause of the second type has not yet been fully explained but is possibly connected with stresses occurring in the material. In some cases the fibrous structure may be removed by annealing at 600° C., in others annealing has no effect.—T. H. Bu.

Graphite and temper carbon obtained from cast iron; Crystalline nature of —. K. Iokibe. Sci. Rep. Tōhoku Imp. Univ., 1920, 9, 275–279.

THE diffraction figures produced by X-rays on compressed cylinders of graphite electrolytically separated from grey cast iron and temper carbon isolated from an annealed white cast iron show that these two forms of carbon are identical with natural graphite. Sugar carbon having no crystalline structure did not show diffraction rings.

—T. H. Bu.

Carbon steels quenched and tempered; State of the carbide in —. S. Saitō. Sci. Rep. Tōhoku Imp. Univ., 1920, 9, 281–287.

SPECIMENS of six carbon steels with carbon varying from 0.5 to 1.95% and of a white cast iron were heated in a vacuum and examined by the magnetometric method. When a quenched steel is tempered cementite separates from solid solution at 288° C. Immediately above this point the greater part readily decomposes into iron and carbon owing to the fineness of its particles. Up to 350° C. re-combination goes on and the magnetisation slightly decreases. Large-grain coagulated cementite corresponding to microstructures ranging from sorbite to pearlite is not decomposed to an appreciable extent below A1. In hypereutectoid steels above the A₁ point some free cementite exists which is partially decomposed into iron and carbon at these high temperatures. Free carbon when dissolved in iron at high temperatures combines to form cementite and during cooling is set free as combined carbon. The atomic configuration

corresponding to the solution of cementite or carbon atoms in austenite is the same. Observations on hypoeutectoid steels are in accordance with the above views.—T. H. Bu.

Steel; Critical heat treatment after critical cold work of a low carbon —. A. Pomp. Stahl u. Eisen, 1920, 40, 1261–1269, 1366–1378, 1403–1415.

A soft steel containing C 0.05%, Mn 0.10%, Si trace, P 0.005%, S 0.035%, Cu 0.024%, and giving the following tests after normalisation at 1000° C., hardness 89, yield point 23.1 kg. per sq. mm., tensile strength 31.2 kg. per sq. mm., elongation 31%, notched bar test 20.3 m.-kg. per sq. cm., was rolled at various temperatures up to the Ar3 temperature, and then annealed at temperatures between 650° and 850° C. Marked grain growth, corresponding to an increase of 22,000–34,000% over the original size, occurred when the reduction in section was between 8 and 16%, being most pronounced at about 11%. At the same time the hardness decreased 9–14%, the yield point 35–47%, the tensile strength 4–11%, and the notched bar test 84–93%, while the elongation increased 9–25%. The reduction in area did not undergo a regular change. Within the limits of critical cold work, 8–16%, divorce of the pearlite occurred, the freed cementite separating in the state of fine threads at the ferrite grain boundaries.—J. W. D.

Steels; The Ar3 point of — and martensite. P. Dejean. Comptes rend., 1920, 171, 791–794.

A REVIEW of the author's previous work (cf. J., 1917, 965) in the light of recent work by Chevenard (cf. Guillet, Rev. gén. Sci., 1920, 31, 1496).—W. G.

Arc-fused steel; Metallography of —. H. S. Rawdon, E. C. Groesbeck, and L. Jordan. Chem. and Met. Eng., 1920, 23, 777–784. (Cf. J., 1920, 752 A.)

ARC-FUSED steel shows characteristic globules of oxide which coalesce to larger globules on annealing; it also shows needles or plates which are probably due to nitrides, as a similar structure is shown by iron which has been nitrogenised. The nitrogen content of the steel after fusion in the arc is considerably increased, whereas most of the other impurities present are burnt out. The nitride plates gradually disappear both from arc-fused and nitrogenised metal on annealing at 1000°–1200° C. *in vacuo*. Thermal analysis of specimens of steel before and after arc fusion showed that the Ar3 and Ac3 points are depressed about 50° C. by the fusion, whereas the Ac2 and Ar2 points are unaffected. Microscopical examination shows that the arc-fused metal is inherently ductile, but that other imperfections mask this quality. The use of a protective coating on the electrodes during welding does not appear to affect the mechanical properties of the weld in any way.—A. R. P.

Plastic substances [steel, glass]; Mechanical properties of —. Importance of reactivity. H. and F. Le Chatelier. Comptes rend., 1920, 171, 695–699.

From the results of torsion tests on glass at its temperature of annealing (540° C.), and on mild steel at its temperature of forging (825° C.), it is shown that at a sufficiently high temperature, and with small forces, plastic substances undergo successively three types of deformation:—(1) An instantaneous elastic deformation which disappears immediately after the suppression of the force; (2) a subpermanent deformation, produced slowly and disappearing slowly after the suppression of the force, and having an order of magnitude comparable with that of the instantaneous deformation; (3) a viscous deformation continuing the subperma-

nent deformation; this is produced with a constant velocity and does not disappear after the suppression of the force. In the case of soft steels Coulomb's modulus of elasticity is probably a linear function of the temperature.—W. G.

Metal [silicon-iron] tubes unattacked by steam at temperatures above 1000° C. P. Askenasy. Z. Elektrochem., 1920, 26, 436.

TUBES made from silicon-iron (as used in the construction of dynamos) are very little attacked by air or mixtures of steam and oxygen at temperatures up to 1100° C. The metal is brittle, but by careful heating may be raised to a dull red heat and can then be bent into a tube which may be autogenously welded. Such tubes when encased by cast iron serve well as superheaters for steam. Chromium-nickel tubes are equally resistant to steam at high temperatures.—J. F. S.

Carbon-manganese alloys; Equilibrium diagram of —. K. Kido. Sci. Rep. Tôhoku Imp. Univ., 1920, 9, 305–310.

THE author has modified and completed the equilibrium diagram of manganese and carbon established by Stadelher (J., 1908, 572). In the solid state manganese carbide (Mn₃C) and carbon are only partially soluble in each other. As the temperature falls the solubility of both decreases and they form a eutectoid containing about 3.7% C. Microscopical examination of specimens quenched at different temperatures indicated the eutectoid temperature to be 930° C. on heating and 830° C. on cooling. This was confirmed by thermal analysis. (Cf. J.C.S., Dec.)—T. H. Bu.

Zinc dust; Influence of foreign gases on the formation of — during the condensation of zinc vapours. O. Ravner. Tekn. Ukeblad, 1920, 67, 404–408, 418–420. Chem.-Zeit., 1920, 44, Rep., 256.

IN order to minimise the deposition of zinc dust during the condensation of zinc vapours the following points should be observed. The temperature of the receiver should be a little above the melting point of zinc and the distance between the retort and the receiver should be as small as possible, as the longer the time that elapses before the vapours condense the greater is the amount of carbon dioxide formed by decomposition of the carbon monoxide present. This leads to the formation of films of zinc oxide on the droplets of condensed zinc, and prevents them running together. Similar effects are obtained if nitride is formed, either from nitrogen in the coke or from leakage of air into the retort; or if the ore or fuel contains sulphur, zinc sulphide being slightly volatile. Traces of moisture in the charge lead to the formation of dust in the pipes, and, in general, the more the zinc vapours are diluted with foreign gases the greater is the dust deposit. Chemical and screen analyses are given of typical coarse and fine zinc dust. In order to obtain a metallic regulus from the dust the best process is to melt it with a mixture of equal parts of zinc and calcium chlorides; in one case a yield of 91% of the total metal present was obtained in this way.—A. R. P.

[Copper-aluminium] alloy; Experiments in manufacturing No. 12 —. R. J. Anderson. Chem. and Met. Eng., 1920, 23, 883–887.

FROM a series of experiments in which No. 12 alloy (Cu 8%, Al 92%) was made by mixing the elements in various manners and observing the dross losses, gas consumption, and microstructure of the resulting castings, the following conclusions were arrived at:—When small quantities of the alloy are being prepared it is sufficient to add light copper sheet to molten aluminium, but for large-scale work it is preferable to add the copper as a rich alloy with

aluminium. The most convenient alloy to employ is that containing equal parts of the two elements, and it should be charged into the furnace at the same time as the aluminium.—A. R. P.

Electrode processes. Influence of rise of temperature and of depolarisers on the form in which nickel separates. H. Stäger. *Helv. Chim. Acta*, 1920, 3, 581—613.

RESULTS are given of investigations on the formation or alteration of deposits of nickel under conditions effecting the depression of the polarisation, and on the behaviour of these deposits when the development of the film of hydrogen, regarded as the true cause of the highly disperse metallic deposits, is suppressed by means of cathodic polarisers. (Cf. J.C.S., Dec.)—T. H. P.

Flotation; Effect of addition agents in —. Part II. M. H. Thornberry and H. T. Maun. *Bull. School of Mines and Met., Univ. Missouri*, 1919, 5, 1—68. (Cf. J., 1918, 93 A, 153 A.)

A CONTINUATION of previous work on the effect of the addition of various salts during the flotation of a lead slime. This part deals with the effect of chlorides, acetates, oxalates, chromates, carbonates, permanganates, tartrates, phosphates, and a few other salts. Tables are given showing the extraction and grade of concentrate obtained with varying quantities of each addition agent. Chromates had a pronounced detrimental effect on both the extraction and grade of concentrate.—A. R. P.

PATENTS.

Casting [steel] ingots. A. V. Carlsson. E.P. 135,207, 13.11.19. Conv., 14.11.18.

THE runner opening or "dead-head" of the mould is rammed with a material, e.g., charcoal breeze, which will burn or evolve heat under the influence of the cast metal. The heat thus liberated is transferred to the upper part of the ingot and prevents or reduces the formation of pipes.

Iron and steel; Extraction of — from ore, and the formation of alloys thereof. T. Rouse. E.P. 152,073, 2.7.19.

FINELY divided ore is mixed or agglomerated with the requisite amount of a ferro-boro-silicate (a concentrated silicate solution containing 3—4% Fe₂O₃ and 5—8% of calcined borax) and reduced to metal in a plant comprising a Siemens furnace provided with electrodes of high conductivity of an alternating three-phase system, so arranged and operated that the initial or heating stages of the process are effected by the gas furnace and the subsequent treatment by the heating action of the three-phase current. Alloying metals may be added at a suitable stage of the process.—J. W. D.

Steel; Manufacture of —. British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 152,371, 5.6.18.

STEELS containing either niobium 6—12%, or niobium 3—6% and tungsten 6—10%, or niobium 2—4%, tantalum 3—6%, and tungsten 4—8% are claimed. Niobium has a greater hardening and toughening effect than other alloying metals, and also has a marked purifying action.—J. W. D.

Chrome iron or chrome steel alloys; Production of —. W. B. Ballantine. E.P. 152,399, 9.7 and 16.8.19.

CHROMIUM for the production of chrome-iron or chrome steel alloys is prepared from a thermo-reaction mixture comprising chromic oxide or a mineral containing it, calcium carbide, and a basic oxide, e.g., calcium or barium oxide, with or without one or more other thermo-reducing agents, such as calcium silicide or aluminium.—J. W. D.

Steel or other magnetic materials; Apparatus for detecting the temperature of demagnetisation of —. C. W. Stopford and C. R. Darling. E.P. 152,501, 23.9.19.

A SOFT-IRON rod pivoted inside a coil of wire moves under the increase of magnetic flux due to the steel under treatment and thereby breaks the circuit of a bell or other indicating device. When the temperature of demagnetisation of the steel is reached the soft-iron rod returns to its original position, the circuit is restored, and an indication is given.

Silicon-iron alloys; Prevention of graphite precipitation in the manufacture of acid-resistant —. Maschinenfabrik Esslingen. G.P. 324,267, 4.9.18. Addn. to 306,001 (J., 1918, 628 A).

FERROSILICON is melted with low-carbon iron in the same apparatus, under such conditions as to exclude introduction of further quantities of carbon. —W. J. W.

Steel castings; Process for manufacturing —. H. Hanemann. G.P. 325,571, 23.12.19.

AIR-HARDENING steel is used in the manufacture of the castings, which are then treated in a known manner. Steels with a high tungsten, nickel, or manganese content and a corresponding carbon content have on air-cooling an austenitic and martensitic structure. If such steels are heated a great improvement of the structure takes place, due to the formation of sorbite and granular pearlite, so that the effect of the casting disappears. The whole of the cementite in the steel can be made to agglomerate into nodules by a special heat treatment, e.g., annealing at 500°—700° C. after cooling, reheating the steel and allowing it to cool in the air, followed, if necessary, by a further annealing. —A. R. P.

Electric arc furnaces [for treatment of steel]. A. P. Heyen. E.P. 152,176, 30.8.19.

AN electric arc furnace for the treatment of steel is provided with hollow electrodes enclosing burner tubes through which is introduced into the space above the molten charge a mixture of air and heavy hydrocarbons, whereby the length of the arc is increased.—J. S. G. T.

Electric [metallurgical] furnaces. Electro-Metals, Ltd., and T. D. Robertson. E.P. 152,145, 18.7.19.

IN electric furnaces of the type wherein the current passes from one or more electrodes above the charge, through the latter and by way of a conducting hearth to electrodes embedded therein, escape of molten metal through interstices in the hearth is prevented by surrounding the latter with a non-conducting envelope extending above the highest level of the molten charge. This envelope is contained within a metal casing through which pass one or more metallic conductors extending from below the hearth to a level within the envelope above that of the charge.—J. S. G. T.

Acid-proof metal alloy; Production of an —. C. Rossi. E.P. 121,730, 13.12.18.

AN alloy containing 13—15% Si for ensuring resistance to chlorine and to nitric, sulphuric, and acetic acids, or 20—21% Si for ensuring resistance to hydrochloric acid, is prepared by melting iron and silicon together in an electric furnace and removing all impurities by crystallisation by cooling to not below 1400° C. The strength of the alloy is improved by addition of small proportions of fluxes, such as iron sulphide.—J. W. D.

Alloy. B. A. Bangertter. E.P. 152,277, 12.4.20.

AN alloy of 3½ pts. of fine silver, 3½ pts. of standard silver, 1 pt. of 12-carat gold.—J. W. D.

Coating articles with metal. G. de Dudzele et Cie. E.P. 139,143, 5.8.19. Conv., 18.2.19.

THE articles (e.g. of iron or steel) are treated with a mercury salt solution, and a thin layer of a metal capable of forming an amalgam having great affinity for the coating metal is deposited by electrolytic means. This layer forms a complex amalgam with the iron and mercury, and the coating metal (e.g., lead, zinc, etc.) is applied by dipping. The electroplating may precede the treatment with a mercury salt.—J. W. D.

Electric arc welding and metal cutting; Electrodes for —. W. H. Boorne. E.P. 152,257, 3.2.20. Addn. to 132,354 (J., 1919, 827 A).

A COVERING for an electrode employed in electric welding in confined positions is composed of calcined magnesite, calcium carbonate, aluminium, graphite, and sodium silicate. Iron peroxide and/or ferrous silicate may be used instead of, or in addition to, the graphite.—J. S. G. T.

Electric welding, cutting, reducing, or like operations; Electrodes for —. C. J. Holslag. E.P. 152,733, 12.7.19.

THE electrode is provided at one or both ends with a coating or covering tapering away from the end.

Magnesium and chlorine; Apparatus and process for the electrolytic decomposition of anhydrous magnesium chloride and production of —. E. A. Ashcroft. E.P. 152,402, 9.7.19.

AN apparatus for the electrolytic production of magnesium from magnesium chloride consists of a pair of cast-steel cells arranged in series, in the lower part of which is a layer of molten lead alloyed with 5–10% of magnesium which is kept at constant level by a weir across the cell. The cells are connected by passages at the bottom with opposite pockets, which enable the alloy to be introduced or withdrawn as required. An electrolyte of fused anhydrous magnesium chloride (E.P. 152,401; p. 783 A) is superposed on the alloy, the cell walls in this part being lined with refractory material. Each cell is provided with a lid from which depend graphite anodes in one cell and iron or steel cathodes in the second one. In operation the magnesium produced in the primary cell is collected in the lead alloy, while in the second cell the metal withdrawn from the alloy floats on the electrolyte and is deposited on the iron or steel cathode from which it is removed periodically. The process is made continuous by ladling lead alloy from the primary into the secondary cell at suitable intervals and by the introduction into the primary cell of fresh magnesium chloride as required. The temperature of the cells should be maintained at 750° C. Quantitative yields of magnesium of a high degree of purity, and of chlorine, are obtained.—W. J. W.

Magnesium or alloys of magnesium; Production of — and a by-product [magnesium chloride]. E. A. Ashcroft. E.P. 152,403, 9.7.19.

HYDRATED magnesium chloride is dehydrated (E.P. 152,401; p. 783 A) and the anhydrous chloride electrolysed to produce magnesium or an alloy (E.P. 152,402; *supra*). The chlorine liberated is absorbed in magnesium oxide emulsion, thus producing magnesium chlorate as by-product and hydrated magnesium chloride, which after separation of the chlorate is utilised again in the process.—J. W. D.

Flux solder for aluminium; Combined —. A. J. Ansell. E.P. 152,486, 8.9.19.

TIN 6lb., zinc 2lb., aluminium 1½ lb., lead ¾ lb., and a hardener, consisting of 2 oz. of copper and 2 oz. of aluminium, are melted together with cryolite powder and lithium fluoride in a graphite crucible,

and the molten material is fluxed with manganese chloride and ammonium chloride.—J. W. D.

Magnetic separators for treating ferrous ores. F. Quinónero. E.P. 152,549, 24.12.19.

A ROTATING cylinder surrounding stationary magnets is mounted above a shaking table, and longitudinal projections (bars of copper) on the periphery of the cylinder engage with the ore particles on the table and facilitate separation of the non-magnetic from the magnetic particles, the latter being carried by the cylinder to a receptacle.

Ore-flotation agent; Process of producing an —. H. Cannon, C. Barbre, and E. H. Leslie, Assrs. to General Petroleum Corp. U.S.P. 1,337,542, 20.4.20. Appl., 8.10.17.

WATER which has been in contact with petroleum or petroleum products, especially with cracked oil during refining, is an efficient flotation agent. It is made alkaline and concentrated for transportation, and when required for use is treated with acid. It is used in conjunction with the oils commonly used for flotation.

Flotation process [for recovery of molybdenite]. E. B. Thornhill, Assr. to General Engineering Co. U.S.P. 1,338,264, 27.4.20. Appl., 8.3.18.

THE ore pulp is treated with oil as in the usual flotation process, whereby molybdenite is "coagulated" in preference to other minerals, e.g., iron and copper pyrites. The resulting concentrate is screened; the "uncoagulated" minerals pass through the screen and are returned for re-treatment.

Zinc and cadmium; Process for separating and recovering — by acid treatment. J. Leib. G.P. 322,142, 15.2.17.

PRODUCTS containing cadmium and zinc are stirred with water and sufficient concentrated acid is added to dissolve the zinc, leaving the other metals as a sponge or scum. The zinc solution is evaporated in a fireclay furnace the floor of which consists of a fireclay arch under which the heat is applied. The cadmium is recovered from the "sponge" by treating it with sulphuric acid and an oxidising agent, e.g., nitrous acid. From this solution the cadmium is precipitated by means of a zinc-iron couple.

—A. R. P.

Ore-roasting furnace; Annular, mechanical —. C. L. A. and H. G. H. Promnitz. G.P. 323,469, 18.3.13.

THE carrier for the stirrers is in the form of a closed ring and serves to close the inner side of the hearth. Each hearth has its own stirring mechanism, which is independent of that of the other hearths, so that it is possible to run each rabble backwards or forwards, fast or slow, or to stop it for cleaning or emptying one hearth without interfering with the operation of the others.—A. R. P.

Zirconium; Production of — from its ores or oxide. W. North and H. Loosli. G.P. 324,945, 15.11.19.

ZIRCONIUM ore or oxide is heated under pressure in an electric furnace in presence of a reducing agent such as charcoal. The formation of zirconium fume is thus avoided.—W. J. W.

Aluminium; Manufacture of —. V. Gerber. E.P. 139,194, 18.2.20. Conv., 18.2.19.

SEE U.S.P. 1,350,150 of 1920; J., 1920, 657 A.

Metal and metallic articles; Preservation of —. N. C. F. Jensen. U.S.P. 1,354,487, 5.10.20. Appl., 23.4.19.

SEE E.P. 149,371 of 1919; J., 1920, 694 A.

Furnace blast tuyères; Manufacture of [water-cooled annular] —. C. O. Bower. E.P. 152,566, 2.3.20.

See also pages (A) 773, *Roasting minerals* (E.P. 130,978). 774, *Kilns* (E.P. 152,509). 783, *Titanium and vanadium compounds* (G.P. 324,581); *Hydrogen* (E.P. 152,554). 781, *Porous material from slag* (E.P. 136,818). 785, *Slag cement* (E.P. 149,988).

XI.—ELECTRO-CHEMISTRY.

Galvanic cells and accumulators; New —. Pradel. Elektrotechn. Anzeiger, 1920, 70, 72. Chem.-Zeit., 1920, 44, Rep., 260.

A dry cell, of which the positive system may be renewed, is constituted of the combination lead peroxide, dilute sulphuric acid, zinc. The average discharge E.M.F. is 2.3 volts. When the cell runs down the zinc plates are replaced by others, and are meanwhile recharged between two lead electrodes in dilute sulphuric acid. Spent manganese dioxide employed as depolariser is revived by powdering, treating successively with solutions of salts which dissolve out salts of ammonium and zinc, with superheated steam under high pressure, and with ozone, hypochlorous acid, or a halogen. Alternatively, oxidation may be effected electrolytically, the electrolyte employed containing hypochlorous acid, chloric acid, a hypochlorite, or salts such as magnesium chloride, which yield a solution of a hypochlorite on being electrolysed, or potassium manganate or permanganate, bichromate, etc. —J. S. G. T.

Electrode processes. Stäger. See X.

PATENTS.

Electric furnaces. H. Wade. From Booth Electric Furnace Co. E.P. 152,904, 9.1.20.

SEE U.S.P. 1,332,795 of 1920; J., 1920, 375 A.

Electrolysis of aqueous solutions by means of propulsive electrodes. A. Luib and E. Steinbuch, Assrs. to Soc. of Chem. Ind. in Basle. U.S.P. 1,354,498, 5.10.20. Appl., 29.1.13.

SEE E.P. 11,872 of 1913; J., 1913, 981.

See also pages (A) 774, *Distillation apparatus* (G.P. 323,414). 782, *Fixation of nitrogen* (U.S.P. 1,334,590). 787, *Electric furnaces* (E.P. 152,145 and 152,176). 788, *Electrodes for welding etc.* (E.P. 152,257 and 152,733); *Magnesium and chlorine* (E.P. 152,402). 802, *Pinnacle* (G.P. 324,919 and 324,920).

XII.—FATS; OILS; WAXES.

Oils; Hydrogenation of —. M. Fréjacque. Chim. et Ind., 1920, 4, 443–456.

LINSEED oil with iodine value 160 was hydrogenated under the same conditions, with basic nickel carbonate, reduced respectively in dry hydrogen and in the oil itself, as catalyst, the amount of nickel in each case being 1% of the oil. With the nickel previously reduced at 160° C. the iodine value after 4 hrs. was 152, and with that reduced at 210° C. it was 133, whilst with the same catalyst reduced in the oil itself at 160° and 210° C. the iodine value of the oil was reduced to 70 and 20 respectively. These results confirm the conclusions of Siegmund and Suida (J., 1915, 970).—C. A. M.

Perilla oil. H. A. Gardner and P. C. Holdt. Paint Manufacturers' Assoc., U.S.A., Circ. 106, Oct., 1920. 8 pp.

PERILLA seed contains 33–35% of a greenish-yellow or brownish oil of characteristic, agreeable odour,

which in many respects resembles linseed oil. It dries to a tough film slightly more slowly than raw linseed oil, but the acceleration of oxygen absorption with the addition of a drier appears to be greater than is the case with linseed oil. Its most obvious defect is the tendency of the film to "crawl" or "creep" into droplets on a surface, a fault which can be inhibited by increasing its viscosity by bodying-up (blowing) or by admixture with an oil of lower surface tension, e.g., linseed or soya bean oil. A sample of perilla oil secured direct from the Orient had the following characters:—sp. gr. at 15.5° C., 0.9351; moisture and volatile matter, 0.07%; acid value, 7.0; iodine value, 198.8; saponif. value, 193.1; unsaponifiable matter, 1.07%; n_D^{25} = 1.4802. The wetting power of perilla oil is greater than that of linseed oil as measured by the determination of the oil absorption of pigments (J., 1920, 306 A), 20 g. of lithopone absorbing 4.7 c.c. and 5.2 c.c. of the two oils respectively. The oil shows no separation of mucilage on heating quickly to 310° C., and little darkening in colour takes place at this temperature, thus indicating its suitability for the manufacture of pale varnishes. As a paint vehicle perilla oil is equal to linseed oil in regard to weathering; the "creeping" effect observed when it is used in conjunction with white lead may be corrected by previously heating the oil to 500° F. (260° C.) or mixing it with a proportion of raw linseed oil.—A. de W.

Chia [Salvia hispanica] oil. H. A. Gardner and P. C. Holdt. Paint Manufacturers' Assoc., U.S.A., Circ. 105, Oct., 1920. 8 pp.

A SAMPLE of chia seeds from Mexico gave the following results:—Oil obtained by pressing, 22.33%; oil in press cake, 10.5%; nitrogen in cake, 3.6%; P_2O_5 in cake, 1.5%. The filtered oil gave the following data:—Sp. gr. at 15.5° C., 0.9338; acid value, 0.6; iodine value, 192.2; saponif. value, 192.2; unsaponifiable matter, 0.8%; n_D^{25} = 1.4855. The oil was clear and of light amber colour. It dried slowly in its raw state and the surface in a film tended to pull together at points, indicating high surface tension. When siccatised, the oil dried as rapidly as linseed oil. The "crawling" tendency of the oil film is practically overcome by heating to 500° F. (260° C.) for 15 mins., this treatment yielding an oil which when siccatised with a liquid drier, showed even superior drying power to linseed oil treated with the same amount of drier. The wetting power of the oil as measured by the method for determining the oil absorption of pigments (J., 1920, 306 A) was lower than that of linseed oil, 6.2 c.c. of chia oil as against 5.1 c.c. of linseed oil being absorbed by 20 g. of white lead. The maintenance of its colour during heat treatment indicates the suitability of chia oil for the manufacture of pale varnishes. It seems probable that several species of *Salvia* contain oils exhibiting varying degrees of drying power. —A. de W.

Fatty acids; Anhydrides of the higher aliphatic —. III. D. Holde and H. Smelkus. Ber., 1920, 53, 1889–1897.

THE dehydration of the fatty acids of linseed oil and of commercial olein by acetic anhydride at 150°–160° C. has been studied. Acetic acid and excess of acetic anhydride cannot be removed from the products by steam since the fatty anhydrides are thereby almost completely converted into the acids. Crystallisation from alcohol and distillation under diminished pressure are unsuitable for the separation of fatty anhydrides from the parent fatty acids, but this can be effected by means of solid sodium carbonate or aqueous solutions of the latter. The proportion of acid to anhydride in the mixtures cannot be determined by titration with N/10 or N/20 alcoholic alkali, which causes a gradual con-

version of the fatty acid into the corresponding ester and salt.—H. W.

Fatty acids; Anhydrides of the higher aliphatic —. IV. D. Holde and I. Tacke. Ber., 1920, 53, 1898—1907. (Cf. *supra*.)

THE mixed fatty acids from linseed and rapeseed oils and oleic acid can be satisfactorily dehydrated at 150°—160° C. with 3.5 times the amount of acetic anhydride theoretically necessary, and it is possible that this proportion can be further greatly reduced and that the operation can be effected at the ordinary pressure. The excess of reagent is removed by distillation under diminished pressure and the products are further purified by agitation with powdered sodium carbonate or aqueous sodium carbonate solution (5%). Their purity is conveniently examined by determination of the mol. wt. in freezing benzene. The mixtures have generally a higher melting point than the acids from which they are derived and, at the ordinary temperature, are semi-solid and sometimes partly crystalline. In viscosity, volatility, and refractive index they closely resemble the corresponding glycerides. They are decomposed by *N*/10 or more dilute alcoholic alkali into almost exactly equivalent amounts of fatty salt and ester, but the so-called saponification value can be determined in the usual manner. Mixed anhydrides of the fatty acid and acetic acid are not present.—H. W.

α -Monoglycerides; New synthesis of —. E. Fischer, M. Bergmann, and H. Bärwind. Ber., 1920, 53, 1589—1605.

ACETONEGLYCEROL, $(\text{CH}_3)_2\text{C} < \begin{matrix} \text{O} \cdot \text{CH}_2 \\ \text{CH} \cdot \text{CH}_2\text{OH} \end{matrix}$, reacts readily with acyl chlorides in the presence of quinoline yielding substances from which the acetone residue is easily removed, generally by treatment with acid, yielding α -monoglycerides. The preparation of α -benzoyl-, α -*p*-nitrobenzoyl-, α -stearyl-, α -lauryl-, and α -palmityl-glycerol is described together with that of several mixed glycerides obtained by the further action of acid chlorides on these products. (Cf. J.C.S., Dec.) —H. W.

Acetoneglycerol [isopropylideneglycerol] and its applicability to the preparation of pure α -glycerides. A compound of phosphoric acid and glycol. E. Fischer and E. Pfähler. Ber., 1920, 53, 1606—1621.

ACETONEGLYCEROL is transformed by acetic anhydride and pyridine into acetylisopropylideneglycerol, b.p. 84° C. at 9 mm., 87° C. at 12 mm., which is converted by *N*/4 sulphuric acid into α -monoacetin, b.p. 103° C. at 0.4 mm. Even at the lowest possible pressure slight decomposition occurs during distillation and it is thus very probable that all the mono- and di-glycerides described previously in the literature and purified by distillation under diminished pressure are not homogeneous. Phosphoryl chloride converts acetoneglycerol into isopropylidene- β - γ -dihydroxypropyl phosphate which is transformed by sulphuric acid into α -glyceryl phosphate and di-isopropylideneglycerol monophosphate. Ethyleneglycol is converted by phosphoryl chloride and pyridine at -20° C. into β -chloroethyl phosphate, $\text{Cl} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{O} \cdot \text{PO}(\text{OH})_2$. The introduction of the dihydroxypropyl complex into other substances is most readily effected by means of acetoneglycerol- α -iodohydrin; the chlorine atom of the chlorohydrin is too firmly retained for this purpose. —H. W.

Glycerin; Manufacture of —. Verbeek. Seifens.-Zeit., 1920, 44, 47, 133, 190, 234. Chem.-Zeit., 1920, 44, Rep., 187.

THE apparatus of van Ruymbeke (E.P. 24,556 of 1893 and 12,367 of 1894; J., 1894, 1070, 1208) is

the most suitable for the distillation of impure crude glycerins, and apparatus with rectifying columns for saponification glycerins. In the former method a sweet water containing 5—15% of glycerol is left, whilst in the latter the sweet water is practically free from glycerol. In van Ruymbeke's process it is best to use steam at a pressure of at least 10—14 atm. so as to obtain a temperature of at least 175° C. Better rectification is effected by having the columnar vessel of the condenser in a slanting rather than in a horizontal position. In the subsequent concentration of the distillate in the receiver a temperature of 125° C. must not be exceeded, and the pressure of steam is therefore reduced to 2 atm.—C. A. M.

Fats and oils as source of vitamin A. Drummond and Coward. See XIXA.

Glycerol substitutes. Neuberg and Reinfurth. See XX.

Butter fat and other fats. Reilly and Hickinbottom. See XXIII.

PATENTS.

Tung or other vegetable oils; Nitration of —. G. H. Howso. E.P. 152,095, 4.7 and 16.8.19.

FATTY oils, fatty acids from these, or mixtures thereof, are nitrated at reduced, atmospheric, or increased pressures with strong or dilute nitric acid in the presence of inert solvents, e.g., petroleum spirit or the naphthene residue from the nitration of Borneo spirit. For example, tung oil is mixed with 50% by vol. of petroleum spirit, the mixture mechanically agitated in a closed jacketed pan, and 10—15% of commercially pure nitric acid slowly run into the mixture, which may be heated not above 40° C. The mixture is maintained at this temperature and agitation continued until only a faint trace of free nitric acid remains. The excess of acid may be removed either by addition of a further quantity of tung oil and solvent or by addition of a solvent capable of nitration, e.g., heavy solvent naphtha. The nitrated mixture may then be used as such in the manufacture of varnishes, paints, rubber substitutes, insulators, etc., or the volatile solvent may be recovered in a reduced pressure recovery plant.—A. de W.

Powdered metals and reduced metallic compounds [as catalysts]; Making —. Process of making catalytic material [for hydrogenating oils]. C. Ellis. U.S.P. (A) 1,329,322 and (B) 1,329,323, 27.1.20. Appl., (A) 1.8.19 and (B) 31.1.19.

(A) A CATALYTIC material containing nickel or other metal with atomic weight between about 58.7 and 63.6 is reduced in paraffin wax, at the cracking temperature of the latter. (B) Catalytic material containing nickel in combination with an acid of the nitrogen oxide group is heated in a reducing atmosphere, sintering being prevented by the introduction of a diluting agent during the early stages of heating.—C. A. M.

Wool fat; Process of saponifying —. I. Lifschütz. G.P. 324,667, 28.5.18.

WOOL fat is digested with aqueous alkali solution at a moderate temperature until the fatty acids liberated from a sample show an acid value corresponding to the saponification value of the original fat.—C. A. M.

Soap; Process for the manufacture of —. J. J. Morel. E.P. 131,881, 13.8.19. Conv., 28.8.18.

THE sodium carbonate used in the manufacture, e.g., of oleic acid soap, is replaced by the crude sodium sulphite obtained, e.g., on fusing benzene-

sulphonic acid with sodium hydroxide. The sulphur dioxide liberated is either collected, or is used for decomposing calcareous soaps.—L. A. C.

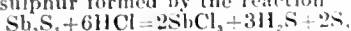
Catalyst [for hydrogenating oils]. H. K. Moore, Assr. to Brown Co. U.S.P. 1,326,767, 30.12.19. Appl., 4.4.16

SEE E.P. 100,095 of 1914; J., 1916, 178

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Antimony sulphides; Chemical examination of —. A. van Rossem and P. Dekker. *Indiarubber J.*, 1920, 60, 905—908.

ANTIMONY pentasulphide is decomposed by hot carbon bisulphide with liberation of sulphur; the determination of the free sulphur content by extraction in the customary manner is therefore inaccurate. Provided that little or no antimony trisulphide is present, the estimation of free sulphur can be effected by treating 1 g. with 25 c.c. of boiling concentrated hydrochloric acid for 10 mins., the sulphur in the insoluble portion being determined by drying, extracting with carbon bisulphide, and weighing; this weight includes both the free sulphur and the sulphur formed by the reaction



and a correction is made in the calculation of the amount of original free sulphur. Weber's method for estimating free sulphur by removing the antimony sulphide with aqueous ammonia is applicable only in the absence of trisulphide and with the use of ammonia of the lowest possible concentration. Calcium sulphate is estimated by extracting with hot water and precipitation as calcium oxalate. In isolating possible adulterants by treating with a solution of caustic alkali, prolonged heating is inadvisable on account of the tendency of part of the antimony to separate again from solution.

—D. F. T.

Carbon black; Channel process of making —. R. O. Neal. *Chem. and Met. Eng.*, 1920, 23, 729—731.

The channel process of making carbon black from natural gas is that most widely employed. The condensing buildings are made of sheet iron attached to a steel frame-work, and in the case of those containing two groups of channels are 18 to 20 ft. wide and 80 to 115 ft. long. The condensation channels are of mild steel, 7 to 8 in. wide, bolted together in lengths up to 100 ft., and are supported by trucks running on overhead rails. The condensed carbon is removed by means of scrapers preferably in direct contact with the channels, while the trestles or tables on which the channels rest are moving in one direction only. Most of the plants have eight rows of such channels upon each table or trestle, which is moved continually to and fro over a distance of 55—60 ins. in 15 mins., and as a rule, there are side scrapers for removing the carbon from the sides of the channel beams. The carbon falls into hoppers and is then conveyed by screw conveyors to the bolting machine. The burners for the gas, between the hoppers, are fitted with lava tips made from steatite, which is subsequently hardened at 2000° F. (1090° C.). They should produce a uniform flame with uniform gas consumption. The size of the steel screen in the bolting machines usually ranges from 45 to 60 meshes per in.—C. A. M.

Carbon black; The disc, plate, and cylinder processes for the production of —. R. O. Neal. *Chem. and Met. Eng.*, 1920, 23, 785—789.

A DETAILED description of the plant used in each process is given, together with instructions for the care of the large number of gears that are required.

The disc and plate processes, in which the black is deposited on rotating discs of 36—42 in. diam., or on stationary or rotating plates of 24 ft. diam., produce approximately the same amount of black per 1000 cu. ft. of gas, but the cylinder system, in which the black is deposited on rotating cylinders, 3—8 ft. long and 8½ in. diam., produces much less, although the product is much more valuable, especially for making high-grade printing inks. Carbon black produced by cracking natural gas is very dense and of a grey colour and contains gritty particles which render it practically useless for most commercial purposes.—A. R. P.

Rosin tung esters ("tunga resin"); Production of — and application in varnish manufacture. H. A. Gardner and R. E. Coleman. *Paint Manufacturers' Assoc.*, U.S.A., Circ. 101, Aug., 1920. 22 pp.

NEUTRAL glycerin-rosin esters for use in varnish manufacture can be prepared in an open varnish pot by employing tung oil as an esterifying catalyst, whereby "tunga resin," an ester-like resin of low acid value is obtained. Rosin lost 19—21% of its weight as fume when heated alone to a temperature and for a time sufficient to carry out esterification with glycerin, whilst the acid value of the residue remained substantially unchanged. The presence of 6% of lime reduced the loss to 7%, and the acid value of the residue dropped from 158.5 to 78.5. On heating such limed rosin with 12% of its weight of glycerin to 350° F. (177° C.) in 20 mins., then to 440° F. (223° C.) in 20 mins., and finally to 500° F. (260° C.) in 10 mins. the acid value fell to 61, and the cloudiness due to the limed rosin disappeared. Under nearly similar conditions of experiment rosin and glycerin combined to only a small extent, a residue with an acid value of 141.6 being obtained, whilst the additional presence of 5% of tung oil resulted in a product with an acid value of 123.4. A brown tunga-resin of acid value (in alcohol) 21.8 (in alcohol-benzol 24.4) was obtained by the following method, the loss by fuming being 8.3%. 80 parts of tung oil is heated in 10 mins. to 350° F. (177° C.), 12 pts. of lime is added, and the temperature maintained for 20 mins. 200 pts. of rosin is then added and the temperature raised in 20 mins. to 425° F. (219° C.), the flocculent calcium tungate formed going into solution. The temperature is allowed to drop to 350° F. (177° C.), 30 pts. of glycerin added, the temperature raised in 20 mins. to 450° F. (232° C.), and then in 7 mins. to 500° F. (260° C.). The mass is then cooled to 475° F. (246° C.), and maintained thereat for 45 mins. The reduced losses by fuming during esterification in the presence of tung oil may be due to the head of froth formed by the tung oil acting as a protecting layer against oxidation by air. The methods of preparation of three typical varnishes comprising tunga-resin and tung oil are given (*cf.* J., 1920, 664A).

A. de W.

Varnish fume recovery. C. H. Jones. *Chem. and Met. Eng.*, 1920, 23, 16, 771—776.

THE necessity of reducing the nuisance caused by the escape of noxious fumes from the factory has led to the recovery of products previously regarded as waste and has thus resulted in considerably more economical working. Typical modern installations are described and illustrated with photographs and plans. The fires are operated with positive suction to ensure a good draught and minimise smoke issuing from a short pipe on the roof of the pent-house which serves in place of a stack. The charged gum pot is placed on the fire and a fume exhaust cover, attached to a collapsible swinging pipe easily manipulated by hand, lowered on to it. "Cooking" for 1½ hrs. was necessary under the old system, but 30 mins. is sufficient with the new arrangement. The lid and exhaust system minimise the possibility

of flashing or burning, and the insertion of screens in the flues permits of the location of spontaneous combustion. After incorporation of the oil the gum pots with their charges are removed to the cooling room and again connected with another portion of the same fume recovery system. The fumes from these two sources are drawn through a water-scrubbing tank in a pent-house on the upper floor, where the heavier particles settle out and the remainder pass through a turbine to a similar tank filled with lime water. Products are recovered from the condensed matter on the inner surfaces of the piping ahead of the water-tank and from the deposit in the water-scrubbing tank, and the lime-soap emulsion formed in the lime scrubber is boiled to remove excess of lime water and neutralised with acid. The recovered products are ultimately returned to the gum pot. The ultimate exhaust from the fume recovery system possesses only a faint soapy odour. The thinner recovery system operates entirely separately from the fume recovery system. The condensing system returns the condensed solvent to the gum pot, a loss of only 2% of the total solvents used being thus obtained. The finished varnish is pumped to large settling tanks and thence through a De Laval centrifugal separator, such clarification having been found superior to methods involving filtration. Records of runs of typical varnishes in a non-recovery plant are given and show the losses in gum-resin as fume to be 30–42%.—A. de W.

Japan lac; Main constituent of —. VII. *Urushiol monomethyl ether and the mechanism of the oxidation of urushiol.* R. Majima and G. Takayama. Ber., 1920, 53, 1907–1916. (Cf. J., 1916, 128, 129, 1007.)

ONE of the two hydroxy groups of urushiol is more readily methylated than the other and the product obtained by partial methylation is shown by a comparison of the properties of its tetrahydro-derivative with those of 2-hydroxy-*m*-tolyl methyl ether to be 2-hydroxy-3-methoxyurushiol. The tolyl derivative can be oxidised to a derivative of diphenquinone and since precisely similar phenomena are observed during the oxidation of hydro-urushiol it appears valid to conclude that analogously constituted products are also formed in this case. (Cf. J.C.S., Dec.)—H. W.

Resin acids; Relationship of retene to —. *Hydrogenated retenes.* A. J. Virtanen. Ber., 1920, 53, 1880–1889.

RETENE is converted by sodium and boiling amyl alcohol into dihydroretene, m.p. 98.5°C., b.p. 208°–210°C. at 10 mm., and tetrahydroretene, yellowish oil, b.p. 180°–183°C. at 10 mm., sp. gr. 1.0057 at 10°/4°C., $n_D^{20}=1.56061$. Fuming hydriodic acid (sp. gr. 1.96) and red phosphorus transform retene into hexahydroretene, b.p. 175°–177°C. at 10 mm., sp. gr. 0.9802 at 20°/4°C., $n_D^{20}=1.54705$, octahydroretene, b.p. 163°–165°C. at 10 mm., sp. gr. 0.9578 at 20°/4°C., $n_D^{20}=1.53023$, dekahydroretene, b.p. 155°–158°C. at 10 mm., sp. gr. 0.9342 at 20°/4°C., $n_D^{20}=1.51501$, and dodekahydroretene, b.p. 148°–150°C. at 10 mm., sp. gr. 0.8985 at 20°/4°C., $n_D^{20}=1.48510$. The author expresses the view that all resin acids having the formula $C_{20}H_{30}O_2$ contain in their molecule a hydrogenised retene nucleus. (Cf. J.C.S., Dec.)—H. W.

Perilla oil. Chia oil. Gardner and Holdt. See XII.

PATENTS.

Phenol-formaldehyde condensation products and the production thereof. A. W. Weller and W. T. Robinson-Bindley. E.P. 152,384, 10.6.19.

THE phenol employed contains up to 0.8% of cellulose acetate or nitrocellulose in solution. The

products are non-brittle and much stronger than those obtained by the usual process.—L. A. C.

Lacquer paints; Preparation of quick-drying —. Deutsch-Luxemburgische Bergwerks u. Hütten-A.-G., and S. Hilpert. G.P. 324,722, 23.11.16.

RESINOUS substances separated from the waste acid from the treatment of coal tar oils, without carbonisation, are dissolved in volatile solvents and mixed with pigments, with or without the addition of other resins, drying oils, driers, etc.—C. A. M.

Luminous radioactive materials and objects; Manufacture of —. E. Parade, E. von Hidwég, and P. Faulstich. G.P. 324,875, 24.11.16.

THE materials are first coated with Sidot blende or zinc sulphide or other luminous composition, and are then treated with a solution of radioactive substance, an economy of the radioactive matter being effected by the application of the same as a separate operation.—A. de W.

Artificial resin; Preparation of — from wood tar. S. Hamburger. G.P. 324,876, 12.12.17.

WOOD tar is heated with the oxides of heavy metals, e.g., with zinc oxide at about 110°C., and then for a short time at a higher temperature. From 10–12% of zinc oxide can be absorbed by the tar without injuring the properties of the resin. —C. A. M.

Lacquers. G.P. 324,786. See V.

Nitration of oils. E.P. 152,095. See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Antimony sulphide. Van Rossem and Dekker. See XIII.

PATENT.

Nitration of oils. E.P. 152,095. See XII.

XV.—LEATHER; BONE; HORN; GLUE.

Tannins. VI. Chebulinic acid. II. K. Freudenberg and B. Fick. Ber., 1920, 53, 1728–1736. (Cf. J., 1919, 591 A.)

THE crystalline product obtained by the action of hot water on chebulinic acid is definitely proved to be a digalloylglucose; since it is unaffected by emulsin, phaeolunatae, or yeast extract it is not a β -glucose. The amorphous acid produced simultaneously yields a sparingly soluble brucine salt and appears to have the formula, $C_{14}H_{14}O_{11}$; it gives pyrogallol when distilled under diminished pressure. (Cf. J.C.S., Dec.)—H. W.

Gelatin; Swelling of — in acids. W. R. Atkin. J. Soc. Leather Trades' Chem., 1920, 4, 248–257.

THE results obtained by different workers at different times in swelling experiments on gelatin left for 48 hrs. in solutions of various monobasic acids of varying concentrations are compared, and it is shown by curves that the maximum swelling occurs at a definite hydron concentration, $p_H=2.4$ or $0.004N$. The curves indicate that acetic acid might also show a maximum swelling if solution of the gelatin did not take place at that concentration. The quantity, e (Pröcter and Wilson, J., 1916, 645), representing the excess of the concentration of diffusible ions in the jelly over that of the external solution, exhibits a maximum value at the point of maximum swelling. Minimum swelling is obtained at the isoelectric point of gelatin, $p_H=4.6$. Thus the quantity, e , and the swelling are functions of the hydron concentration of the external acid solution.—D. W.

PATENTS.

Tannin; Manufacture of —. Chem. Fabr. Grünau, Landshoff and Meyer, A.-G., and L. Schlein. G.P. 323,135, 25.1.16.

SUBSTANCES containing tannin are extracted with water, the extract is kept below 11° C. for a prolonged period, and the non-tannins which separate are filtered off.—W. J. W.

Leather for linings, especially from inferior portions of hides; Process for increasing the durability of —. H. Hoffmeister and J. Paessler. G.P. 324,495, 16.3.17.

LEATHER, after tanning, is steeped in a solution of wood-tar in benzene or methyl alcohol, in presence of an organic acid, then dried, and dressed. If a vegetable tanning agent has been used, the excess is first washed out with water containing formalin, and the leather is dried before steeping in the wood-tar solution. In the case of tawed leather, the treatment with wood-tar is preceded by immersing the leather in a strong solution of a tanning agent or in purified, concentrated cellulose waste-liquor, followed by drying.—W. J. W.

XVI.—SOILS; FERTILISERS.

Soils; Flocculation of —. N. M. Comber. J. Agric. Sci., 1920, 10, 425—436.

THE silt fraction of soils, when suspended in water, is most easily flocculated by calcium salts when the suspension is neutral, the addition of alkali stabilising the suspension. The clay fraction, however, is flocculated from alkaline suspensions more readily than from neutral suspensions. It is suggested that the clay particles are protected by emulsoid colloids, and thus behave as emulsoids and not suspensoids. In this way the action of lime which, although alkaline, flocculates clay is correlated with the present views on colloid chemistry. The experimental results indicate that the clay, as an emulsoid, protects the larger particles in a soil which by themselves are suspensoid. Fine silt soils are not flocculated by calcium hydroxide owing to the inefficiency of the relatively small amount of emulsoid clay in protecting the larger suspensoid surface exposed by the fine silt.—W. G.

Soils; Qualitative test for sour —. N. M. Comber. J. Agric. Sci., 1920, 10, 420—424.

ABOUT 2—3 g. of the air-dried soil is shaken in a test-tube with 5 c.c. of a 1° alcoholic solution of potassium thiocyanate. The development of a pink or red coloration in the liquid on standing indicates that the soil has a lime requirement as measured by the Hutchinson-MacLennan method (J., 1914, 932). The test is based on interchange between the potassium ions in the thiocyanate solution and the iron and aluminium ions in the sour soil, and cannot be used for quantitative measurements, but on the same soil the test may afford useful indications of the variation in acidity.—W. G.

Soil; Determination of nitrates in —. A. L. Whiting, T. E. Richmond, and W. R. Schoonover. J. Ind. Eng. Chem., 1920, 12, 982—984.

ONE hundred g. of the soil is shaken for 3 hrs. with 300 c.c. of 0.5% hydrochloric acid, the mixture allowed to settle for 18 hrs., and 200 c.c. of the clear liquid then transferred to a flask containing 5 g. of sodium hydroxide. The contents of the flask are boiled until the volume is reduced to about 25 c.c. (if urea is present, the evaporation is continued to dryness), 200 c.c. of water is added together with 0.5 g. of Devarda's alloy (Al 50, Cu 45, Zn 5%) and the mixture is distilled for 40 mins., the ammonia being collected in standardised acid.

—W. P. S.

Aluminium as a factor in soil acidity. J. J. Mirasol. Soil Sci., 1920, 10, 153—217.

FURTHER evidence is adduced in support of the view that aluminium is apparently the determining factor in the acidity of many soils (cf. Hartwell and Pember, J., 1919, 112a). The toxic effect due to aluminium may be corrected by the application of calcium carbonate, and may be reduced by the application of calcium superphosphate. In so far as aluminium is a factor in soil acidity, the author considers that Hopkins' method (cf. U.S. Dept. Agric. Bur. Chem. Bull., 1903, 73, 114) is the best one for soil acidity determinations. (Cf. J.C.S., Dec.)—W. G.

Nitrogen economy in the soil as influenced by various crops grown under control conditions. R. C. Wright. Soil Sci., 1920, 10, 249—289.

IN pot culture experiments it was found that frequent cultivation and consequent excessive aeration caused a loss of total nitrogen. Under certain crops there was a loss of nitrogen in excess of that recovered in the crops, the actual loss varying with the crop and the soil on which it was grown. This loss was found with certain leguminous as well as non-leguminous crops. Under the experimental conditions, where there was nitrogen-fixation by the growth of certain legumes, this nitrogen was found in the crop above the ground, and if the crop was removed and not dug in, the soil was depleted of nitrogen in the same way as if a non-leguminous crop had been grown and removed. The losses of nitrogen are to be attributed to volatilisation of either free nitrogen or ammonia.—W. G.

Phosphate rock and sulphur; Co-operative experiments for the composting of —. W. B. Ellett and W. G. Harris. Soil Sci., 1920, 10, 315—325.

UNDER the experimental conditions the addition of sulphur to a compost of soil and rock phosphate increased the availability of the phosphate, and a slightly better result still was obtained if, in addition, horse manure was added. In the absence of sulphur the manure was not effective. Oxidation of the sulphur preceded the increase in availability of the phosphate. The addition of rock phosphate to manure (a mixture of horse manure and cut straw) checked fermentation slightly and reduced the loss of dry matter and nitrogen. A still further reduction was effected when sulphur also was added. In this case addition of phosphate favoured nitrate formation, whilst addition of sulphur favoured ammonification. A large number of Virginia soils were examined and showed very great variation in their powers of oxidising sulphur.—W. G.

Sulphur; Influence of initial reaction on the oxidation of — and the formation of available phosphates. J. G. Lipman and J. S. Joffe. Soil Sci., 1920, 10, 327—332.

THE results given show no advantage in starting with a relatively high hydrogen-ion concentration in the soil, produced by the addition of sulphuric acid, for the conversion of rock phosphate into a soluble form (cf. J., 1920, 166a).—W. G.

Phosphates in basic slag; Solubility in citric acid of —. A. Kaysser. Chem.-Zeit., 1920, 44, 826.

IN molten slag the phosphate exists in the form of tricalcium phosphate having a high solubility in citric acid. If the slag is slowly cooled, brown crystals are formed which are only slightly soluble; these consist of tetracalcium phosphate produced by interaction of tribasic phosphate with lime separated from its combination with silicic acid in presence of iron and manganese. Rapid cooling prevents this conversion of the soluble into the insoluble compound. Addition of silica to the slag increases the solubility of the phosphate owing to

its combination with free lime, and possibly in part to a mechanical cooling effect. On the other hand, fluorspar increases the amount of lime and causes loss of silica, the result being to diminish the solubility of the phosphate.—W. J. W.

Crude calcium cyanamide; Determination of cyanamide-nitrogen in —. H. Neubauer. *Z. angew. Chem.*, 1920, 33, 247–248, 254–256.

ESTIMATIONS of the total nitrogen in crude cyanamide do not indicate the condition of the material, as they do not discriminate between nitrogen present as cyanamide and as dicyanodiamide, which latter compound is formed in deteriorated cyanamide and is injurious. In the method of treating an aqueous cyanamide solution with nascent hydrogen, the most accurate results are obtained when a weak acid is used, and at the same time an active evolution of hydrogen is secured, and for this reason acetic acid has been employed with Arnd's alloy (Cu 60%, Mg 40%). The crude cyanamide should first be treated with pure acetone to dissolve any dicyanodiamide present. 1 g. of finely powdered material is placed on a cotton wool filter, and 100 c.c. of acetone in successive amounts of 20 c.c. is poured on it. The residue is transferred to a distillation flask, and 3 g. of Arnd's alloy, 5 c.c. of 20% magnesium chloride solution, and 15 c.c. of glacial acetic acid are added, the mixture being shaken, and loss by spitting guarded against. After 10 mins. a further 3 g. of alloy is added, the sides of the flask are rinsed and the contents allowed to stand for 10 mins., after which they are diluted to 300 c.c., some paraffin is added to reduce frothing, and after addition of alkali, the ammonia is distilled off and estimated; the nitrogen present as cyanamide can then be calculated. To ascertain the degree of deterioration of the original material, the acetone filtrate may be collected in a flask containing oxalic acid to fix any ammonia present, the acetone distilled off on a water-bath, and the nitrogen estimated by the Kjeldahl method. Small amounts of nitrogen retained by the carbon in the crude cyanamide are determined by acidifying the residue from the ammonia distillation, adding water, and shaking the mixture, after which the separated residue is treated by the Kjeldahl process.—W. J. W.

Oat grain and straw. Berry. See XIXa.

Pickering sprays. Cook. See XIXb.

PATENTS.

Phosphates; Mechanical treatment of tricalcic —. W. P. Thompson. From La Comp. des Phosphates de Constantine. E.P. 151,684, 1.4.6.19.

NATURAL phosphates consisting of material rich in phosphate associated with poor material are treated by grinding sufficiently to reduce the rich material to a granular condition, the poor material coating the richer as a powder. The material is passed through a rotary disintegrator, which picks the material up and causes it to fall on to beater arms. In this way the granules are separated from the powder and the whole is held in suspension in the air. A strong current is blown through the disintegrator and the material is carried into a series of compartments in which the particles are separated according to density. The granules fall first, forming a fraction richer in phosphate than the original. The powder forms a second fraction, and is sufficiently fine to dissolve completely in citric acid solution.—J. H. J.

Trees and other growing plants with wood-like stems; Treatment of growing —. A. A. Lockwood. E.P. 152,269, 15.3.20.

To stimulate the growth of trees, or to free them

from parasites, or to improve their fruit, a hole is bored in the lower part of the stem, or in the root, and suitable liquids or solutions are poured or siphoned in, the treatment commencing after the sap has begun to flow. Ammonium or sodium nitrate is beneficial to growth, solutions of citric or malic acid and iron citrate improve the quality and colour of pears and apples, while for the destruction of parasites, solutions containing both nitrogen and sulphur, such as ammonium sulphate, or mixtures of sodium sulphide and an alkali nitrate, have proved effective. Examples are given of the use of 5% sodium nitrate solution and 10% ammonium sulphate solution.—W. J. W.

Soil and seed inoculation. C. F. Diller. U.S.P. 1,354,808, 5.10.20. Appl., 29.10.19.

SELECTED soil organisms are freed from organic nutrient material and grown in water containing a small amount of an inorganic stimulating salt.—J. H. J.

XVII.—SUGARS; STARCHES; GUMS.

Cane juice; Influence of the amino-acids in — on sugar technology. H. I. Waterman and J. W. L. van Ligten. *Chem. Weekblad*, 1920, 17, 559–562.

THE previous observation of Waterman (J., 1918, 133A), that amino-acids inhibit the breaking down of the monoses by sodium hydroxide, is found to hold also for calcium hydroxide. Dextrose solutions were heated to various temperatures in presence of various quantities of lime, both with and without addition of amino-acids, and the quantity of sugar left in each case determined by the polarimeter. In the control tests the sugar was rapidly destroyed, and the solutions darkened, but amino-acids were found in nearly all cases to exert important inhibitory effects. The observations are of importance for the sugar industry, since the amino-acids present in the expressed juices must to some extent prevent destruction of the monoses by the lime used; the lime solutions used are controlled by titration with acids in presence of phenolphthalein; the amino-acids do not markedly affect this indicator.—S. I. L.

Beet-sugar factory products; Determination of moisture in —. V. L. Aikin. *J. Ind. Eng. Chem.*, 1920, 12, 979–981.

ABOUT 25 g. of sand (digested previously with hot hydrochloric acid, washed, dried, and passed through a 0.25 mm.-mesh sieve) is placed in a basin, dried, and weighed; a quantity of the sample, e.g., molasses, massecuites, etc., containing not more than 1 g. of dry substance, is then weighed into the basin, 1 c.c. of water is added, the basin is placed on a water-bath, and the mixture stirred for 3 mins. and dried at 105° C. for 6 hrs., or until the loss in weight after 1 hr. drying is less than 0.1%. —W. P. S.

Sucrose; Mechanism of the inversion of —. *Studies in catalysis.* XII. C. M. Jones and W. C. McC. Lewis. *Chem. Soc. Trans.*, 1920, 117, 1120–1133.

THE velocity of the inversion of sucrose in presence of N/10 sulphuric acid was determined at 20°, 30°, 40°, and 50° C., at various concentrations of sucrose from 10 g. to 70 g. per 100 c.c. of solution. With increasing concentration a definite increase in the velocity constant was observed. The average activities of the hydrogen ion were determined electrometrically in the various solutions, and it is shown that the alteration in the velocity constant can be completely accounted for by allowing for the stoichiometric correction for the water present

and for the change in the activity of the hydrogen ions. The inversion process is bimolecular, consisting first of the union of the non-hydrated hydrogen ion with a non-hydrated sucrose molecule, this process being practically instantaneous, and secondly the actual inversion reaction. The activity of the hydrogen ion is found to be an exponential function of the concentration of sucrose and water present, and the environmental catalytic influence of a molecule of sucrose is identical in magnitude with that exerted by a molecule of dextrose plus a molecule of levulose.—G. F. M.

Polysaccharides. I. Methylation of starch. P. Karrer. *Helv. Chim. Acta*, 1920, 3, 620—625.

THREE products obtained by methylating starch contained respectively 1, about 1.5, and 2 methoxy groups per $C_6H_{10}O_5$; they did not reduce Fehling's solution and yielded respectively reddish-violet, brownish-yellow, and almost imperceptibly yellow colorations with iodine. Preliminary molecular weight determinations on the third preparation indicated the value 1000—2000. (Cf. J.C.S., Dec.) —T. H. P.

PATENTS.

Adhesive composition and process of making same. W. Alexander. U.S.P. 1,337,382, 20.4.20. Appt., 13.3.17.

AN adhesive composed of about equal weights of calcium chloride and dextrinised starch substantially free from glucose, shows no tackiness on its exposed surface when cold.

Charcoal. G.P. 307,761. See IIa.

Acetates. U.S.P. 1,338,040. See VII.

Yeast. G.P. 324,865. See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Diastatic preparations; Technical method for the determination of the saccharogenic power of —. K. Oshima. *J. Ind. Eng. Chem.*, 1920, 12, 991—993.

TEN c.c. of a solution of the sample is added to 100 c.c. of 2% soluble starch solution heated previously to 40° C.; the mixture is kept at this temperature for 30 mins., and 10 c.c. of N/5 sodium hydroxide solution is then added. Graduated quantities of this mixture are added to a series of test-tubes each containing 5 c.c. of Fehling's solution, the tubes are heated in a boiling water-bath for 10 mins., and the smallest quantity of the mixture required to reduce the Fehling's solution is noted. Formulæ and tables are given for calculating the results obtained into Lintner values.

—W. P. S.

Saccharase (invertase) preparations; Preparation of highly active —. H. von Euler and O. Svanberg. *Z. physiol. Chem.*, 1919, 167, 269—314.

VARIOUS highly active preparations of invertase were obtained from autolysed yeast by means of precipitation with alcohol. By quantitative estimations of the activity of the different preparations it was found that about two-thirds of the total invertase of the autolysed yeast remains in the pasty part after the juice has been filtered off. The authors calculate from the relative activities of one of their purest preparations and of dried yeast that their preparation of invertase forms about 1/100 part by weight of the total dry matter of the cell. Allowing for the moisture of the fresh yeast cell and for the invertase retained by the pasty portion of the

autolysed yeast, the active preparation would therefore form 1/900 part of the fresh yeast from which it was prepared. The authors at the same time admit that their best preparation is by no means pure.—S. S. Z.

Invertase action; Theory of —. I. Michaelis and M. Rothstein. *Biochem. Zeits.*, 1920, 110, 217—231.

INVERTASE combines with 1 mol. of sucrose and forms acid having a dissociation constant 3×10^{-7} . This acid dissociates in solution. The undissociated molecules of the acid decompose spontaneously into the products of the enzymic degradation. The anions, on the other hand, are stable. This theory explains the influence of the reaction on the action of the enzyme within certain limits of hydrogen ion concentration.—S. S. Z.

Enzymes; Influence of temperature on — especially on rennet and pepsin. A. König. *Biochem. Zeits.*, 1920, 110, 266—287.

RENNET and thrombin are more stable against high temperatures in concentrated solutions and in glycerol than in dilute solutions. The inactivation of rennet by heat may be delayed by increasing the quantity of milk, as the latter contains protective substances. Increase in temperature up to a certain limit accelerates the action of rennet. The inactivating influence becomes evident only at 45° C. The inactivation of pepsin and diastase also only becomes apparent at 45° C. No difference as regards inactivation by heat is observed in concentrated and in dilute solutions of pepsin. The different behaviour of pepsin and rennet as regards inactivation by heat supports the theory that they are different enzymes.—S. S. Z.

Yeast nutrition and fermentation. Does development of yeast occur without fermentation of sugar? T. Bokorny. *Centr. Bakt.*, 1920, 11, 50, 23—33.

THE author finds that it is possible for considerable increase of yeast to occur in solutions containing no trace of sugar. Besides the ordinary fermentable sugars, rhamnose, arabinose, lactose, peptone, asparagine and other amides, tartaric, acetic, and citric acids and, under certain conditions, glycerol are all capable of supplying yeast with the carbon necessary for its growth. (Cf. J.C.S., Dec.) —T. H. P.

Alcoholic fermentation. Effect of pyruvates, aldehydes and Methylene Blue on fermentation of glucose [dextrose] by yeast juice and zym in presence of phosphate. A. Harden and F. R. Henley. *Biochem. J.*, 1920, 14, 642—653.

EXPERIMENTAL evidence is brought forward to show that aldehydes and pyruvates act as hydrogen acceptors. According to the pyruvic acid theory, the final stage of the alcoholic fermentation of sugar is the reduction of acetaldehyde (produced by decompositions of pyruvic acid), a reaction which proceeds so rapidly that only an extremely small concentration of aldehyde is present during normal fermentation. Further, the production of the pyruvic acid from sugar appears only to be possible when some acceptor for hydrogen is present. Some such acceptor is probably more or less rapidly formed and reduced during the period of delay which follows on the addition of phosphate, this process being accompanied by a corresponding increase in the formation of pyruvic acid, until sufficient of this is being produced to provide the amount of acetaldehyde necessary for the maximum effect. When, however, easily reducible aldehydes or Methylene Blue are added, these act as acceptors and a much more rapid attainment of the maximum becomes possible.—J. C. D.

Typhus-paratyphus group; Decomposition of pentoses by bacteria of the ——. W. Stern. Centr. Bakt., 1918, I., 82, 49—56.

Typhus bacilli decompose xylose, but not arabinose. Paratyphus A bacilli decompose arabinose with liberation of gas, but they do not attack xylose; paratyphus B bacilli, however, decompose both arabinose and xylose, a reducing action and formation of gas taking place in most cases. This behaviour towards pentoses is absolutely characteristic and furnishes a valuable means of differentiating typhus and paratyphus bacteria.

—T. H. P.

Alcohol; Detection of denatured — in spirits. P. Hasse. Pharm. Zentralh., 1920, 61, 613—616.

THE method depends on the detection of acetone and pyridine which are contained in German denatured alcohol. Ten c.c. of the spirit (e.g., brandy) containing not more than 50% of alcohol is acidified with sulphuric acid and 5 c.c. is distilled; 0.5 c.c. of the distillate is then treated with 1 c.c. of ammonium sulphate solution (ammonium sulphate, 30 g., water 45, and concentrated ammonia, 10 c.c.) and 2 drops of sodium nitroprusside solution (sodium nitroprusside, 1 g., water, 4 c.c.). A violet-red coloration develops within 30 mins. if as little as 0.01% of acetone is present. To detect pyridine, the distillation residue is rendered slightly alkaline and its odour observed. The alkaline mixture is then distilled, and 1 c.c. of distillate collected and treated with 0.3 c.c. of 2% mercuric chloride solution. A white crystalline precipitate forms gradually according to the amount of pyridine present. On applying the test to brandy to which denatured alcohol containing 0.5% of pyridine (4 drops to 100 c.c. of brandy) had been added the precipitate appeared after about 20 hrs.; with larger quantities of the denatured alcohol the precipitate appears much sooner.—W. P. S.

Inversion of sucrose. Jones and Lewis. See XVII.

Yeast fermentation. Masters and Maughan. See XIXA.

PATENTS.

Malt; Method and apparatus for grinding —. H. Schott. E.P. 147,072, 6.7.20. Conv., 25.6.19.

A THREE-passage mill with only three rollers is claimed. The malt is crushed between the first and second rollers, separated into groats and spelt by sifting, and these are ground separately but simultaneously between the second and third rollers. The groats between the middle portions and the spelt between the two end portions of the rollers.

Yeast; Manufacture of — using waste beetroot pressings and other expressed saccharine liquids. O. Reinke. G.P. 324,865, 1.11.18.

THE saccharine liquid used for cultivation of the yeast is enriched, after pitching, with inorganic nutrient constituents by the addition of sludge from the refining of sugar, or a solution obtained by treating such sludge with hydrochloric acid until an acid phosphate reaction is obtained.—C. A. M.

XIXA.—FOODS.

Flours; Action of hydrogen peroxide on —. Marion. Comptes rend., 1920, 171, 804—806.

ALL flours contain a catalase, in amount varying with the percentage extraction, the offals having the highest content. By measuring the volume of oxygen liberated in a given time from a given

amount of hydrogen peroxide by a given weight of flour, an idea of the amount of extraction of the flour can be obtained. The method is more accurate and more rapid than the ordinary chemical methods.—W. G.

Bread-making properties of flour; Effect of certain organic substances on the — and on the fermentation of yeast. H. Masters and M. Maughan. Biochem. J., 1920, 14, 586—602.

THE amount of fermentation produced by yeast with sugar and water and in the dough varied with the age of the yeast. The maximum effect was obtained on the second or third day after the yeast had been compressed. The ratio of fermentation of the yeast with sugar and water to the fermentation in the dough was approximately constant. The maximum increase in volume on baking was obtained, under the conditions of the experiments, when the dough was allowed to rise for 40 mins., but this factor may vary with the proportion of the different ingredients used. The acidity of the dough increased with the time of fermentation, and the acidity of the bread decreased with the moisture content. The addition of lime-water to bread does not appear to prevent the production of acid, but it neutralises any acid formed; in the presence of lime-water a smaller loaf was obtained. On the addition of fresh ox-serum the maximum increase in the size of the loaf was produced with 1 pt. of serum to 100 pts. of flour. The power of the serum to increase the size of the loaf diminished rapidly on keeping, but was not destroyed by heating to 50°—60° C. With phosphates the best results were obtained when monosodium dihydrogen phosphate was added in the proportion of 1 pt. of phosphate to 200 pts. of flour. Neither phosphate nor serum produced any increase in the size of the loaf when the bread was raised with a chemical raising agent. Raw potato, cooked potato, and potato flour, when substituted for an equivalent amount of wheat flour, diminished the expansion of the dough on fermentation, but both raw and cooked potato gave a larger loaf, the increase being greater with cooked than with raw potato.—J. C. D.

Milk; Limitations of the reductase test for determining the quality of —. O. Rahn. Milchw. Zentr., 1920, 49, 287—290.

WHILST the Methylene Blue reduction test has a certain value and is better than no test at all, the results obtained by it show many abnormalities, and the information yielded by the test is in no way as reliable as that given by a chemical and bacteriological examination.—W. P. S.

Tripeptide from casein which contains tryptophan. S. Fränkel and E. Nassau. Biochem. Zeits., 1920, 110, 287—299.

A TRIPEPTIDE containing 2 mols. of tryptophan and 1 mol. of alanine was obtained from the products of hydrolysis of casein with potassium hydroxide. (Cf. J.C.S., Dec.)—S. S. Z.

Proteins; Free amino groups of the —. I. S. Edlbacher. Z. physiol. Chem., 1919, 107, 52—73.

THE N-methyl values of proteins of different origin and composition such as gelatin, casein, globin, Bence-Jones protein, edestin, and pumpkin globulin are of the same order. Clupeine sulphate and sturine sulphate also show similar N-methyl values, while esocin sulphate and scombrin sulphate are not methylated at all. Gelatin hydrolysed with alkali for $\frac{1}{2}$ hr. gives a higher N-methyl value than unhydrolysed gelatin. On hydrolysing gelatin with acid the ratio N-methyl value/"formol" value falls after the first $\frac{1}{2}$ hr. of hydrolysis to about a half of the original value, but subsequently remains constant. The same fall in the ratio of the two

values is observed when gelatin and casein and clupeme sulphate are hydrolysed by tryptic digestion.—S. S. Z.

Proteins; Ion series and the physical properties of —. J. Loeb. *J. Gen. Physiol.*, 1920, 3, 85—106.

THE author deals fully with the influence of acids and alkalis on the physical properties of proteins such as gelatin and egg albumin. (*Cf. J.C.S., Dec.*) —J. C. D.

Meats; Utilisation of kid, rabbit, horse and seal — as food. A. D. Holmes and H. J. Denel, jun. *J. Ind. Eng. Chem.*, 1920, 12, 975—977.

THE digestibility of the four meats mentioned was proved to be essentially the same as that of other and better-known meats.—W. P. S.

Oat grain and straw; Composition and properties of —. R. A. Berry. *J. Agric. Sci.*, 1920, 10, 359—414.

A VERY detailed study of the factors affecting the composition and properties of oat grain with a briefer study of the straw. The moisture content of the grain varies considerably with the locality in which it is grown, the length of time after threshing, and the hygrometric condition of the atmosphere. Most of the fatty substance in the grain is present in the aleurone layer, only about 11—12% being found in the germ. The ether extract always contains some finely divided solid matter in suspension, varying in amount from 0.25 to 6% of the total. Free fatty acids, whilst almost absent in the oil from freshly ground kernels, rapidly increase if the meal is kept. Drying the grain, as is done in the manufacture of oatmeal, diminishes the rate of hydrolysis of the fat. In the growing oat, under the soil and climatic conditions at Kilmarneck, the absorption of nitrogen and potassium from the soil is practically complete by the time the kernel begins to develop, whilst the absorption of phosphorus and total mineral matter and the accumulation of dry matter continues until the plant is mature. Migration of nitrogen, phosphorus, and potassium compounds from the straw, husk, and chaff into the kernel proceeds until maturity is reached. The component grains in a spikelet vary in weight, proportion of husk, and chemical composition; on an average the larger grains are richer in oil and nitrogen. The compositions of the kernel and of the grain of a large number of varieties of oats are tabulated. In a particular group in the scheme of tabulation a connexion is shown to exist between the weight of the kernel and other characters of the grain. Thus in the case of white grains with a thin husk, as the kernel increased in weight, the proportion of husk decreased, the oil and fibre diminished, while the carbohydrates, the yield of grain per acre and the proportion of grain to grain and straw increased. The "meal power" of oats calculated from the "kiln dried" grain varied in proportion to the amount of kernel in the grain. Oatmeal from Scotch oats contained a larger proportion of water, nitrogen, and ash than did an inferior sample from Plate oats. Yield of grain per acre is associated directly with the average size of individual grains. The larger grain gives the greater yield, weight per bushel and proportion of kernel. The production of straw varies in the opposite direction to that of the yield of grain. Seasonal changes and application of fertilisers produced comparatively little change in the composition of the grain. Seed sown in January yielded grain, on an average, heavier and containing less husk than later sown seed. Locality, including elevation, climatic, and soil conditions, was the cause of the greatest modification in the characters of the grain. Compared with home-grown oats,

imported oats arriving in Glasgow had a grain which was drier, smaller, contained a thicker husk, was richer in nitrogen and oil, and had a lower weight per bushel. The kernel contained more fibre. Of the imported oats, the grain from Manitoba was the larger and heavier per bushel. —W. G.

Moisture content of cereals; Automatic indicator of the —. M. Chopin. *Comptes rend.*, 1920, 171, 860—862.

AN automatic drying apparatus is figured and described. The grain is measured, not weighed, and its passage through a vertical electrically-heated tube of known capacity is controlled by valves. The moisture driven off is led to a condenser and is collected and measured. By means of suitable mechanical devices the process can be made continuous.—W. G.

Straw; Digestibility of — after treatment with soda. W. Godden. *J. Agric. Sci.*, 1920, 10, 437—456.

WHEN chopped oat straw is soaked overnight in a 1.5% solution of sodium hydroxide and then steamed the next morning for 1 hr. in a farm steamer, a product is obtained which is practically neutral and may be fed as such or after washing with water. By this treatment the digestibility (for sheep) of the organic matter of the straw is increased from 47.3% to 73.7%, of the nitrogen-free extractives from 39.6% to 62.9%, and of the crude fibre from 60.1% to 87.4%. For maintenance purposes, as estimated by the proportion usefully digested, the dry matter of the treated straw has roughly 1½ times the value of the original dry matter, whilst for production purposes, measured by the proportion available after digestion for producing growth, its value is nearly double. The loss of dry matter involved in the treatment amounts to 20%, or if the straw is washed after treatment to 33.5%. Allowing for this, there is still a clear gain in efficiency of about 20% for maintenance purposes and 45% for production purposes if the treated straw is fed without washing.—W. G.

Peat moss; Digestibility of — after treatment with acid. W. Godden. *J. Agric. Sci.*, 1920, 10, 457—459.

A PROPRIETARY article prepared by subjecting finely shredded peat moss to the action of gaseous hydrogen chloride, and subsequently expelling the latter from the residual product, when examined as to its digestibility and feeding value, gave results indicating that it can only be classed as a low-grade fodder.—W. G.

Fruit; Chemical method for the detection of a prior frozen condition in —. W. M. Dehn and M. C. Taylor. *J. Ind. Eng. Chem.*, 1920, 12, 977—979.

WHEN fruit is frozen the sucrose present is converted rapidly into invert sugar as a result of the destruction of the membranes or tissue and the free mingling of the sucrose with inverting acids and invertase. If, therefore, the ratio of invert sugar to sucrose be determined in a fruit before and after freezing (in the laboratory) the data obtained will afford evidence as to whether or not the fruit has been frozen previously. For example, a sound apple was divided into two parts, A and B; the former was analysed directly and the latter after being frozen (in a mixture of ice and salt) for 16 hrs., thawed, and left standing for 24 hrs. The results obtained were:—

	Invert sugar.	Sucrose.	Ratio.	Percentage inversion.
A ..	0.1360	0.1073	100 : 79.1	37.47
B ..	0.2285	0.0863	100 : 37.7	

The percentage inversion is calculated from the formula: $10000(S-s) \div 100s + 1.0528s$, where the value $79.1 = S$, $37.7 = s$, and 1.052 is the ratio of molecular

weights of 2 mols. of hexoses to 1 mol. of sucrose. In the case of peaches the percentage inversion value is 8.93—12.00. This percentage inversion value decreases when the fruit has been frozen previously.—W. P. S.

Vitamins; Nomenclature of —. J. C. Drummond. *Biochem. J.*, 1920, 14, 660.

It is proposed to drop the final "e" of Funks' word *vitamine*. This removes most of the objections to the original term and classifies the substances as of undefined constitution. The various vitamins are conveniently referred to as vitamin A, B, C, etc. —J. C. D.

Vitamin A; Technique of feeding tests on —. J. C. Drummond and K. H. Coward. *Biochem. J.*, 1920, 14, 661—664.

THE method employed by the authors for routine testing is described. Great emphasis is laid on the purity of the basal diet with regard to the A vitamin.—J. C. D.

Vitamin A; Nuts as source of —. K. H. Coward and J. C. Drummond. *Biochem. J.*, 1920, 14, 665—667.

THE common varieties of edible nuts appear to be of little value as sources of the fat-soluble vitamin A.—J. C. D.

Vitamin A; Value of animal and vegetable oils and fats as sources of —. J. C. Drummond and K. H. Coward. *Biochem. J.*, 1920, 14, 668—677.

No hard and fast line can be drawn between these two classes of oils and fats when their value as sources of the fat-soluble vitamin A is considered. Certain vegetable oils, as for example palm and yellow maize oils, are in the crude condition good sources of the vitamin. The presence of yellow lipochrome pigments, formerly believed to be an indication of the presence of the vitamin, is now found unreliable for this purpose. The diet of an animal is an important factor governing the food value of the fat derived from that animal. Both animal and vegetable oils and fats tend to lose vitamin A on refining.—J. C. D.

Enzymes. König. See XVIII.

Phosphatides. Fritsch. See XX.

Butter fat. Reilly and Hickinbottom. See XXIII.

Proteins. Fearon. See XXIII.

PATENTS.

Preserving potatoes and the like vegetable substances; Process for —. Wanquier et Cie. E.P. 137,808, 30.6.19. Conv., 15.1.19. (Cf. E.P. 122,422; J., 1920, 382 A.)

VEGETABLE material is scraped, shredded, or otherwise comminuted and deprived of its moisture in a perforated drum centrifuge, filter-press, or strainer. The liquid obtained is passed through a non-perforated drum centrifuge to remove suspended matter, or is allowed to settle. The solids obtained in the second operation are added to those obtained in the first, and the whole is desiccated.—J. H. J.

Milk-fat; Manufacture of —. E. B. Phelps, A. F. Stevenson, and J. C. Baker, Assrs. to A. W. Johnston. U.S.P. 1,354,683, 5.10.20. Appl., 26.6.19.

MILK is separated and the cream washed first with water and then with acidulated water to render the emulsion unstable and the fat particles capable of coalescence. The fat and acid wash water are then separated.—J. H. J.

Protein and phosphate of calcium and magnesium from acid yeast waters; Process of recovering —. A. Giesecke. U.S.P. 1,354,822, 5.10.20. Appl., 10.3.19.

THE acid waste waters from corn (maize) steeping are partially neutralised, with the result that a combination of protein and calcium and magnesium phosphates is precipitated.—J. H. J.

Bran; Production of albumin and fat suitable for fodder from —. Versuchs- und Lehranstalt für Brauerei in Berlin. G.P. 324,122, 9.5.18.

IN order to liberate the albumin and fat from the aleurone cells in bran, it is boiled for about 5 mins. with sulphuric acid (5%) or hydrochloric acid (20%) or with alkali, under which treatment the cell-walls swell, after which slight pressure suffices to burst them.—W. J. W.

Fodder; Manufacture of — from straw. Veredelungsges. für Nahrungs u. Futtermittel. G.P. 324,648, 19.5.18. Addn. to 305,641 (J., 1919, 789A).

THE constituents dissolved from straw which has been treated as described in the original patent are precipitated, and the whole mass, freed from salts, is used as fodder.—C. A. M.

Straw or similar material; Treatment of — for fodder production. Veredelungsges. für Nahrungs- und Futtermittel. G.P. 324,893, 23.10.18. Addn. to 305,641 (J., 1919, 789 A).

IN the process described in the original patent, deep digesting vessels are used, and dilute alkaline liquors are employed, to admit of the complete immersion of the straw without undue consumption of alkali. For 1 pt. of straw, 16 pts. of 1% caustic soda liquor is used, the waste liquor being regenerated by addition of 0.06 pt. of sodium hydroxide. The consumption of sodium hydroxide after four treatments amounts to 8% of the straw.—W. J. W.

Fodder containing calcium chloride; Manufacture of — from straw etc. O. Demisch. G.P. 324,828, 16.5.18.

CHOPPED straw etc. is treated with hydrochloric acid and neutralised with calcium carbonate, and the mass dried by heat until suitable for grinding, and stored for 2—3 months. The dry product should contain about 80 g. CaCl₂ per kg.—C. A. M.

XIXB.—WATER PURIFICATION; SANITATION.

Waters; Relationship of hydrogen ion concentration to carbon dioxide content in natural —. R. E. Greenfield and G. C. Baker. *J. Ind. Eng. Chem.*, 1920, 12, 989—991.

FAIRLY accurate calculations of the hydrogen ion concentration of natural waters can be made from the simple mass law equation of the primary ionisation of carbonic acid, $(H^+)(HCO_3^-)/(H_2CO_3 + CO_2) = 3.0 \times 10^{-7}$ (Landolt and Börnstein's formula) or $(H^+)(CO_3^{--})/(HCO_3^-) = 6.0 \times 10^{-11}$ (Auerbach and Pick's formula). When carbon dioxide and bicarbonate are determined and expressed in terms of c.c. of CO₂ per litre, $(H^+) = 3.5 \times 10^{-7} CO_2 / (HCO_3^-) = 1 \times 10^{-8}$.—W. P. S.

Water softening with permutite. R. Mezger. *J. Gasbeleucht.*, 1920, 63, 644—648.

A SOLUTION of magnesium chloride corresponding to a hardness of 18° was passed through a tall cylinder of 1.2 l. capacity filled with sodium permutite at the rate of 1.1 l. per hr. for 100 hrs., and the filtrate was tested for hardness. At first all the magnesia was retained, but after 25 hrs. it began to come through, increasing rapidly at first

and then more slowly, until after 70 hrs. the filtrate had regained the original hardness. The filter then contained magnesium permittite and was used for the treatment of a solution of calcium chloride of 21° hardness. From the first magnesia came through in gradually decreasing quantity, until after 70 hrs. it had all been removed. Calcium did not appear in the filtrate until 18 hrs. had elapsed, after which it came through in increasing amounts until the original hardness was reached. The filter was then reconverted to sodium permittite and equivalent amounts of magnesia and lime were applied together to the filter. Magnesia began to come through after 26 hours and lime after 51 hours. Similar results were obtained with Stuttgart water containing lime and magnesia hardness. The experiments show that it would be possible to remove magnesia hardness with certainty by this method and thus prevent the formation of boiler-stone, which consists of magnesium carbonate, magnesium hydroxide, and calcium carbonate.—J. H. J.

Disinfection processes; Specific —. II. *Action of salts and ions on bacteria*. P. Eisenberg. *Centr. Bakt.*, 1918, I., 82, 69—208.

EXTENSIVE experiments on the action of salts on gram-positive and gram-negative bacterial species show that the toxicity of salts may be regarded principally as an additive function of the toxicities of the component ions, although purely molecular actions are not to be excluded. In evaluating antiseptics it is necessary to employ a number of representative bacterial species. (*Cf.* J.C.S., Dec.)

T. H. P.

Disinfecting values of the three isomeric cresols. F. Diithorn. *Centr. Bakt.*, 1918, I., 82, 483—491.

m-CRESOL outstrips its two isomerides in germicidal power, which is not very marked with bacterial suspensions in salt solution and bouillon, but is considerable with liquids containing proteins. Of the two other isomerides, the ortho-compound possibly has a slightly greater action than the para-compound. In practice 2—2.5% solutions of the mixed isomerides are used and the differences observed are then of little importance.—T. H. P.

Toxicity [to micro-organisms] and chemical potential. W. L. Miller. *J. Phys. Chem.*, 1920, 24, 562—569.

THE toxicity of a solution containing phenol and an indifferent salt depends primarily on the chemical potential of the phenol in the solution. Two solutions have the same toxicity when they are in equilibrium with the same solution of phenol in an immiscible solvent such as toluene or kerosene. Complications may arise from the toxicity of the salts themselves, or in dilute solutions from the plasmolysis of the cell, independently of the toxicity of the solutions employed. One or two individual cases do not fall under these heads and need further study. The observation of Paul and Krönig that solutions of mercuric chloride in aqueous alcohol show a maximum of toxicity when the ratio of alcohol to water in the solution is 1 to 3 affords another illustration of the principle, since Laird has shown that the solubility of mercuric chloride in aqueous alcohol passes through a minimum at the same ratio.—J. R. P.

Toxicity towards anthrax and staphylococcus of solutions containing phenol and sodium chloride. J. S. Lemon. *J. Phys. Chem.*, 1920, 24, 570—584.

EXPERIMENTS with anthrax spores showed that the increased toxicity of phenol observed when salt is added to its solution is in accordance with the assumption that two solutions of phenol, with or without salt, are equally toxic if their compositions

are such that both would be in equilibrium with the same solution of phenol in toluene (*cf. supra*). Experiments with staphylococcus, in which lower concentrations of phenol were used, showed that while the assumption is in fair accord with the behaviour of 0.80% phenol, in the case of 0.60% phenol the chemically equivalent solution containing salts is much less toxic; 0.70% phenol occupies an intermediate position.—J. R. P.

Iodine; Use of — for disinfecting the skin. J. Seedorf. *Act. Chirurg. Skand.*, 1920, 52, 436—483. *Chem. Zentr.*, 1920, 91, IV., 516.

IX experiments upon the sterilising action of iodine upon liquid cultures of *S. pyogenes aureus* and tetanus bacilli, the best results were obtained by the use of a solution of iodine (up to a maximum of 1%) in 50% propyl alcohol. A solution of iodine in 96% alcohol also gave good results, whilst aqueous and ethereal solutions were nearly as effective, but a solution in benzene had only a slight sterilising action.—C. A. M.

Wood; Regularities in the preservation of —. *Poisoning action of inorganic compounds (salts) on fungi*. F. Moll. *Centr. Bakt.*, 1920, II., 51, 257—279.

THE results of experiments on *Penicillium glaucum* show that the poisonous action of salts is an additive property of the ions, the poisonous ions arranged in the order of diminishing activity, being: mercury, silver, cadmium, cyanogen, copper, zinc, iron, cobalt, chromium, fluorine. Most acid ions and the ions of the alkali and alkaline-earth metals, magnesium, and aluminium may be regarded as inactive in this respect. (*Cf.* J.C.S., Dec.)

—T. H. P.

“*Pickering*” {*fungicidal*} *sprays [for plants]*. F. C. Cook. *J. Franklin Inst.*, 1926, 190, 571.

“*PICKERING*” sprays prepared by mixing dilute solutions of copper sulphate with saturated lime-water (J., 1912, 196) were twice as effective upon potato plants as the ordinary Bordeaux mixture, per unit of copper present. The sprays adhered to the leaves equally well and had no injurious effect upon hardy foliage. Satisfactory results were also obtained with a spray in which the lime-water was replaced by baryta-water.—C. A. M.

Copper; Non-toxicity of — for moulds in general and for mildew in particular. G. and G. Villedieu. *Comptes rend.*, 1920, 171, 737—739.

IT was found that copper compounds (sulphate, hydrated oxide) in the presence of citric or tartaric acid are not toxic for *Peronospora* when grown on nutrient agar.—W. G.

Tobacco and other vegetable substances; Conversion of air into a lethal mixture of gases by storage of —. R. C. Frederick. *J. Hyg.*, 1920, 19, 205—207.

SAMPLES of air drawn from a storeroom containing a large quantity of tobacco in a mouldy condition showed an abnormally high content of carbon dioxide, *viz.*, 2.27%. Experiments in which samples of tobacco of varying physical condition were kept in bottles with vaselined stoppers secured by rubber bands showed that wet tobacco kept in an unventilated store is liable to convert the air into a lethal mixture of gases; mouldiness is not a causative agent, but accelerates the reaction, and heat increases the decomposition. For example, in one experiment the air from a bottle in which wet mouldy tobacco had been kept for 18 days at 12° C. and 37° C. contained respectively 49.95 and 39.85% of CO₂ and 1.021 and 0.29 O₂. With dry tobacco containing less than 10% of moisture a

dangerous amount of carbon dioxide was not formed even after 32 days. The effect is independent of materials added to tobacco in course of manufacture. It was further found that wet hay and potatoes which had been inoculated with mould were deleterious. After 18 days' storage at 12° and 37°, the air from the sample of hay contained 21.92 and 24.83% CO₂, and 0.25 and 0.26% O₂, whilst that from potatoes after 40 days at 12° C. indicated 21.21% CO₂ and 0.21% O₂. In no instance was carbon monoxide or other combustible gas generated.—W. J. W.

Carbon tetrachloride fire extinguishers; Poisonous gases from —. A. C. Fieldner, S. H. Katz, S. P. Kinney, and E. S. Longfellow. J. Franklin Inst., 1920, 190, 513–565.

As a result of a fatal accident from the use of a carbon tetrachloride fire extinguisher in a confined space, experiments were made with pure carbon tetrachloride and the liquids contained in fire extinguishers. When the liquid was poured on to a fire or hot iron in a closed chamber, or the vapour was passed, together with air, through an iron or quartz tube heated to 600°–800° C., toxic quantities of phosgene (12.3–13.8%) and chlorine (12–20%) and smaller amounts of hydrogen chloride (5.6–11.9%) were formed; the figures represent percentages of the original carbon tetrachloride present in the decomposition products. In some of the tube experiments half of the carbon tetrachloride was decomposed. The presence of turpentine reduced the amount of chlorine liberated from the carbon tetrachloride, but had little, if any, inhibiting effect on the phosgene. It is recommended that carbon tetrachloride fire extinguishers should not be used under conditions where the fumes would be breathed. Efficient protection is afforded by the Army (U.S.A.) type of gas mask. (*Cf.* J.C.S., Dec.) —C. A. M.

Typhus and paratyphus bacteria. Stern. See XVIII.

PATENTS.

Softening water; Process of and apparatus for —. T. R. Duggan, Assr. to The Permutit Co. U.S.P. 1,354,604, 5.10.20. Appl., 26.9.17.

In softening water by filtration through a bed of base-exchanging material contained in a closed filter, occasional sudden pulsations are communicated to the water in the filter which are sufficiently energetic to cause a disturbance of the base-exchanging material.—J. H. J.

Filtering compound. L. W. Shull, Assr. to Allen Filter Co. U.S.P., 1,336,591, 13.4.20. Appl., 1.11.16.

FILTERS for removing germs from water or blood serum are made from a mixture of kieselguhr, asbestos, and a third substance, *e.g.*, whiting, lime, alumina, magnesium chloride, or the like. The materials are intimately mixed to a slip with water, moulded or cast, and fired at a high temperature.

Filter for the purification of water. Radiorex, G.m.b.H., Ver. Magnesia-Co., and E. Hildebrandt, A.-G. G.P. 300,051, 9.11.13.

The filtering medium consists either wholly or partly of a radioactive substance, such as pitchblende. The filter does not become useless by reason of an accumulation of water bacteria, and has a long life.—A. R. P.

Disinfectants; Manufacture of —. A. Hahn and M. Federer. G.P. 322,739, 29.6.11.

DERIVATIVES of laminaric acid are mixed with disin-

fectants in aqueous solution, the proportion of the disinfectant not being appreciably in excess of that required for sterilising the laminaric acid derivative. The sodium-ammonium salt of laminaric acid increases the disinfectant properties of sodium fluoride, mercuric chloride, and formaldehyde.

—W. J. W.

Drying trough. G.P. 323,462. See I.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Opium preparations of the type of "pantopon"; Determination of subsidiary alkaloids and also of narcotine and papaverine in —. E. Anneler. Arch. Pharm., 1920, 258, 130–137.

To determine the total alkaloids other than morphine, 1.5 g. of the mixed hydrochlorides is dissolved in 8 c.c. of water, treated with 90 g. of benzene and 0.5 g. of sodium carbonate, shaken at intervals during $\frac{1}{2}$ hr., and then treated with 5 g. of anhydrous sodium sulphate and 0.5 g. of powdered tragacanth. After the solid matter has settled the liquid is filtered, and an aliquot part is evaporated to dryness, redissolved in alcohol, and again evaporated. To separate the narcotine from the other bases it is converted into sodium narcotinate. The mixture obtained as above is dissolved in 6 c.c. of benzene, treated with 1 c.c. of alcoholic potassium hydroxide solution, allowed to stand for $\frac{1}{2}$ hr. with frequent shaking, and then the benzene solution is shaken in a separating funnel with successive quantities of sodium hydroxide solution, and the united aqueous extracts are repeatedly shaken with small quantities of chloroform. To regenerate the narcotine the alkaline solution is neutralised with hydrochloric acid, made up to 100 c.c., heated with 3 c.c. of concentrated hydrochloric acid for 20 mins. at 80°–90° C., cooled rapidly, treated with an excess of sodium carbonate solution, and shaken out with benzene or chloroform. To determine the papaverine the combined benzene and chloroform extracts, obtained in separating the narcotine, are evaporated to dryness. The residue is dissolved in 10 c.c. of water and 1 c.c. of hydrochloric acid, filtered, treated with a 2% ammonia solution, drop by drop, and stirred meanwhile, until a slight permanent turbidity appears, and then, after addition of 2 g. of sodium acetate, left to stand for 24 hrs. The papaverine is collected on a filter, redissolved in warm alcohol, and the solution evaporated to dryness.—J. H. L.

Glucosides. VII. Constitution of amygdalin. P. Karrer, C. Nägeli, and L. Lang. Helv. Chim. Acta, 1920, 3, 573–583.

HEPTA-ACETYLCELLOSIDOMANDELIC acid, obtained in small quantities by the interaction of inactive silver mandelate and acetobromocellobiose, is not identical with hepta-acetylamygdalinic acid, hence the corresponding non-acetylated acids are not identical, and the unknown disaccharide of amygdalin cannot be cellobiose; the β -glucosidic nature of the union of the disaccharide in the two cases is shown by the fact that hydrolysis is effected by emulsin in either case. (*Cf.* J.C.S., Dec.)—T. H. P.

Phosphatides; Preparation of — from coloured plant organs. R. Fritsch. Z. physiol. Chem., 1919, 107, 165–176.

VARIOUS plants containing chlorophyll and other plant pigments have been examined for their phosphatide content. Only a small part of the total phosphorus could be traced to the phosphatides of the plants. In the leaves of the maple the phosphatide-phosphorus amounted to 4.78%, in the

leaves of the ash to 3.46% of the total phosphorus. No calcium inosinate was found in the green assimilating organs. In order to obtain phosphatides with the theoretical content of phosphorus, large quantities of tissue must be employed. In grass stored in silos the phosphatides are almost entirely decomposed.—S. S. Z.

Guanidine; Preparation of — by the interaction of dicyanodiamide and ammonium thiocyanate. E. A. Werner and J. Bell. Chem. Soc. Trans., 1920, 117, 1133–1136.

PURE guanidine thiocyanate was readily obtained in 90% yield by melting together dicyanodiamide (43.5 g. of 95.5% purity) and ammonium thiocyanate (76 g.), and maintaining the mixture at 120°C. for about 3½ hrs. Depolymerisation of the dicyanodiamide is the first phase of the reaction, which is expressed by the equation: $C_2H_4N_4 + 2NH_4SCN \rightarrow 2CH_3N_3HSCN$. A subsidiary reaction results in the formation of thioammeline, which is, however, readily separated by digesting the reaction mixture, without previous cooling, with 250 c.c. of water, and filtering, thioammeline (7–8 g.) separating as an amorphous residue. The filtrate, after concentration as far as possible at 100°C., sets to a crystalline mass of guanidine thiocyanate, which may be further purified by recrystallisation from water. —G. F. M.

Salvarsan; Colloidal properties of aqueous solutions of —. Z. Klemensiewicz. Bull. Soc. Chim., 1920, 27, 820–824.

THE viscosity of aqueous solutions of salvarsan depends on the concentration of the solution, the length of time it has been prepared, the temperature at which it has been kept, and the presence of acids or alkalis. (Cf. J.C.S., Dec.)—W. G.

Acetylsalicylic acid; Melting point of —. G. Cappelli. Giorn. Chim. Ind. Appl., 1920, 2, 291–300.

WHEN heated in a capillary tube immersed in a bath, acetylsalicylic acid begins to melt in contact with the walls of the tube at 132°C. and becomes completely transparent only at 135°. (Cf. J., 1919, 157A.)—T. H. P.

Benzyl alcohol; Stability of solutions of —. D. I. Macht and A. T. Shohl. J. Pharm. Exp. Ther., 1920, 16, 61–69.

SOLUTIONS should be preserved in non-soluble glass containers if deterioration is to be prevented. —J. C. D.

Lachrymatory power of irritant substances; Measurement of the —. C. Dufraisse and J. C. Bongrand. Comptes rend., 1920, 171, 817–819.

IF C_1 be the concentration of benzyl bromide, and C_2 that of the substance under examination, required in an atmosphere just to produce an irritant action on the eyes at the end of 30 secs., the lachrymatory power is given by C_1/C_2 , that of benzyl bromide being taken as unity. The following values were obtained: Benzyl iodide 2, bromoacetone 1.8, sym-dibromomethyl oxide 1/4, chloropierin 1/6, chloroacetone 1/8, acrolein 1/10.—W. G.

Carbon bisulphide; Interaction of — with ammonium carbonate. F. A. Gilfillan. J. Amer. Chem. Soc., 1920, 42, 2072–2079.

CARBON bisulphide and ammonium carbonate react at 160°C. to give a mixture of ammonium thiocyanate and thiourea, the equilibrium depending upon the concentration of the reagents, the temperature and duration of heating, and the concentration of the hydrogen sulphide produced in

the reaction. The latter favours the reversion of the thiocyanate to thiourea. The yields of thiourea are much below those obtained by direct heating of ammonium thiocyanate. Ammonium thiocyanate cannot be estimated accurately by the indirect titration method (Krall, Chem. Soc. Trans., 1913, 103, 1385) in the presence of more than 15% of thiourea, but direct titration with silver nitrate may be used with mixtures containing as much as 40% of thiourea. In solutions containing not more than 5 mg. of thiourea per 100 c.c. this substance may be estimated by titration with iodine.—W. G.

Glycerol substitutes (per- and perka-glycerol); Alkali lactates as —. C. Neuberg and E. Rein-furth. Ber., 1920, 53, 1783–1791.

THE technical uses of glycerol, apart from the production of explosives, depend on its viscosity, hygroscopicity, and neutrality; these properties are also exhibited by aqueous solutions of sodium and potassium lactates (per- and perka-glycerol respectively), which have been extensively used by the belligerent forces of the Central Powers and for medical and cosmetic purposes during the late war. Economically, their preparation permits a much better utilisation of the initial material (sugar etc.), since this can be fermented so as to yield 95% and more lactic acid, whilst, on the technical scale, it only gives one-fifth to a quarter of its weight of glycerol; further, for many purposes, the glycerol substitutes can be used in considerably more dilute solution than glycerol itself. The paper contains an extended series of tables and graphs showing the relationship between specific gravity and concentration of aqueous solutions of per- and perka-glycerol at 15°C., the freezing point of solutions of per-glycerol at different concentrations, the comparative freezing point of equimolar solutions of per-glycerol and glycerol, the boiling points of solutions of per-glycerol, the comparative hygroscopicity of solutions of glycerol and per-glycerol at 25°C., and the viscosity of per-glycerol. (Cf. J.C.S., Dec.)—H. W.

Ethylene bromohydrin; Preparation and characterisation of —. J. Read and R. G. Hook. Chem. Soc. Trans., 1920, 117, 1214–1226.

PURE ethylene bromohydrin may be prepared in quantity by the action of ethylene on cold dilute bromine water. Under favourable circumstances the molecular ratio of ethylene bromohydrin to ethylene dibromide formed is 10:1, or even higher in very dilute solutions, but the ratio decreases continuously with increasing concentration of bromohydrin and hydrogen bromide in the solution and also with rise of temperature. The reaction is accelerated by cooling and also by sunlight. Ethylene bromohydrin may be distilled without change *in vacuo*, but at ordinary pressures it decomposes considerably at its boiling point. It has sp. gr. 1.7629 at 20°/12°C., and $n_D^{20} = 1.4915$. It forms with water a mixture of constant b.p. (99.1°C. at 762.4 mm.) at a concentration of 35%. Ethylene bromohydrin is hydrolysed more readily than the chlorohydrin, hydrolysis being perceptible, although extremely slow, in cold aqueous solutions.—G. F. M.

Acetoneglycerol. Fischer and Pfähler. See XII.

PATENTS.

Esters; Apparatus for the manufacture of —. U.S. Industrial Alcohol Co., Assees. of A. A. Backhaus. E.P. (a) 130,969, and (a) 130,970, 23.6.19. Conv., 7.8.18.

CLAIM is made to apparatus for carrying out the process described in E.P. 130,968 (J., 1920, 674A), using sulphuric acid, alcohol, and (a) a dilute organic acid, e.g., vinegar, or (b) a concentrated organic acid, e.g., glacial acetic acid.—L. A. C.

Trimethylacetylsalicylic acid compounds. W. Grüttefen, Assr. to Alien Property Custodian. U.S.P. 1,338,297, 27.4.20. Appl., 27.4.19.

TRIMETHYLACETYSALICYLIC acid (CH_3)₃C.CO.O. C_6H_4 .COOH, white leaflets, m.p. 135° C., is prepared by the interaction of salicylic acid and trimethylacetyl chloride in ether and pyridine. Trimethyl-*m*-cresotic acid, prepared in an analogous manner, melts at 160° C. These acids, both in the free state and as salts, are of value as antirheumatics, analgesics, and diuretics.

Amylodextrin and silicic acid; Manufacture of colloidal preparations of —. Lecinwerk E. Laves. G.P. 323,596, 12.3.19.

SILICIC acid is produced in an aqueous solution of amylo-dextrin by the decomposition of silicates with acids, or by hydrolysis of esters of silicic acid or halogen compounds of silicon. The product is precipitated by alcohol or acetone, and dried at a low temperature. Solutions containing less than 1% of silicic acid may be sterilised without gelatinisation by passing a current of steam through for several hours. The amylo-dextrin compounds, both in the solid state and in sterilised solution, are suitable for therapeutic purposes.—W. J. W.

Amylo-dextrin and calcium phosphate, or calcium sulphate; Manufacture of colloidal preparations of —. Lecinwerk E. Laves. G.P. 323,783. 31.10.18.

CALCIUM phosphate or calcium sulphate is formed by double decomposition in an aqueous solution of amylo-dextrin, or of carbohydrates containing it, the amylo-dextrin compound produced being then separated by precipitation with an organic solvent, or by careful evaporation of the solution *in vacuo* at a low temperature. The calcium phosphate compound consists of tricalcium-phosphate-amylo-dextrin. The products have a therapeutic application.—W. J. W.

Oils, resins, waxes, balsams, and extracts; Method for converting — into dry powders. R. Marcus. G.P. 323,908, 8.10.15.

THE materials are mixed with gelatinous silicic acid, and the mixture is allowed to dry under moderate heat. Smooth powders are thus produced with 1 pt. of silicic acid and 2 pts. of *Oleum rosei*, or 1.4 pts. of protargol, or 2.5 pts. of lemon oil.

—W. J. W.

Glycerophosphate solution; Conversion of — into a crystalline preparation. P. Bergell. G.P. 324,029, 1.4.19.

A CONCENTRATED solution of sodium glycerophosphate is treated with partly dehydrated disodium hydrogen phosphate in such quantity that the water in the solution does not exceed the water of crystallisation removed from the salt. When dried by exposure to the air the mass is a crystalline powder containing 7 mols. of water. It has a pleasant taste, and can be compressed into tablets for therapeutic use.—C. A. M.

Pinacone; Process for the manufacture of —. Farbenfabr. vorm. F. Bayer und Co. G.P. (A) 324,919, 8.5.17 (Addn. to 306,523; J., 1919, 158 A), and (B) 324,920, 13.9.18.

(A) In the process described in G.P. 306,523, the use of cathodes of an alloy of 90% Pb and 10% Sn, or of brass or bronze, yields a product containing pinacone and isopropyl alcohol in a ratio of about 10:1. (B) Acetone is reduced in alkaline solution using graphite electrodes. The product contains 70% of pinacone and 10–15% of isopropyl alcohol.

—L. A. C.

Double compound containing quinine; Process for the manufacture of a —. A. C. Röttinger. G.P. 325,156, 22.11.18.

THE double compound, which has a bitter, acid taste, but does not show the after-taste or bad effects of quinine, is prepared by the interaction of equimolecular proportions of quinine, tartaric acid, and hexamethylenetetramine in alcoholic solution.

—L. A. C.

Phenylated aminohydrindenecarboxylic acid; Process for the manufacture of a —. Chem. Werke Grenzach A.-G. G.P. 325,713, 29.6.18.

THE condensation product from α -hydrindone and isatin, a colourless powder of m.p. above 295° C., is reduced in boiling amyl alcohol with metallic sodium, and the sodium salt produced is subsequently converted to the free phenylated aminohydrindenecarboxylic acid. The crystalline acid, which has therapeutic value, contains 76.43% C and 6.1% H, softens with red coloration and liberation of carbon dioxide at 140° C., and melts at 180° C.; the hydrochloride has m.p. 205° C.

—L. A. C.

N-Methylhexamethylenetetramine; Process for producing salts of —. R. Tschunke, Assr. to The Chemical Foundation, Inc. U.S.P. 1,336,709, 13.4.20. Appl., 19.1.14. Renewed 26.6.19.

SEE G.P. 270,486 and 275,092 of 1913 and First Addn. to F.P. 466,619; J., 1914, 439, 807, 845.

Allyl ester of 2-phenylquinoline-4-carboxylic acid. A. Gams, Assr. to Soc. of Chem. Ind. in Basle. U.S.P. 1,336,952, 13.4.20. Appl., 29.5.19.

SEE E.P. 150,401 of 1919; J., 1920, 704 A.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photographic sensitisers; Studies on —. II. The carbocyanines. W. H. Mills and W. J. Pope. Phot. J., 1920, 60, 253–266.

THE carbocyanines are produced by the condensation of 2 mols. of a quinaldine alkyl iodide with 1 mol. of formaldehyde under the influence of alkalis. The formula adopted for the class assumes the linkage :CH.CH:CH. between the two quinaldine residues, attached at the 2 position in each case, thus: $\text{N(R).C}_6\text{H}_4\text{:CH.CH:CH.C}_6\text{H}_4\text{.N.R.X}$, the simplest member being the 1.1'-dimethyl compound. Sensitol Red (German, Pinacyanol) is 1.1'-diethyl-carbocyanine iodide, and is, so far, the most important dye of the class. Eighteen members of the series are described both as to their chemical properties and as to their sensitising as determined by the method previously described (J., 1920, 468 A). The change from the 1.1'-dimethyl to the 1.1'-diethyl compound is accompanied by a considerable increase in sensitising action; further increase in the weight of the substituent groups in these positions results in a steady decrease of sensitising action. The effect of introducing substituent groups in other positions in the molecule varies with the nature of the group and the position. Generally speaking there is a reduction of sensitising action as compared with the 1.1'-compound, which increases with the size of the substituting groups. Substitution in the 6-6' positions has a slight reducing effect, in the 5-5' positions a strong reducing effect. It is suggested that the distinguishing characteristic of the carbocyanines as compared with the isocyanines (*loc. cit.*) and on which the extension of their sensitising action into the far red depends is not the position of attachment to the

quinaldine residues, but the presence of the linkage :CH.CH:CH. instead of :CH.—B. V. S.

Neutral-wedge photometer; New —, for sensitometry for photographic copying processes, and scientific light-measurement. J. M. Eder. Phot. Korrr., 1919, 56, 244—271.

This is an adaptation of a Goldberg neutral wedge in combination with a special form of millimetre scale. In one form a wedge 9×12 cm. is used having a constant of 0.40137 per cm. and being provided also with colour strips, red, yellow, green, and blue, for the testing of chromatic and panchromatic plates; another form is 16×3 cm. with a constant of 0.30484 per cm., this being chiefly intended for actinometric and similar work. The use of wedges in sensitometry, in photometry, and in actinometry is described and tables are given showing the relative proportions of light absorbed at all points of the wedge, comparing the wedge scale readings with Scheiner scale numbers, giving comparative results obtained by the wedge with Hefner and electric light for speeds of ordinary and colour-sensitive plates, etc. There is also a comparison of the various types of actinometer papers which may be used.—B. V. S.

Eder-Hecht neutral-wedge photometer for sensitometry and light-measurement. II. Magnesium as a secondary standard light source. J. M. Eder. Phot. Korrr., 1920, 57, 1—9, 41—44, 83—86.

THE use of magnesium light as a secondary standard light source is advantageous by reason of its simplicity and its approximation, in colour value, to average daylight. A suitable unit is 2 mg. at a distance of 3 m., which is approximately equivalent to a Hefner-candle-metre-minute. The magnesium, about 3 mm. of ordinary ribbon, is held on the point of a steel needle at the correct distance from the plate to be exposed. The use of the Eder-Hecht photometer (*cf. supra*) with magnesium as the light source is fully described, comparative results with the Hefner lamp for various plates being given as well as sundry tabulated constants of the wedge. A full scheme for testing and evaluating plates and papers with the aid of the wedge is described and also the use of the wedge for evaluating exposures in light-therapy.

—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Nitro-derivatives of toluene. Brady and Williams. See III.

PATENTS.

Ammonium nitrate explosives; Manufacture of —. E. Müller. E.P. 152,199, 2.10.19.

To obtain from mixtures of ammonium nitrate and a hydrocarbon, such as petroleum, explosives which are insensitive to concussion but yet can be readily detonated, the constituents are melted together under a pressure of $\frac{1}{2}$ to 1 atm. Other ingredients may be added. Suitable mixtures are: ammonium nitrate, 94%; petroleum, 6%; ammonium nitrate, 92%; woodmeal, 1%; petroleum, 7%; ammonium nitrate, 95%; paraffin, 5%; ammonium nitrate, 93%; phenol, 7%.—W. J. W.

Propellant powder and process of making same. C. I. B. Henning, Assr. to E. J. du Pont de Nemours and Co. U.S.P. 1,336,463, 13.4.20. Appl., 23.8.19.

A POWDERED metal capable of reducing metallic fouling, e.g., tin or a tin-lead alloy, is incorporated with nitrocellulose powder during the process of manufacture.

Granulated "safety" explosives; Manufacture of —. Oberschlesische Sprengstoff-A.-G. G.P. 298,448, 16.12.16.

EXPLOSIVES of a dusty nature, and which can be detonated only with difficulty, may be converted into granular safety explosives, which can be readily detonated, by mixing them with finely-divided, fusible carbonaceous substances such as naphthalene, resin, or dextrin, and agitating the mixture with steam.—W. J. W.

Chlorate explosives; Manufacture of —. G. A. Krause und Co., A.-G. G.P. 299,630, 23.1.17.

A CHLORATE solution containing in suspension charcoal or other material is evaporated by ejecting it from the evaporating vessel in the form of a spray and directing against the outside of this a stream of gaseous drying agent. An intimately mixed, granular powder is thus obtained without danger.—W. J. W.

[Smokeless powder;] Process for drying [—] by means of hot air. K. Himpel and A. Besler. G.P. 301,607, 3.4.17. Conv., 17.2.16.

THE air used for drying explosives is cooled to a definite temperature, and the condensed solvent is completely separated by mechanical means. The content of condensable vapour in the air can thus be accurately adjusted.—W. J. W.

Nitrocellulose; Manufacture of —. Deutsche Gasglühlicht A.-G. (Auerger.). G.P. 302,494, 1.12.16.

AFTER nitration of the cellulose, the bulk of the waste acid is displaced by sulphuric acid of 55°—60° B. (sp. gr. 1.616—1.712), previously cooled to 0° C. Up to 80—90% of the nitration acid is recovered as waste acid, from which nitric acid of commercial strength is obtainable.—W. J. W.

Pyrotechnics [; Compositions for —]. A. Brock. E.P. 152,529, 3.11.19.

HEXAMETHYLENETETRAMINE, or its salts, is substituted for, or added to, the usual combustibles, such as charcoal, shellac, dextrin, sugar, and oil, in fire-work compositions, specially for indoor displays.

—W. J. W.

XXIII.—ANALYSIS.

Anemometer; Directional hot wire —. J. S. G. Thomas. Phil. Mag., 1920, 40, 640—665.

THE directional hot wire anemometer consists of two fine wires mounted parallel and one behind the other in juxtaposition, transversely to the direction of flow of the gas in the pipe or channel. The wires constitute two arms of a Wheatstone bridge the remaining arms of which are formed of a resistance of 1000 ohms and an arm capable of adjustment. A constant current is maintained in the bridge and the battery terminals are connected through a rheostat with the appropriate ends of the platinum wires so that the maximum heat is developed in these wires. As the velocity of the gas stream to which the heated wires are exposed is gradually increased from zero, the galvanometer deflection increases until a critical value of the gas stream is attained. With further increase of the impressed velocity of the gas stream the galvanometer deflection slowly diminishes; on reversing the direction of flow of the gas current the galvanometer deflection is reversed. The relative sensitiveness of the instrument has been investigated with the wires in different positions, and the connexion between the critical velocity and the heating current and the diameter of the wires is also dealt with. (*Cf. J.*, 1918, 165 r.)—J. F. S.

Analysis; Qualitative chemical — V. Macri.
Boll. Chim. Farm., 1920, 59, 385—389.

The following scheme serves for the detection of the metals in solution. The solution is neutralised with ammonia solution, treated successively with ammonium sulphide and carbonate, and allowed to digest on a water-bath with occasional stirring. A small portion of the liquid is tested with the above reagents (to ensure that they are in sufficient quantity) and with ammonium phosphate; if a precipitate forms, more of the reagent is added. The filtrate or solution is rendered acid with hydrochloric acid and any precipitate tested for As, Sb, Sn, Pt, Au; the filtrate is evaporated to dryness and tested for K, Na, Li. The original phosphate precipitate is washed with water containing hydrogen sulphide and heated with hydrochloric acid, and the residue tested for Ag, Pb, Hg, Cu, Bi, Cd, Ni, and Co; the solution is freed from hydrogen sulphide and boiled with sodium acetate; the precipitate may contain Al, Fe, and Cr, from which the Al is separated by potassium hydroxide. The solution is treated with hydrogen peroxide, when manganese is precipitated, and the filtrate treated with sodium sulphate and alcohol. The precipitate is tested for Ba, Sr, and Ca, and the solution rendered ammoniacal and treated with phosphate; magnesium is precipitated, and zinc is tested for in the solution by means of hydrogen sulphide.

—T. H. P.

Analysis of organic compounds; Use of reduced copper in the elementary — E. Cherbuliez.
Z. anal. Chem., 1920, 59, 297—302.

At a red heat copper containing small proportions of iron or zinc causes appreciable reduction of carbon dioxide to carbon monoxide and is, therefore, unsuitable for use in the determination of hydrogen and carbon in nitrogen compounds by combustion or in the estimation of nitrogen.

—T. H. P.

Bromine; Determination of traces of — in organic matter. A. Damiens. Comptes rend., 1920, 171, 799—802.

The organic matter is dried at 100°—105° C., mixed with 5 pts. of potassium nitrate and 10 pts. of sodium carbonate and heated in a silver crucible to just short of fusion. After cooling, the mass is extracted with water and the solution filtered. In a portion of the filtrate the iodine is estimated either by Labat's method (distillation in presence of iron ammonium alum), or by precipitation as silver iodide, subsequent liberation by chlorine, and colorimetric estimation in chloroform solution. In another portion of the filtrate the whole of the halogens are precipitated as their silver salts, which are afterwards decomposed by zinc and sulphuric acid, the silver being filtered off, and the bromine estimated in the filtrate by the method of Denigès and Chelle (J., 1918, 284A). In the presence of iodide, the iodine is first removed from the last filtrate by Labat's method before the bromine is estimated. Chlorine is estimated by difference from the total halogen content.—W. G.

Distillation in steam; Applications of the method of — [Differentiating butter fat and other fats.] J. Reilly and W. J. Hickinbottom. Sci. Proc. Royal Dublin Soc., 1920, 16, 131—142.

The limitations and possibilities of the distillation method for determining molecular structure and alteration in the state of molecular aggregation are discussed. A method is given for differentiating between butter fat and other fats containing a comparatively high proportion of volatile acids; 100 c.c. of the filtered Reichert-Meissl distillate is neutral-

ised with barium hydroxide solution, the barium is then precipitated by the addition of the requisite amount of sulphuric acid, the mixture diluted to 200 c.c., and distilled from an ordinary round-bottomed flask provided with a steam-jacket. The distillate is collected in 10 g. fractions, which are titrated. The percentage of acid distilling in each fraction is calculated on the acid present in 100 c.c. of the original Reichert-Meissl distillate. Results obtained with typical fats are as follows:—

Weight of oil distillate in g.	Percentage of acid distilling in each fraction.				
	Butter fat.	Palm kernel oil.	Babassu fat.	Coconut oil.	Chemical.
10	12.5	20	25	22	22
20	23.0	35	42	38	38
30	35.5	49	57	53	53
40	42.5	60	70	65	65
50	50.0	68	80	75	75

—W. P. S.

Protein; Adamkiewicz' reaction for — Mechanism of Hopkins-Cole test for tryptophane. W. R. Fearon. Biochem. J., 1920, 14, 548—564.

The author shows that the colour tests mentioned are dependent on the formation of colour condensation products of the aldehyde present in the reagent and tryptophane. A number of these pigments have been isolated. Their properties and chemical characters are fully discussed. (Cf. J.C.S., Dec.)

—J. C. D.

See also pages (A) 777, Nitro compounds (English). 781, Ammonium salts (Van Zijp); Cyanic acid (Fosse); Phosphates and arsenates (Denigès). 791, Antimony sulphide (Van Rossem and Dekker). 793, Sour soils (Comber); Nitrates in soils (Whiting). 794, Calcium cyanamide (Neubauer); Moisture in sugar products (Aikin). 796, Denatured alcohol (Hasse); Flours (Marion); Milk (Rahn). 797, Moisture content of cereals (Chopin). Fruit (Dehn and Taylor). 800, Opium preparations (Anneler). 801, Lachrymatory power (Dufraisse and Bongrand). 803, Photometer (Eder).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

- American Metal Co. Rotary kilns. 32,359. Nov. 16. (U.S., 8.12.19.)
- Avrutik. Separation of liquids and solids. 31,582. Nov. 8.
- Barrett Co. Manufacture of carriers for catalytic material. 32,068. Nov. 12. (U.S., 13.11.19.)
- Bateman. Separators for liquids of different density. 31,575. Nov. 8.
- Beeston. Regenerative furnaces. 33,082. Nov. 24.
- Bing-Werke. Preventing boiling-over of liquids. 32,562. Nov. 18. (Ger., 2.7.19.)
- Beythe. Drying ovens or stoves etc. 32,373. Nov. 17.
- Doherty. Mixing machines. 33,276. Nov. 25.
- Emmens. Settling-tanks, storage tanks, etc. 31,597. Nov. 8.

Hoyle, Centrifugal separators. 33,179. Nov. 24.
Morgan, and Thermal Industrial and Chemical Research Co. Evaporation of liquids which deposit matter during the process or on cooling. 33,312. Nov. 25.

Muchka, Storing, conveying, etc. highly inflammable liquids. 32,337, 32,339, 32,340. Nov. 16. (Austria, 6.11.14, 19.6.16, 30.10.16.)

Muchka, Production of protective gas. 32,338. Nov. 16. (Austria, 21.9.15.)

Muchka, Production of mixtures of nitrogen and carbonic acid deficient in oxygen. 32,341. Nov. 16. (Austria, 3.12.17.)

Nelson, Kilns. 33,309. Nov. 25.

Orr, Rotary crushing and mixing mills etc. 33,517. Nov. 27.

Reid, Furnaces. 33,196. Nov. 24.

Robertson (Power Speciality Co.), Stills. 31,465-8. Nov. 26.

Sears and Twigg, Furnaces. 31,942. Nov. 11.

Skudder, Apparatus for separating powdered minerals etc. suspended in liquid. 33,571. Nov. 27.

Smallwood, Furnaces. 31,747. Nov. 10.

Soderlund, and Techno-Chemical Laboratories, Ltd. Recovery of heat from treated material. 32,127. Nov. 13.

Statincau, Catalytic process for obtaining reactions between a gas and another substance. 32,932. Nov. 22. (Switz., 22.11.19.)

Velten, Treating material by a rising fluid current. 31,616 and 31,870. Nov. 9 and 11. (Ger., 12.12.19 and 18.2.20.)

Wake, Ring pulverising-mills. 32,819. Nov. 20.

COMPLETE SPECIFICATIONS ACCEPTED.

17,451 (1919). Akt. de Norske Saltverker. Preventing formation of gypseous incrustations in evaporators etc. (131,279.) Nov. 17.

22,724 (1919). Woltersdorf, Grinding and disintegrating machines. (132,794.) Nov. 24.

25,153 (1919). Hardingham (Redman), Furnaces. (153,451.) Nov. 17.

25,729 (1919). Bollmann. *See V.*

32,582 (1919). Rosanoff Process Co. Fractionating apparatus. (137,300.) Nov. 17.

1114 (1920). Niessen, Apparatus for boiling and drying organic matter. (137,843.) Nov. 24.

9297 (1920). Lambot, Rocking-furnaces. (141,054.) Dec. 1.

10,205 (1920). Akt. Vaporackumulator, Digesters. (141,708.) Nov. 17.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

Bamber and Parker, Gas-scrubbers. 32,120. Nov. 13.

Bates, Raising sp. gr. and flash-points of liquids. 31,634. Nov. 9. (U.S., 10.11.19.)

Bates, Manufacture of fuel. 32,008. Nov. 12.

Bates, Fuel. 32,262. Nov. 16.

Bates, Liquid fuel. 32,263. Nov. 16.

Bates, Mixed fuels. 33,135 and 33,501. Nov. 24 and 27.

Chandler and others. 33,036-7. *See XXIII.*

Clark and Co., and Tervet, Heat treatment of oils etc. 32,250. Nov. 15.

Cordes and Thiele, Preparation of lubricating and cylinder oils. 32,955. Nov. 12. (Ger., 24.11.19.)

Denington and Perry, Treatment of peat etc. 32,600-1. Nov. 18.

Dixon and Garton, Briquetting coal etc. 31,776. Nov. 10.

Ehrat, Production of petroleum and natural gas. 33,461. Nov. 26.

Emerson, Conversion of hydrocarbon oils. 31,555. Nov. 8. (U.S., 6.5.20.)

Foster, Vertical gas-retort etc. 32,169. Nov. 15.

Gooday, Purifying lubricating oil. 32,679. Nov. 19.

Mawson, Gas-producers. 31,931. Nov. 11.

Muchka, 32,337-33,341. *See I.*

Parr and Pearson, Desulphurising hydrocarbons etc. 32,195. Nov. 15.

Rambush, Gas-producers etc. 33,144. Nov. 26.

Robus, Apparatus for distilling peat. 31,470. Nov. 8.

Wells, Gas-producers. 31,788. Nov. 10.

Wilson, Distilling fuels, shales, etc. 32,458. Nov. 17.

COMPLETE SPECIFICATIONS ACCEPTED.

11,419 (1919). Marks (U.S. Industrial Alcohol Co.), Non-freezing fuel. (153,925.) Dec. 1.

11,774 (1919). Doherty, Coal-carbonising furnaces. (132,489.) Nov. 17.

19,421 (1919). Kormann, Alteration of high-boiling hydrocarbons to low-boiling hydrocarbons. (153,654.) Nov. 24.

19,650 (1919). Edwards, Extracting oil from shale, coal, etc. (153,663.) Nov. 24.

19,702 (1919). Rambush, Removal of sulphur from gases. (153,665.) Nov. 24.

2718 (1920). Avellana, Wood-gas producer. (138,362.) Nov. 17.

3503 (1920). Igranic Electric Co. *See XXIII.*

9676 (1920). De Laval Separator Co. Purifying used mineral oil. (142,059.) Dec. 1.

10,390 (1920). Smith, Purifying mineral oils. (153,844.) Nov. 24.

13,888 (1920). Hulton and Neilson, Refining mineral oils. (153,857.) Nov. 24.

15,778 (1920). Lowenstein, Irinyi, and Kayser, Apparatus for low temperature distillation of coal, shale, etc. (144,712.) Dec. 1.

16,757 (1920). Meister, Lucius, u. Brüning, Manufacture of methane. (146,110.) Nov. 21.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

Atack and Soutar, Manufacture of chloroamino-anthraquinone. 31,615. Nov. 9.

Atack, Oxidation of aromatic hydrocarbons. 31,971. Nov. 12.

Atack, Manufacture of anthraquinone. 33,374. Nov. 26.

British Dyestuffs Corp., Frank, Gibson, and Robinson, Manufacture of a chlorinated naphthalene derivative. 32,241. Nov. 15.

Dawson, Purification of anthraquinone. 32,286. Nov. 16.

Kolshorn, Manufacture of derivatives of *p*-aminophenol and of its *o*-alkyl ethers. 33,206-7. Nov. 21. (Ger., 12.12.19.)

Parr and Pearson. 32,195. *See II.*

Wilson, Distillation of tar and oils. 32,391. Nov. 17.

COMPLETE SPECIFICATIONS ACCEPTED.

11,457 (1920). Kinzberger u. Co. Purification of anthraquinone. (113,885.) Nov. 17.

15,534 (1920). Kinzberger u. Co. Purification of crude anthracene. (144,648.) Nov. 17.

18,758 (1920). Meister, Lucius, u. Brüning, Manufacture of pyridine bases. (146,869.) Nov. 17.

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

British Dyestuffs Corp., Frank, Gibson, and Robinson, Manufacture of azo colouring-matters and an intermediate. 32,242. Nov. 15.

Davies, and Scottish Dyes, Ltd. Colouring-matters. 33,558. Nov. 27.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

Barnett, and Imperial Trust. Production of cellulose esters. 31,684. Nov. 9.

Burgess, Ledward and Co., and Harrison. Treatment of cotton yarn. 32,508. Nov. 18.

Courrier. Producing paper pulp. 31,527. Nov. 8. (Fr., 11.11.19.)

Dreaper. Manufacture of viscose solutions. 31,465. Nov. 8.

Dreaper. Manufacture of artificial silk etc. 31,854. Nov. 10.

Dreaper. Manufacture and treatment of artificial filaments of organic origin. 32,706. Nov. 19.

Manoury. Obtaining cellulose and paper pulp from alfa grass etc. 32,123. Nov. 13.

Milne. Paper-pulp-refining engines. 33,360. Nov. 26.

Novosselsky. 32,005. See IX.

Roberts. Manufacture of articles from cellulose or its compounds. 33,427. Nov. 26.

COMPLETE SPECIFICATIONS ACCEPTED.

21,378 (1919). Drets and Noorden. Extraction of fibre from leaves etc. (153,413.) Nov. 17.

24,737 (1919). Muller. Manufacture of artificial threads, films, etc. from viscose. (153,444.) Nov. 17.

25,729 (1919). Bollmann. Removal of volatile solvents from materials treated therewith. (151,044.) Dec. 1.

1116 (1920). Hashimoto. Fibre, and process for obtaining same. (153,801.) Nov. 24.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Bowden and Bowden. Machines for bleaching, dyeing, etc. cloth. 32,509. Nov. 18.

Horsnell, and Simplex Patent Dyeing Machine Co. Dyeing, washing, etc. machines. 33,246. Nov. 25.

Marks (Duratex Co.). Coated fabric. 33,162. Nov. 24.

Nathan, Nathan, and Nathan. Treatment of textile fabrics. 32,657. Nov. 19.

Nelson. Apparatus for mercerising cotton. 32,651. Nov. 19.

Nelson. Mercerising cotton. 33,073. Nov. 24.

COMPLETE SPECIFICATION ACCEPTED.

23,199 (1919). Arent. Fireproofing and/or waterproofing treatment of materials. (132,813.) Dec. 1.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Jenkins. Apparatus for converting brine into salt. 32,178. Nov. 15.

Lundén and Thorsell. Production of nitrogen compounds. 32,060. Nov. 12. (Sweden, 3.12.19.)

Lundén and Thorsell. Production of pure nitrogen. 32,360-1. Nov. 16. (Sweden, 19.12.19.)

Ratner. Fixation of atmospheric nitrogen. 31,592. Nov. 8.

Roiboul. 32,711. See VIII.

COMPLETE SPECIFICATIONS ACCEPTED.

11,621 (1919). Catlett. Oxysalt compositions. (126,646.) Nov. 17.

17,084 (1919). Sherwin. Precipitation of aluminium hydrate. (153,352.) Nov. 17.

18,847 (1919). Henwood. Manufacture of nitric acid. (153,362.) Nov. 17.

19,391 (1919). Hobsbawn (Grigioni). Recovery of nitrate of soda from solutions. (153,649.) Nov. 24.

27,016 (1919). Hultman. Manufacture of chrome alum. (138,594.) Nov. 17.

30,832 (1919). Marks (West Coast Kalsomine Co.). Manufacture of iron compounds. (153,792.) Nov. 24.

1711 (1920). Sieurin. Production of aluminium oxide from the chloride. (153,500.) Nov. 17.

6009 (1920). Fabr. de Prod. Chim. de Thann et de Mulhouse. Manufacture of potassium sulphate and hydrochloric acid. (154,111.) Dec. 1.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Atkinson, Moreshead, and Stein and Atkinson. Glass furnaces. 32,703. Nov. 19.

Feldenheimer and Plowman. Treatment of clay. 31,505. Nov. 8.

Hancock. Saggers for firing pottery. 33,518. Nov. 27.

Roiboul. Manufacture of film silica, alumina, etc. 32,711. Nov. 19. (Fr., 16.9.20.)

Wade (Buckman and Pritchard). Manufacture of refractory material. 31,587. Nov. 8.

COMPLETE SPECIFICATION ACCEPTED.

20,339 (1920). Schidrowitz, Feldenheimer, and Plowman. Treatment of clay. (153,861.) Nov. 24.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Barrie and Chadwick. Aromatic hydrocarbon cement. 32,456. Nov. 17. (Australia, 27.5.18.)

Marks. Concrete. 31,969. Nov. 12.

Naaml. Vennoots. Netherland Colonial Trading Co. Composition for preserving wood etc. 32,501. Nov. 17. (Belg., 8.9.20.)

Novosselsky. Solutions for mineralising etc. wood, cardboard, etc. 32,005. Nov. 12.

COMPLETE SPECIFICATIONS ACCEPTED.

14,855 (1919). Williams. Effecting colour transformation in wood. (153,619.) Nov. 24.

23,606 (1919). Ward. Manufacture of artificial stone. (153,433.) Nov. 17.

189 (1920). Smidth and Co. Building-material for concrete structures. (137,330.) Nov. 24.

10,436 (1920). Grubenholz-Imprägnierung Ges. Impregnation of wood. (141,728.) Nov. 24.

X.—MÉTALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Baker and Co. Alloys. 33,167. Nov. 24. (U.S., 20.1.20.)

Bowes. Soldering. 33,352. Nov. 25.

Bowes. Welding aluminium or its alloys. 33,353. Nov. 25.

British Scientific Instrument Research Assoc., Imperial Trust, Haigh, and Jackson. Manufacture of solder. 31,839. Nov. 10.

Brunskill. Preventing surface corrosion in aluminium alloys. 32,738. Nov. 19.

Dawson, and Ferranti, Ltd. Cast iron alloy. 33,290. Nov. 25.

Gibbons Bros., and Marle. Continuous annealing-furnaces. 32,767. Nov. 20.

Hess. Recovery of metal from chippings, residues, and ashes. 32,048. Nov. 12. (Ger., 3.4.18.)

Lemmon, Sulman, and Minerals Separation, Ltd. Recovery of gold from pyritic ores. 31,963. Nov. 11.
 MacLaren. Production of ferro-alloys. 32,198. Nov. 17.
 Ros. Oxidation of metals. 32,116. Nov. 13.
 Seiffert. Production of zinc dust. 32,028. Nov. 12. (Ger., 12.12.19.)
 Skudder. 33,571. See 1.

COMPLETE SPECIFICATIONS ACCEPTED.

7482 (1919). Bastian. Magnetic apparatus for use in heat-treatment of steel. (153,336.) Nov. 17.
 12,415 (1919). Goodwin and Cadenhead. Recovering vanadium from certain iron ores. (153,926.) Dec. 1.
 25,677 (1919). Sandberg and Humfrey. Heat treatment of steel. (153,756.) Nov. 21.
 3311 (1920). Edgley. Washing ores etc. (153,503.) Nov. 17.
 5519 (1920). Coles. Electrodeposition of alloys of copper and zinc. (154,108.) Dec. 1.
 5724 (1920). Hall, and Rolls-Royce, Ltd. Aluminium alloys. (153,514.) Nov. 17.
 5725 (1920). Hall, and Rolls-Royce, Ltd. Aluminium alloys. (153,823.) Nov. 24.
 7626 (1920). Elliott. Manufacture of wrought iron. (153,523.) Nov. 17.
 8727 (1920). Metallbank u. Metallurg. Ges. Bearing metals. (140,790.) Nov. 17.
 27,659 (1920). Goldschmidt A.-G. Increasing yield of chromium in the aluminothermic production of carbon-free ferrochromium or multiple alloys thereof from chrome-iron ore. (152,990.) Nov. 24.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Chloride Electric Storage Co. (Smith). Secondary battery plates or electrodes. 32,031. Nov. 12.
 Harrison (Dow Chemical Co.). Electrolytic cells. 32,208. Nov. 15.
 Masterman and Rogers. Electrolytic apparatus for preparing hypochlorite solutions. 32,039. Nov. 12.
 Pepper. Electric batteries. 33,562. Nov. 27. (U.S., 29.11.19.)
 Soc. Anon. Kummier & Matter. Manufacture of electrical resistance material. 31,804. Nov. 10. (Switz., 10.11.19.)
 Stewart. Electrodes for accumulators. 31,908. Nov. 11.

COMPLETE SPECIFICATIONS ACCEPTED.

17,603 (1919). Universal Accumulators, Ltd., and Martis. Plates for accumulators. (153,935.) Dec. 1.
 20,034 (1919). Campbell. Electric furnaces. (153,951.) Dec. 1.
 20,688 (1919). Smalley. Heating elements for electric furnaces. (153,701.) Nov. 24.
 23,750 (1919). Hepburn, and Mather and Platt. Bi-polar electrode electrolyzers. (151,029.) Dec. 1.
 5549 (1920). Coles. See X.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Bates. Saponification. 32,007. Nov. 12.
 Clark and Co., and Tervet. 32,250. See II.
 Ellis. Thickening and bleaching oils. 33,405. Nov. 26.
 Jarrett. Manufacture of soap and soap powder. 32,000. Nov. 12.
 Mezger. Manufacture of grease. 32,925. Nov. 22.

COMPLETE SPECIFICATIONS ACCEPTED.

17,356 (1919). Baker and Bonsall. Detergents. (153,634.) Nov. 24.
 19,286 (1919). Seobel. Producing solidification of oleaginous substances. (153,942.) Dec. 1.

19,739 (1919). Stephens (Sharples Specialty Co.). Extraction of lumpy and oily matters from wet sludges etc. (153,668.) Nov. 24.
 1129 (1920). Nordiske Fabr. De-No-Fa Akt. Deodorising and purifying fish oils. (140,372.) Dec. 1.

1974 (1920). Verein. Chem. Werke. See XVIII.
 17,464 (1920). Mayrhofer. Manufacture of a cleaning agent. (146,222.) Nov. 24.
 25,144 (1920). Davton Metal Products Co. Hydrogenation apparatus. (150,991.) Dec. 1.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Fitzgerald. Substitute for shellac etc. 32,878. Nov. 22.
 Marks (Stanley Insulating Co.). Producing and applying enamel coatings. 33,163. Nov. 24.
 Melamid. Manufacture of resinous substances. 33,435. Nov. 26. (Ger., 17.5.20.)
 Schou. Emulsions for painting and priming etc. 33,189. Nov. 24.

COMPLETE SPECIFICATIONS ACCEPTED.

31,550 (1919). Scudder and Pettigrew. Production of phenol-resin condensates. (153,796.) Nov. 24.
 1034 (1920). Birkby and Birkby. Production of phenolic aldehyde condensation products. (153,494.) Nov. 17.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Balke and Leysieffer. Production of plastic bodies resembling vulcanised rubber. 32,496. Nov. 17. (Ger., 19.3.19.)
 Earp and Wood. Press for continuously moulding, vulcanising, or curing rubber etc. 32,251. Nov. 15.
 White (Goodrich Co.). Vulcanisation of rubber. 31,955. Nov. 11.

COMPLETE SPECIFICATIONS ACCEPTED.

13,992 (1919). Schidrowitz, Feldenheimer, and Plowman. Manufacture of rubber. (153,343.) Nov. 17.
 19,380 (1919). Young and Benner. Reclaiming rubber. (153,646.) Nov. 24.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Carmichael and Ockleston. Tanning. 31,494. Nov. 8.
 Chem. Fabr. Worms. Manufacture of tanning materials. 32,457 and 32,608. Nov. 17 and 18. (Ger., 18.11.19 and 20.8.17.)
 Lambeck. Making colouring of natural horn water-resisting. 32,891. Nov. 22. (Ger., 26.9.19.)
 Melamid. Manufacture of artificial tanning substances. 33,436. Nov. 26.

COMPLETE SPECIFICATIONS ACCEPTED.

537 (1920). Nakayama and Adachi. Utilising waste or scraps of leather. (154,095.) Dec. 1.
 3106 (1920). Wood. Puering or bating skins. (154,103.) Dec. 1.
 8417 (1920). Knudsen. Manufacture of fish-glue. (153,526.) Nov. 17.

XVI.—SOILS; FERTILISERS.

COMPLETE SPECIFICATIONS ACCEPTED.

23,730 (1919). Hyatt and Fellowes. Fertilisers and their manufacture. (153,434.) Nov. 17.

26,780 (1919). Baker and Shepherd. Fertiliser and insecticide. (154,057.) Dec. 1.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

Cutler. Process of refining molasses. 31,540. Nov. 8.

Denington and Perry. 32,598-9. *See* XVIII.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Denington and Perry. Treating wood. 32,598. Nov. 18.

Denington and Perry. Apparatus for treating wood etc. to obtain fermentable sugars, furfural, acetic acid, etc. 32,599. Nov. 18.

Denington and Perry. 32,600-1. *See* 11.

Distillers Co., and Meyer. Process for denaturing alcohol. 31,589. Nov. 8.

COMPLETE SPECIFICATIONS ACCEPTED.

19,733 (1919). Akt. Dansk Gaeringsindustri, and Sak. Manufacture of yeast. (153,667.) Nov. 24.

19,830 (1919). Groll. Manufacture of fermentation preparations. (131,589.) Nov. 24.

1974 (1920). Verein. Chem. Werke. Manufacture of glycerol from sugar. (138,328.) Dec. 1.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Archer and Stevenson. Preserving and sterilising milk etc. 32,230. Nov. 15.

Callimachi. Treatment of potatoes etc. 32,602-3. Nov. 18.

Craig, and Spence and Sons. Purification of water. 32,644. Nov. 19.

Fitzgerald. Production of commercial products from blood. 32,741. Nov. 19.

Forget-me-not, Ltd., and Hutchinson. Ageing wheat and flour. 32,802. Nov. 20.

Hegney and Hegney. Sterilising milk. 31,603. Nov. 8.

COMPLETE SPECIFICATIONS ACCEPTED.

3245 (1919). Linden. Reducing the percentage of water in sewage, sludge, etc. (153,335.) Nov. 17.

20,298 (1919). Lendrich. Production of coffee substitutes. (153,971.) Dec. 1.

24,823 (1919). Hoyberg. Butter-making. (153,446.) Nov. 17.

7141 (1920). Manganoozone Soc. Anon. Sterilising and clarifying water. (140,077.) Nov. 24.

7381 (1920). Anhydrous Food Products Co. Drying fruits, vegetables, meats, etc. (140,102.) Nov. 24.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Bader, and Brit. Cellulose and Chemical Manuf. Co. Manufacture of alkyl compounds. 32,037. Nov. 12.

Denington and Perry. 32,598-9. *See* XVIII.

Green and Levinstein. Manufacture of a toxic product. 32,243. Nov. 15.

Kolshorn. 33,206-7. *See* III.

Marchand. Preparation of terpineol. 31,841. Nov. 10. (Holland, 10.11.19.)

Marchand. Preparation of terpin hydrate. 31,842. Nov. 10. (Holland, 10.11.19.)

Merek and Wolfes. Preparation of tropin monocarboxylic acid esters. 32,349-32,351. Nov. 16. (Ger., 23.8.19.)

Metcalfe and Usher. Extracting essential oils etc. 33,482. Nov. 26.

COMPLETE SPECIFICATIONS ACCEPTED.

10,923 (1919). Commercial Research Co. Manufacture of chlorhydrins or bromhydrins. (126,311.) Nov. 17.

6081 (1920). Napp (Hoffmann-La Roche & Co.). Manufacture of colloidal soluble metal pyrophosphate casein compounds. (154,112.) Dec. 1.

6329 (1920). Soc. Chim. Usines du Rhône. Manufacture of β -dialkylaminoethylaminobenzoic alkyl esters. (153,827.) Nov. 24.

7018 (1920). Soc. Chim. Usines du Rhône. Manufacture of saccharin. (153,520.) Nov. 17.

9826 (1920). Moureu, Dufraisse, Robin, and Pougnet. Stabilisation of acrolein. (141,361.) Nov. 24.

19,962 (1920). Goldschmidt A.-G., and Matter. Production of ethylene chloride. (147,908.) Nov. 17.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Dörendahl and Schoepp. Photographic films. 32,729. Nov. 19. (Ger., 26.11.19.)

Dye Impression Photos, Ltd., and Edridge. Photographic films etc. 33,449. Nov. 26.

Hauff u. Co. Photographic developers. 32,883. Nov. 22. (Ger., 23.10.18.)

Warner. Producing photographs in natural colours. 31,846. Nov. 10.

XXII.—EXPLOSIVES; MATCHES.

COMPLETE SPECIFICATION ACCEPTED.

31,201 (1919). Dynamit A.-G. vorm Nobel u. Co., and Naoum. Manufacture of gelatinous nitroglycerin explosives proof against firedamp. (140,746.) Nov. 17.

XXIII.—ANALYSIS.

APPLICATIONS.

Chandler, and South Metropolitan Gas Co. Apparatus for determining calorific value of combustible gases. 33,036. Nov. 23.

Chandler, Evans, Hollings, and South Metropolitan Gas Co. Determining calorific value of coal gas etc. 33,037. Nov. 23.

Kessler. Specific-gravity testers. 32,622. Nov. 18.

COMPLETE SPECIFICATIONS ACCEPTED.

16,649 (1919). Meadows. Apparatus for testing the physical properties of materials. (153,621.) Nov. 24.

18,280 (1919). Wollaston. Apparatus for continuous analysis of gases. (153,637.) Nov. 24.

3503 (1920). Igranic Electric Co. (Cutler-Hammer Manuf. Co.). Determining calorific value of combustible gases. (153,817.) Nov. 24.

16,503 (1920). Soc. des Hauts-Fourneaux de Rouen. Calorimeters. (145,437.) Dec. 1.

I.—GENERAL; PLANT; MACHINERY.**PATENTS.**

Furnaces; Process and apparatus for operating —. G. and K. H. Siegwart, Asses. of R. Siegwart. E.P. 130,596, 25.7.19. Conv., 29.7.18.

AIR previously heated in a regenerator passes across the surface of a refractory tile or "gasifier" plate, and hydrocarbons and water (or steam) are supplied through separate jets approximately perpendicular to the gasifier plate. The plate may have a convexity, a concavity, or both upon its surface.

—B. M. V.

Heat-treating furnace. C. J. Kirk. E.P. 152,177, 25.8.19.

A LONG chamber is divided by a longitudinal partition into two compartments through which two sets of the objects to be heated are conveyed in opposite directions. Heat is transmitted from the hot goods leaving the chamber to the cold goods entering by means of air circulating through holes in the partition. The heating medium, e.g., hot products of combustion, is supplied from two separate combustion chambers situated near the middle of the length of the chamber, and passes through two separate sets of flues in the walls and roof surrounding the goods chamber, the two sets of flues leading towards opposite ends of the furnace. The hottest part of the furnace is at the middle, i.e., near the combustion chambers, whilst near the ends the incoming goods are heated partly by the heating flues and partly by air circulation from the hot goods leaving the furnace.—B. M. V.

Furnaces [; Recuperative] —. A. Smallwood. E.P. 153,042, 24.6.19. Addn. to 125,744 (J.), 1919, 489 A).

THE heated air for combustion is admitted to the combustible gas after the latter has left the primary combustion chamber, the admixture being made while the gases are in or leaving the intermediate heating flues. The air is preferably introduced into the heating flues by means of several ports distributed along their length.—B. M. V.

Shaft furnaces; Utilisation of the waste heat from —. A.-G. für Zinkindustrie vorm. Wilhelm Grillo. G.P. 321,524, 28.4.16.

TWO or more shafts are connected below by means of a combustion chamber or a reverberatory furnace, and each shaft is used in turn as a heat regenerator. For example air is preheated in one shaft (previously used as burning shaft) and passes to the combustion chamber, the hot gases from which heat the material in the second shaft. Alternately the gas may be preheated instead of the air, or by using four shafts, the air may be preheated in one pair and the gas in the other.

—W. J. W.

Drying apparatus. C. Whitfield. E.P. 152,712, 12.6.19.

THE goods to be dried are conveyed through a long chamber with perforated bottom under which radiators are placed. Air is circulated through the chamber and at the same time given a vibratory motion by means of a fan. There are two sets of radiators: primary ones, heated from an external source and preferably situated near the exit end of the chamber, and secondary ones through which the hot saturated air from the drying chamber is drawn by the fan, after which it passes to a vessel in which condensed moisture is deposited and is then fed back to the entrance end of the drying chamber. If desired, the secondary radiators may be in the roof of the chamber.

—B. M. V.

Drying apparatus. J. Killby and F. C. Allen. E.P. 152,922, 5.3.20.

A NUMBER of rotary drying chambers are arranged in a series so that the material lifted by the rotation of a chamber drops on to a shoot and is thus transferred from one chamber to another and finally discharged. Each chamber has a separate supply of drying gases, but these supplies may be derived from a single gas generator and the draught provided by a single suction fan.

—B. M. V.

Drying machine. J. L. Pierce. U.S.P. 1,356,469, 19.10.20. Appl., 11.6.19.

THE material to be dried is conveyed through a hot air chamber upon endless conveyors supported on ribs.—B. M. V.

Dryer; Chamber —. A. E. H. Beyer. G.P. 317,689, 3.5.14.

THE drying chamber is divided into separate compartments and hot air is led in both at the top and through the floor. The end compartments are connected below the floor by conduits for conveying hot air from the one to the other, or for separating moisture from the air; the conduits are connected with each compartment by valves operated from the top, and are also connected with the apparatus in which the moist vapour is condensed.—L. A. C.

Mercury or other fluid having a high boiling point; Boilers particularly intended for vaporising —. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 152,785, 23.7.19.

THE boiler consists of a number of separate sections assembled within a casing, each section comprising a pair of vertical plates slightly spaced apart to form a container for the mercury. The sections are provided with external vertical ridges on their faces, formed by pressing the plates, so that when assembled a tortuous passage alternately upward and downward is formed for the heating gases. The interior of the sections is provided with vertical partitions forming similar passages for the mercury. The passages are so arranged that when the mercury enters the section it travels upwards in the same direction as the hot gas, but in its final movement it travels in the opposite direction to that of the hot gas, the mercury making three passes and the hot gas two passes in each section. The sections are supported so as to provide for expansion, and each is provided with preheating passages at the outer edges. The mercury vapour is used to drive a turbine or other prime mover, the exhaust from which is used to generate steam, and the hot waste gases are used for superheating steam.

—W. F. F.

Refrigerating; Method of and apparatus for —. B. Thoens. E.P. 152,898, 21.12.19.

IN a method of refrigerating by evaporation of a volatile fluid such as ammonia from its solution, a part of the cooled solution is used to absorb the vapour removed from another part by evaporation, the second part being then used for refrigerating. Part of the cool refrigerant from the evaporating tank is circulated through a closed circuit which includes the absorbing tank but not the refrigerator, and another part is circulated through a separate closed circuit which includes the refrigerator but not the absorbing tank.—W. F. F.

Refrigerating machine. R. Plersch and H. Meidinger. G.P. 322,796, 28.2.19.

THE vapours of the refrigerating agent coming from the evaporator are condensed by a jet compressor contained within the condensing apparatus. The refrigerating liquid, cooled by means of cold vapours

arising from the evaporator is employed as working fluid in the jet compressor, and is kept in circulation by means of a pump.—J. S. G. T.

Grinding ores, minerals, stones and the like; Machines for — J. R. Broadley. E.P. 152,724, 8.7.19. Addn. to 116,583 (J., 1918, 453A).

By rearranging the parts of the screening device the passage for the return of oversize material to the mill may be formed as a single trough or tube of involute shape. Trunnions may be used to support the mill instead of tyres and rollers.—B. M. V.

Pulverising apparatus. J. E. Kennedy. U.S.P. 1,356,292, 19.10.20. Appl., 2.2.18.

CRUSHING rollers mounted on vertical spindles which revolve around a central vertical axis move in contact with a fixed grinding ring. Material is fed between the ring and rollers from below, and a central fan is provided for directing air under pressure between the ring and rollers from below.

—W. F. F.

Evaporator and method of operating such evaporator. G. H. Benjamin. U.S.P. 1,355,935, 19.10.20. Appl., 23.2.18.

LIQUID is evaporated in a chamber at a controlled temperature by means of steam coils, and the vapour passes into a superheating chamber, where it is heated to a controlled temperature by an electric heater.—W. F. F.

Condensing [evaporating] apparatus. I. S. Merrell, Assr. to Merrell-Soule Co. U.S.P. 1,356,082, 19.10.20. Appl., 26.12.16.

AN evaporating chamber and its cover are both carried by the same framework, and are movable relatively to one another. A rotary heater is provided in the evaporating chamber, driven by a shaft passing through the cover, the shaft being separable into two sections when the cover is lifted.—W. F. F.

Evaporators; Means for ensuring the formation of a thin film of the liquid under treatment on the inner walls of the tubes of vertical tube — A. G. der Maschinenfabr. Escher, Wyss und Co. G.P. 321,937, 22.1.19.

THE top of each tube is opened out to form a curved surface of such a shape that the descending liquid tends to flow along the inner wall of the tube, leaving the central portion empty. The tops of the tubes are at progressively lower levels the greater their distance from the liquid inlet.—L. A. C.

Container for alkaline substances. G. W. Howlett, Assr. to The Research Laboratories Co. U.S.P. 1,355,976, 19.10.20. Appl., 10.7.19.

AN alkali-proof coating composition for fibre receptacles consists of a cellulose ester dissolved in a solvent and mixed with a non-solvent and with a material to render the coating less brittle.

—B. M. V.

Distillation or the like; Apparatus for — V. L. Emerson. U.S.P. 1,356,057, 19.10.20. Appl., 11.5.20.

A FURNACE for heating a tubular evaporator is arranged so that the combustion products pass downwards into a pit and thence to the bottom of the stack. The pit is constructed so that when filled with liquid the passage leading from the furnace is closed.—W. F. F.

Distillation; Process of — D. L. Newton. U.S.P. 1,356,878, 26.10.20. Appl., 11.9.18.

A LIQUID containing a volatile substance is passed in the form of a film through a distilling chamber at ordinary temperature, and subjected to a partial vacuum to distil off the volatile substance.

—W. F. F.

Vapour in a natural gas; Method of condensing a — J. T. Duffy. U.S.P. 1,356,196, 19.10.20. Appl., 27.5.18. Renewed 6.5.20.

THE gas is simultaneously cooled and subjected to the action of an alternating brush discharge, thereby producing a nucleus around which the vapour will condense.—L. A. C.

Separating apparatus. E. L. Marshall. U.S.P. 1,356,384, 19.10.20. Appl., 11.8.19.

MATERIAL to be separated is fed from a hopper, and meets a horizontal blast of air. The heavier particles fall into a conduit, from which they pass out of the apparatus, while the lighter particles are carried through an opening between vertical plates, one of which is adjustable to vary the opening. The heavier particles fall into a conduit and are separated into two grades in passing over a perforated floor, the coarser grade being returned by a conveyor to the hopper. The air carrying the lighter particles then passes into a larger chamber, where the particles are kept in suspension by suction and the lightest particles are drawn off. The heavier particles are partly deposited on a sloping floor and are withdrawn through an outlet, while the air carrying the remainder of the material passes to another chamber, in which the heavier particles are again separated, and are directed by guide plates to the sloping floor, while the air containing the finest particles in suspension passes out at the top of the casing.—W. F. F.

Electrical precipitation of suspended matter in gaseous and fluid bodies; Apparatus for — Removing suspended particles from fluid or gaseous bodies. Producing electrical precipitation of particles from fluid or gaseous streams. A. F. Nesbit, Assr. to International Precipitation Co. U.S.P. (A) 1,356,462, 19.10.20, (B) 1,357,201, and (C) 1,357,202, 26.10.20. Appl., (A, B) 17.11.14, (C) 16.9.15.

(A) AN electrode frame is arranged between spaced plate-like receiving electrodes forming a gas passage. A series of parallel, flexible discharge members extending longitudinally through the passage are held rigidly by the frame. (B) The discharge system of opposing electrode systems comprises a number of wires arranged spirally in the direction of length of the system, each wire having its axis out of the axis of symmetry of the opposing electrode system, whereby an unsymmetrical field is produced in the various cross-sections of the system, through which the fluid stream tends to flow spirally. (C) The discharge system of opposing electrode systems comprises a frame carrying rigidly a number of electrode elements arranged parallel to one another and to one side of the frame.

—J. S. G. T.

Oxidation process; Catalytic acceleration of — C. S. Piggot. U.S.P. 1,357,000, 26.10.20. Appl., 3.1.20.

THE material to be oxidised is heated with a gas containing oxygen in the presence of a catalyst consisting of an alloy of metals precipitated by hydrogen sulphide in alkaline solution.—L. A. C.

Heat exchanger for condensation of petroleum or tar vapours etc. A. J. Irinyi. G.P. 322,745, 22.3.14.

THE vapour to be condensed and the cooling liquid are passed through series of adjacent channels, wide at one end and narrow at the other, formed by suitably corrugated partitions, consecutive channels tapering in opposite directions. The vapour and the cooling liquid circulate at right angles to the length of the channels and from the wide to the narrow ends, so that the velocity acquired at the narrow end of one channel is transformed into a

whirling motion when the fluid enters the wide end of the next. The channels through which the vapour passes may be provided with discharge outlets for condensed liquid at their lowest points.

J. H. L.

Filters; Operation of drum —. G. Polysius. G.P. 324,730, 27.6.19.

The filter fabric is fastened to the drum by windings of wire or cord, consecutive turns being disposed a short distance apart. The filter cake is removed from the rotating drum by means of jets of liquid, and as a result of the uneven surface produced by the wire or cord it comes off easily in narrow strips, so that jets operating under a lower pressure than usual can be employed, and wear of the filtering fabric is consequently diminished.

—J. S. G. T.

Filter drum. H. Hencke. G.P. 324,797, 3.8.18.

The filter cloth is secured to the circumference of the filter drum by means of pressure bars or rollers etc.—J. S. G. T.

Centrifugal machine. P. T. Sharples. E.P. 153,041, 23.6.19.

SEE U.S.P. 1,320,119 of 1919; J., 1920, 215 A.

Centrifugal separator. R. S. Brownlow. U.S.P. 1,356,805, 26.10.20. Appl., 13.2.19.

SEE E.P. 120,319 of 1918; J., 1918, 767 A.

Kilns and other structures; Heating arrangement for —. G. C. Heilman. Reissue 11,972, 26.10.20, of U.S.P. 1,237,036, 14.8.17. Appl., 13.8.19.

SEE J., 1917, 1049.

Furnace; Forced rotary-draught furnace. J. Gaskill. U.S.P. 1,357,491, 2.11.20. Appl., 3.9.18.

SEE E.P. 132,266 of 1919; J., 1920, 602 A.

Refrigeration plants; Agent for —. A. Henning. U.S.P. 1,356,765, 26.10.20. Appl., 31.10.19.

SEE E.P. 131,573 of 1919; J., 1919, 751 A.

Heat-exchange apparatus. P. Audianne. U.S.P. 1,358,050, 9.11.20. Appl., 4.1.19.

SEE E.P. 142,522 of 1918; J., 1920, 487 A.

[Blast] furnaces; Apparatus for charging —. F. Clements. E.P. 153,032, 5.5.19.

Distillation and fractionation. E.P. 152,791. See II.A.

Preheater. U.S.P. 1,337,287. See II.A.

Evaporator. G.P. 323,341. See XVII.

II.A.—FUEL; GAS; MINERAL OILS AND WAXES.

Hoo canal. Strata associated with coal. I. F. S. Sinnatt and M. Barash. Bull. 6, Lancs. and Cheshire Coal Res. Assoc., 1920, 15 pp.

The term Hoo canal is largely used in Lancashire for canals containing a high percentage of ash to distinguish them from low-grade shales. Seams varying in thickness from a few inches to several feet occur in the Lancashire coalfield. The average composition of the specimen (from Hindley) examined was: moisture 2.10%, volatile organic matter 31.60%, coke 66.30%, fixed carbon 39.78%, ash 24.42%; C 60.74%, H 4.23%, N 1.30%, S 1.23%, and O 8.08%. The calorific value was 11,000 B.Th.U. per lb. (14,700 B.Th.U. on an ash-free basis). Extraction with pyridine yielded 12.7% of extract compared with 29.4% of extract from the coal associated with the canal (both figures calcu-

lated to an ash-free basis). The ash would seem to be an inherent constituent of the canal substance, since no improvement in composition could be effected by crushing and washing the material. Hoo canal can be pulverised approximately much more easily than Arley coal, and might be suitable for powdered fuel firing. Similar material from the Burnley area averaged 15.5% of ash and 19.0% of volatile organic matter; from the Bickershaw area 32.1% of ash and 28.3% of volatile organic matter; and from the Garswood area 10.1% of ash and 48.1% of volatile organic matter. Carbonisation of the Hindley canal at 920° C. yielded 9870 cb. ft. of gas per ton, or corrected to 16 c.p. 9210 cb. ft. at n.t.p. The c.p. was 15.0 and the calorific value 521 B.Th.U. per cb. ft. The coke yield was 68.9% and the tar yield 8.0 gals. per ton (4.1% by weight). The tar had sp. gr. 1.119 at 20° C. and yielded the following fractions: Up to 170° C., 6.8%; 170°–230° C., 15.5%; 230°–270° C., 8.95%; 270°–330° C., 22.9% and 45.0% of a brittle pitch. There is a much smaller yield of naphthalene and a much larger yield of anthracene from canal tar than from coal tar.—A. G.

Sulphur in coal; Reactions of — in the coking process. A. R. Powell. J. Ind. Eng. Chem., 1920, 12, 1069–1076.

In the coking of coal pyrites and marcasite are decomposed completely into ferrous sulphide, pyrrhotite, and hydrogen sulphide, the reaction beginning at 300° C., usually reaching its maximum between 400° and 500° C., and being complete at 600° C. Sulphates are reduced to sulphides, the reaction being complete at 500° C. Primary decomposition of organic sulphur compounds to form hydrogen sulphide is most active below 500° C.; about a half of the organic sulphur is thus eventually decomposed. A small portion of the organic sulphur reacts at the lower temperatures of the process to form volatile organic sulphur compounds, most of which will be found in the tar. The remainder of the organic sulphur undergoes a pronounced change between 400° and 500° C. The total amount of sulphur in the coal is the most important factor affecting the sulphur content of the coke. The secondary reaction between the sulphur in the red-hot coke and the hydrogen in the by-product gases passing through it cause a more pronounced reduction than the primary reactions would indicate (*cf. infra*). Coke has been found to contain a magnetic iron sulphide, probably pyrrhotite.—C. A. M.

Coke; Desulphurising action of hydrogen on —. A. R. Powell. J. Ind. Eng. Chem., 1920, 12, 1077–1081.

HYDROGEN passed through coke for 3 hrs. at 1000° C. removes most of the sulphur without affecting the character of the coke. The effect of passing hydrogen through coal in the process of coking is to modify three of the sulphur reactions. The pyrites decomposes at a lower temperature (500° C.) without, however, affecting the final results. The decomposition of organic sulphur to form hydrogen sulphide is but little affected below 500° C., but is very greatly increased between 500° and 1000° C. This increased decomposition is the cause of the desulphurising action of hydrogen. A larger amount of the sulphide is converted into a "carbon-sulphur" combination. Gaseous mixtures containing hydrogen, such as coke-oven gas, although effective, are much slower in their action and would probably never effect so complete a removal of sulphur as hydrogen. The circulation of by-product gas through the coking mass may, however, be of commercial value. The desulphurising process is not affected by the original state of subdivision of the coal.—C. A. M.

Coke-ovens; Regulation of gas pressure in —. A. Thau. Glückauf, 1920, 56, 901—906, 925—930.

No coke-oven being a gas-tight chamber, pressure differences between the chamber, the heating flues, and the outside air lead to leakages. With a positive pressure in the oven the consequences are loss of gas, and carbon deposits in the heating flues. With a negative pressure partial combustion of the gas takes place in the oven and the resulting rise in temperature damages the exit pipes and leads to decreased yields of tar, benzol, and ammonia, and to poor gas. For ordinary working a slight positive pressure should be maintained. This principle applies to all retort practice, with or without steam, and whether gas or coke is the main desideratum. The idea that the yields of by-products can be increased by applying suction is erroneous. In many coking plants the pressure is regulated by the aid of a control flame allowed to burn on a branch pipe from the gas main. Detailed pressure measurements taken on a Koppers oven, with corresponding gas analyses, are given to show the defects of this method. The pressure is lowest at the two end doors of the oven, somewhat higher along the top, and much higher in the gas-main. Hence suction on the oven and a bad gas composition can co-exist with pressure on the gas-main and the control flame burning. The device of allowing control flames to burn at the observation holes of several oven doors is preferred. Attempts to devise an automatic control operating from a manometer to the blowers have met with great difficulty owing to the tar vapour and temperature of the gas. To render such control practicable the difference of pressure between the ovens and the point in the main connected with the manometer, which varies for each plant and may vary in the same plant with the moisture content of the coal and the deposition of carbon in the pipes, must be accurately known. A description of a membrane micromanometer and of the mechanical details of automatically controlled blowers is given.—C. I.

Flue gases derived from the combustion of coal; Graphic representation of the analysis of —. F. Schulte. Glückauf, 1920, 56, 532—536.

The system of triangular coordinates introduced by Ostwald (J., 1919, 492A), whereby the characteristics of combustion are represented by points associated with a right-angled triangle is used to represent the results of combustion of coke, anthracite, lean coal, fat coal, gas coal, and coal very rich in volatile combustible matter. Analyses of typical samples of these various coals are given and are accompanied by triangular diagrams appropriate to the respective cases. The method of construction of the diagrams is explained, and data are provided for their construction in the case of any of the typical coals enumerated. The application of the diagrams to the determination of the characteristics of the combustion, utilising the results afforded by analysis of the flue gases, is briefly explained. The correctness or otherwise of the analysis is thus readily ascertained; likewise the completeness or otherwise of the combustion and whether excess of air is being used or not.—J. S. G. T.

Flames; Silent aerated —. H. Davies. Yorks. Junior Gas Assoc. Gas World, 1920, 73, 387—388, 407—409.

The noise of an aerated flame is not necessarily due to imperfect mixing of gas and air, nor to the presence of eddies in the current of the mixture. A flame produced at the orifice of a single tube by the combustion of a homogeneous gas-air mixture is noisy if a critical consumption, depending upon the length and bore of the tube, is exceeded. The consumption rises to a maximum corresponding to a particular composition of the gas-air mixture.

With this mixture consumption increases as length of the tube increases until a critical length is reached, after which the consumption is constant. When lengths equal to, or greater than, the critical lengths of tubes of different diameter are compared, the consumptions producing just silent "silent steady" flames are nearly proportional to the internal diameters or perimeters of the tubes. The author explains these observations by suggesting that differences in frictional resistance with different lengths of tubes and with tubes of different diameters, cause differential velocities of the gas stream. A certain differential value is necessary for a steady flame.—W. P.

Radiation in explosions of hydrogen and air. W. T. David. Proc. Roy. Soc., 1920, A 98, 183—198.

THE radiation phenomena in hydrogen mixtures are, in general, similar to those in coal-gas mixtures (cf. J., 1919, 707A; 1920, 218A). The maximum rate of emission, however, occurs approximately at the point of maximum temperature, and not during the explosion period, as in the case of coal-gas mixtures. Further, the proportion of the heat of combustion lost by radiation during explosion and subsequent cooling decreases with the mixture strength (and hence with the maximum temperature developed) much more rapidly in the hydrogen mixtures than in the coal-gas mixtures. The 2.8μ emission band of steam ceases to be emitted when the temperature has fallen to about 1000° abs., and the results are taken to indicate that the vibratory degrees of freedom of the steam molecules corresponding to radiation of 2.8μ share the heat energy of the molecules only above 1000° abs.; this may account for the rapid increase of specific heat in this region.

—J. R. P.

Gases; Behaviour of explosive mixtures of — at low pressures. A. Stavenhagen and E. Schuchard. Z. angew. Chem., 1920, 33, 286—287.

EXPLOSIVE mixtures of gases no longer ignite when the pressure falls below a definite limit. The intensity of the explosion decreases with the fall in the pressure, and below the critical pressure slow combination takes place without flame. The critical pressure stands in relationship to the nature of the gas, the proportions of the mixture, the moisture, and the initial impulse. The possibility of a second explosion in a closed space is greater in proportion to the smallness of the pressure developed by the first explosion, or to the reduction in pressure following the first explosion. (Cf. J.C.S., Jan., 1921.)

—C. A. M.

Hydrocarbons; Dispersion of the refraction of —. E. Darmon. Comptes rend., 1920, 171, 952—955.

THE specific dispersion, $\Delta n/d$, where Δn is the difference in the refractive indices for $H\alpha$ and $H\gamma$, and d is the density at the same temperature, is apparently constant to within one or two units for each class of hydrocarbons, and may be used to distinguish between saturated, unsaturated, and benzenoid hydrocarbons. In the case of unsaturated hydrocarbons the value of the ratio increases with the number of double bonds present. The following values are given: Saturated aliphatic hydrocarbons and saturated cyclic hydrocarbons (cyclohexane series), 155; unsaturated cyclic and unsaturated aliphatic hydrocarbons with one double bond, 190; unsaturated aliphatic hydrocarbons with two double bonds, 225—230; benzenoid hydrocarbons, 300.—W. G.

Oils; Apparatus for determining the fluidity of — at low temperatures. Giorn. Chim. Ind. Appl., 1920, 2, 388—390.

THE oil is introduced into a calibrated U-tube 6 mm. in diam. up to the zero point of a scale

graduated in mm. The tube is chilled in a bath of the required temperature, and a pressure of 50 mm. of water is exerted in one of the two limbs of the tube. The amount of displacement of the oil in a given time, as measured on the scale, affords an index of its fluidity. A special chamber to receive a number of such tubes is described, in which the low temperature is produced by the expansion of liquid carbon dioxide from a cylinder attached to the chamber by means of a valved tube. The pressure applied to the tubes is regulated by a water manometer, and the chamber is provided with an observation window.—C. A. M.

Lubricants and emulsification. V. R. Abrams, C. H. Osmond, and T. G. Delbridge. Amer. Soc. Testing Materials. Petrol. Mag., 1920, 10, 126—152.

STEAM is injected into a 20 c.c. sample of oil contained in a test-tube. The emulsion is maintained at constant temperature by means of a water jacket at a definite temperature. The tube is then transferred to a separating bath maintained at 200°—203° F. (93°—95° C.), and the time taken for the oil to separate completely, or if complete separation has not taken place in 20 mins. the volume of separated oil, is determined. If the emulsification is commenced at 67° F. (19.5° C.) it is complete in 4—7 mins., by which time the temperature will have reached 190°—195° F. (88°—90.5° C.).—A. E. D.

Ammonium sulphate. Raschig. See VII.

Waste roofing felt. Hawley and Kress. See IX.

PATENTS.

Coke oven. A Roberts, Assr. to American Coke and Chemical Co. U.S.P. 1,356,788, 26.10.20. Appl., 15.9.16.

A **COKE-OVEN** heating wall provided with internal flues is constructed of blocks laid in courses, each block having three of its faces, perpendicular to the wall surface, made plain, and the fourth surface notched, the notches extending across the courses. The ends of the blocks are in alinement vertically, and spacing blocks are placed between the ends of consecutive courses of blocks so that the central portions of the blocks are in communication with the recesses formed by the notches.—W. F. F.

Gas retorts; Vertical —. Soc. Anon. d'Ougrée Marihay. E.P. 135,214, 15.11.19. Conv., 28.9.16.

COAL is supplied continuously to the top of vertical retorts of oblong cross-section, the side walls of which are heated by adjacent combustion chambers, while the end walls are not heated. The pasty layer of coal is of V-cross-section and gas-discharge openings are arranged in the unheated walls along or parallel to the V outline to prevent overheating of the products. The gas-collecting compartments are moderately heated either by the combustion air from the lower parts of the regenerator or by the fire gases which have passed along the retort walls or by special burners.—W. F. F.

Retorts for the distillation of carbonaceous materials; Heating of vertical —. J. West, W. Wild, and West's Gas Improvement Co. E.P. 153,139, 30.8.19.

VERTICAL retorts for coal distillation are heated by external combustion chambers, and to equalise the temperature in these chambers each is provided with two secondary air inlets, one adjacent to the flame orifice at which a limited amount of air is admitted, and the other midway between the ends of the combustion chamber to admit air to complete the combustion.—W. F. F.

Water-gas; Manufacture of —. A. Meade. E.P. 153,051, 28.6.19.

The supply of steam to the generator is automatically controlled in accordance with the pressure in the gas-exit pipe, and therefore with the temperature in the generator, by means of a flexible membrane stiffened at the centre. One side of this membrane is exposed to the pressure of the gas, and the movement of the central stiffening piece controls the steam valve by means of a valve rod and link motion.—C. I.

Lignite; Pre-treatment of friable — for gasification in by-product gas producers. Deutsche Erdöl A.-G., F. Seidensehnur, and C. Koettnitz. G.P. 323,588, 29.11.16.

The raw lignite is mixed with sulphates and formed into lumps. 1000 kg. of lignite, mixed with 250 kg. of concentrated magnesium sulphate solution, may be dried in a rotary drum and the dried balls fed into a generator and gasified; the sulphur of the sulphate is evolved as hydrogen sulphide, from which sulphur is obtained by interaction with sulphur dioxide. If calcium sulphate is employed it is reduced to sulphide, which is extracted with water and decomposed with carbon dioxide, and from the hydrogen sulphide evolved sulphur is obtained by a known method.—J. H. L.

Gas from blast furnaces, gas generating plants and the like; Apparatus for washing and drying —. E. F. Hickman. E.P. 153,053, 17.7.19.

Gas is passed downwards through a column fitted with water sprays at the top and also with further water-supply connexions at different heights. The column contains a number of turbines driven by the falling water, with interposed distributors for the gas and water. The washed gas is dried by a turbine rotated by the gas pressure and is finally passed through an annular perforated plate or filter.—C. I.

Tar-extractors and scrubbers for gas. J. F. Wells. E.P. 153,109, 7.8.19.

A **SERIES** of chambers is arranged one above the other in vertical column, each chamber being provided with a central gas inlet and outlet and a disc-shaped horizontal baffle fitted with vanes and revolving on a central vertical shaft. A water jet is introduced above each baffle, and the water and tar are drawn off through outlets at the periphery. The gas undergoes a final cleaning in an annular chamber packed with sawdust at the top of the column.—C. I.

Inert gases [products of combustion]; Process and apparatus for producing —. J. J. Shea, Assr. to The Hartford Rubber Works Co. U.S.P. 1,356,888, 26.10.20. Appl., 5.4.20.

A **COMBUSTIBLE** gas mixed with its exact equivalent of air is burnt on refractory surfaces, and the gases of combustion are dried.—C. I.

Lubricants; Wire-drawing —. A. S. Cache-maille. From Westinghouse Lamp Co. E.P. 137,329, 2.1.20.

FINELY divided graphite is held in suspension by a gelatinous, non-adhesive vegetable medium, e.g., agar-agar. The product is specially suitable for use as a lubricant for hot wire drawing, e.g., of tungsten.—A. E. D.

Petroleum or other hydrocarbons, or mixtures of volatile liquids; Apparatus for continuous distillation and fractionation of —. H. L. Allen. E.P. 152,791, 21.7.19.

An improvement of the method and apparatus described in Fr. Pat. 481,131 of 1916 (J., 1917, 127). The vapours from the stills pass through pre-

heaters, which are constructed in three superimposed compartments. Crude oil is fed into the lowest compartment and thence passes through tubes extending up through the middle compartment and discharging into the upper compartment. The intermediate compartment is heated by the vapours from the stills. From each pre-heater the crude oil flows in series to the next, and from each upper compartment of the pre-heater vapour passes into a fractionating device consisting of atmospheric condensers connected in cascade fashion. It is claimed that results superior to those provided by existing dephlegmation methods are obtained.

—A. E. D.

Petroleum, tar, and the like industry; Preheater for use in the chemical industry, especially for use in the —. L. Steinschneider, Assr. to The Chemical Foundation, Inc. U.S.P. 1,337,287, 20.4.20. Appl. 3.11.14.

A PREHEATER for use in the distillation of petroleum, tar, and the like consists of an upright vessel to which the liquid to be preheated is supplied and within which is a system of horizontal tubes through which the vapours evolved when the liquid is distilled are passed. The system of tubes is enclosed in a jacket of smaller cross-sectional area than the upright vessel, and means are provided for controlling the circulation of the liquid being preheated through and around the jacket.

Petroleum; Apparatus for cracking —. H. W. Jones, Assr. to The Kansas City Gasoline Co. U.S.P. 1,356,208, 19.10.20. Appl. 27.5.16.

OIL is preheated in a low-pressure boiler and passed continuously through a number of separate, subdivided units, arranged in parallel, wherein it is cracked at high temperature and pressure. The cracked product passes from each unit into a common receiver and thence into condensers.

—L. A. C.

[Paraffin] wax; Method of extracting —. H. M. Nichols, Assr. to The Atlantic Refining Co. U.S.P. 1,356,550, 26.10.20. Appl. 7.3.19.

A COOLED wax-bearing liquid, e.g., a paraffin distillate, is pressed into contact with one side of a filtering medium, the opposite side of which is in free contact with air maintained at a temperature below that of the liquid. The wax collects upon the filter medium and the liquid passes through and is collected. (Cf. U.S.P. 1,302,832; J., 1919, 493 A.)

—L. A. C.

Low-boiling hydrocarbons, e.g., petroleum spirit; Manufacture of — from natural gas or other purified gases. Apparate-Vertriebs-Ges.m.b.H. G.P. 307,624, 26.8.16.

THE gas is alternately compressed and allowed to expand, and as it expands it is scrubbed with a cooled atomised wash liquor.—L. A. C.

Montan wax; Production of — from lignite. A. Riebeck'sche Montanwerke A.-G. G.P. 325,165, 25.1.19.

IN a process of extracting lignite containing not more than 25% of moisture, with a mixture of benzene and a second solvent, the second solvent employed is a mixture of alcohol (in particular methyl alcohol) and acetone, preferably acetone oil. The yield of bitumen extracted is much greater than when benzene alone is used.—J. H. L.

Fuel suitable for a substitute for spirit and the like, and process for producing the same. W. Hänig. G.P. 325,711, 20.9.19.

THE fuel consists of hexamethylenetetramine, in the form of powder or compressed tablets or flakes, to which may be added other substances which facili-

tate combustion or ignition. Porous or absorbent materials, such as wicks, kieselguhr, etc., may be impregnated with solutions of hexamethylene-tetramine.—J. H. L.

Filtering agents [fuller's earth from treatment of oils]; Revivification of —. C. F. Kennedy, Assr. to The Atlantic Refining Co. U.S.P. 1,356,631, 26.10.20. Appl., 11.8.19.

THE material is treated with an aqueous soap solution.—L. A. C.

Ammonia contained in gases from coke ovens; Recovery of the —. A. E. White. From Soc. Franco-Belge de Fours à Coke. E.P. 153,177, 14.11.19.

SEE U.S.P. 1,315,639 of 1919; J., 1919, 809 A.

Ammonia from distillation-gases; Process for the direct recovery of —. J. Picette, Assr. to Soc. Franco-Belge de Fours à Coke. U.S.P. 1,356,885, 26.10.20. Appl., 11.9.18.

SEE E.P. 141,093 of 1918; J., 1920, 405 A.

Hydrocarbons; Process and apparatus for the continuous treatment of — with liquid sulphur dioxide. Allgem. Ges. für Chem. Ind. E.P. 133,962, 15.10.19. Conv., 27.4.15.

SEE G.P. 297,131 of 1915; J., 1919, 167 A.

Gas producers and other furnaces; Rotary grates for —. L. Tréfois. E.P. 153,163, 14.10.19.

Gas generators; Apparatus for agitating the fuel in —. G. H. Bentley and E. G. Appleby. E.P. 153,248, 10.7.20.

Heat exchanger. G.P. 322,745. See I.

Separating water etc. from crude oil. G.P. 322,895. See III.

Alcohol or ether from ethylene. E.P. 152,495. See XX.

II.B.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

"Norit"; Adsorbent power of — compared with that of blood charcoal. H. R. Kruyt and C. F. van Duin. Rec. Trav. Chim., 1920, 39, 679—684.

BLOOM charcoal, purified with acid, has a stronger adsorbent power for hydrogen and hydroxyl ions, phenol, bromine, and the sulphoeinamic ion than either commercial norit or norit purified by boiling first with dilute hydrochloric acid and then with water.—W. G.

Tungsten pigments. Gardner and Reilly. See XIII.

PATENTS.

Wood, woody fibre and similar carbonaceous substances; Destructive distillation of —. P. Poore. E.P. 152,741, 15.7.19.

WOOD, woody fibre, or the like is distilled in a retort heated externally, and after the temperature has been raised above 100° C. steam at a temperature of 180°—270° C. is admitted. A high yield of acetic acid, wood naphtha, etc., is obtained.—W. F. F.

Carbonisation of wood; Process and apparatus for the — in ovens. M. Fritz. G.P. 321,376, 30.6.14.

THE gases and vapours are withdrawn from the vertical carbonising chamber through outlets distributed over the whole height of the charge and are sucked in the direction of that end of the oven at which the carbonisation of the charge commences. Condensation in the uncarbonised parts

of the charge and the need for the re-vaporisation of the condensed products are in this way avoided.

—G. F. M.

Carbonisation of cellulose, peat, etc., by treatment under pressure and heat in presence of liquids. J. Billwiller. G.P. 323,595, 6.3.17.

Viscous liquids, such as tar oils, petroleum, mercury, salt solutions, or fused materials, are employed, and the temperature is raised to over 360° C. according to requirements. Homogeneous anthracitic products are obtained which are specially suitable for the manufacture of electrodes. As by-products, considerable quantities of tar and compressed gases (hydrogen, carbon dioxide, methane, etc.) are obtained.—J. H. L.

Kiln; Two-chambered shaft — for low temperature carbonisation of bituminous substances. Torfentgasung-Stauber G.m.b.H. G.P. 321,577, 29.1.19.

The dividing wall between the two chambers does not reach the top, so that a gas-collecting space common to the two chambers is formed into which a steam jet and the gas exit bells are built. The gas is removed from the apparatus before the temperature of decomposition of ammonia is reached.

—C. L.

Incandescence bodies consisting of a conductor embedded in rare earths; Preparation of —. J. F. Rahtjen. G.P. 321,304, 30.4.14. Addn. to 308,036 (J., 1918, 728 A).

A CONDUCTING wire is wound helically round a core of textile fibres and then covered with fabric and impregnated as in the main patent (*loc. cit.*) The wire cuts into the fibrous core and the latter then insulates the individual turns of the coil from one another and prevents short-circuiting. After burning off, the incandescence body is strengthened by heating for a short time by the electric current in a neutral atmosphere such as hydrogen.—W. J. W.

Incandescence cathode for use in [cathode ray] discharge tubes employing a high vacuum. Ges. für drahtlose Telegraphie m.b.M. G.P. 325,002, 13.12.17.

THE specific electronic emission of a tungsten cathode is increased by subjecting it to the action of hydrogen before producing the vacuum in the discharge tube. In this manner, employing a heating current of 2.1 ampères, the electronic emission is increased from 0.5 milliamp. to 150 milliamp.

—J. S. G. T.

Electrode for are lamps of high current density. Siemens u. Halske A.-G. G.P. 325,214, 19.5.18.

A SMALL carbon core is encased with aluminium.

—J. S. G. T.

Charcoal; Process for producing a highly active decolorising —. A. Knöplmacher, Assr. to The Chemical Foundation, Inc. U.S.P. 1,358,162, 9.11.20. Appl., 9.11.16.

SEE E.P. 102,608 of 1916; J., 1917, 589.

Electric arc devices; Enclosed —. The British Thomson-Houston Co., Ltd. From General Electric Co. E.P. 153,331, 16.11.16.

SEE U.S.P., 1,208,587 of 1916; J., 1917, 128.

Searchlights; Method of operating electrodes for —. E. A. Sperry. U.S.P. 1,357,827, 2.11.20. Appl., 22.12.15.

SEE E.P. 102,971 of 1916; J., 1918, 263 A.

III.—TAR AND TAR PRODUCTS.

Naphthalenesulphonic acids; Sparingly soluble salts of certain —. J. A. Ambler. J. Ind. Eng. Chem., 1920, 12, 1081—1085.

THE α - and β -naphthylamine salts of naphthalene β -, 1.5-, 1.6-, 2.6-, and 2.7-sulphonic acids have been prepared in crystalline condition, and their physical and chemical characters are described. All of them dissolve with difficulty in water, the 1.5 salt being the least soluble, and all are readily hydrolysed when boiled with water. Ferrous naphthalene- β -sulphonate, $\text{Fe}(\text{C}_{10}\text{H}_7\text{SO}_3)_2 \cdot 6\text{H}_2\text{O}$, crystallises in colourless glistening plates. It dissolves with difficulty in cold water, but is readily soluble in hot water. When heated at 150°—160° C. it loses its water of crystallisation. It is characterised by its unusually low refractive index (D)— n_D^{20} 1.500. (*Cf.* J.C.S., Jan., 1921.)—C. A. M.

Naphthalenesulphonic acids; Method for the detection of certain —. J. A. Ambler and E. T. Wherry. J. Ind. Eng. Chem., 1920, 12, 1085—1087.

FOR detecting the various sulphonic acids formed in the sulphonation of naphthalene the following tests are applied to a solution of the sodium salts of the acids. The presence of naphthalene- β -sulphonic acid is shown by precipitation of glistening crystals of the ferrous salt; these separate only after some time from dilute solutions. From the boiling filtrate a granular precipitate of α -naphthylamine naphthalene-1.5-disulphonate produced by means of a freshly-prepared solution of α -naphthylamine hydrochloride indicates the corresponding acid. The liquid is filtered hot, the excess of the base removed by titration with sodium hydroxide, and the solution cooled and filtered. The filtrate is heated to boiling and treated with a large excess of boiling β -naphthylamine hydrochloride, which will form a white precipitate of β -naphthylamine naphthalene-2.6-disulphonate if the corresponding acid is present. The solution is filtered rapidly while hot, and the filtrate cooled, and left for at least 1 hr. Should a jelly be formed the liquid is diluted, boiled, and again cooled. The salts are then separated, dried *in vacuo* at 100° C., and a small amount boiled with acetone, the liquid filtered and the filtrate cooled. The separation of crystals of β -naphthylamine naphthalene- α -sulphonate indicates the presence of α -sulphonic acid. The optical behaviour of the salts (see preceding abstract and J.C.S., Jan., 1921) is utilised to detect the presence of naphthalene-2.7- and 1.6-disulphonic acids.

—C. A. M.

Hydrocarbons. Darmois. See II A.

Waste roofing felt. Hawley and Kress. See IX.

PATENTS.

Tar, crude oil and the like; Separation of water and other impurities from —. J. Billwiller. G.P. 322,895, 7.1.17.

AFTER addition of electrolytes (bases, salts, etc.) the material is heated at a high temperature under pressure, and suitably stirred or centrifuged. Not only are the coagulated or colloiddally dissolved solid impurities removed, but the whole of the water is separated, and the ammonium salts etc. are decomposed so that the whole of the ammonia is evolved in the free state with the light oil and aqueous liquor in the subsequent distillation.

—J. H. L.

Tar; Producing resinous bases from —. E. Wirth, Assr. to The Chemical Foundation, Inc. U.S.P. 1,339,310, 1.5.20. Appl., 20.5.16. Renewed 6.8.19.

SEE G.P. 303,273 and 304,306; J., 1920, 184 A.

Anthraquinone derivatives; Manufacture of —. R. B. Ransford. From L. Cassella und Co. E.P. 153,055, 21.6.18

SEE U.S.P. 1,285,726 of 1918; J., 1919, 130 A.

Heat exchanger. G.P. 322,745. See I.

Preheater. U.S.P. 1,337,287. See IIa.

Ultramarine. E.P. 152,916. See XIII.

Coating composition. U.S.P. 1,356,380. See XIII.

IV.—COLOURING MATTERS AND DYES.

Extract of old fustic (morine) and quercitron extract (quercitrin); Differentiation of —. E. Justin-Mueller. Bull. Soc. Chim., 1920, 27, 844—846.

THESE two extracts appear quite different under the microscope, that of quercitron showing more or less agglomerated granules, while that of old fustic shows well-defined crystals. If a trace of either extract (dry or concentrated liquid) is dissolved in sulphuric acid (66° B., sp. gr. 1.84) and then diluted with water, the extract of old fustic remains bright orange-yellow, while that of quercitron is decolorised.—W. G.

PATENTS.

Azo dyestuffs; Manufacture of — and intermediate products therefor. O. Imray. From Soc. of Chem. Ind. in Basle. E.P. 149,553, 12.5.19.

NAPHTHALENEDISULPHOCARBOXYLIC acids containing a sulpho group in the 1-position are nitrated, the resulting 8-nitro-derivatives are reduced, the amino group replaced by a hydroxyl group, and the products subjected to partial fusion with alkali. The 1,8-dihydroxynaphthalenesulphocarboxylic acids obtained in this way yield mordant dyestuffs when coupled with diazo compounds, the azo group entering the 2- or 7-position without elimination of the carboxyl group (cf. U.S.P. 1,323,285 of 1919; J., 1920, 58 A).

4-Nitro-β-naphthol; Manufacture of —. G. T. Morgan, and British Dyestuffs Corp., Ltd. E.P. 152,437, 17.7.19.

2,4-DINITRO-α-NAPHTHYLAMINE, which may be obtained by the action of alcoholic ammonia at 220° C. on 2,1-dinitro-α-naphthol, or by the nitration and subsequent hydrolysis of aceto-α-naphthalide, is treated with nitrosyl sulphate, or sodium nitrite in concentrated sulphuric acid, and the solution added to water to precipitate 4-nitronaphthalene-1-diazo-2-oxide. On warming this diazo oxide with ethyl alcohol, either alone or in presence of a metallic catalyst such as zinc, copper, aluminium, or a zinc copper couple, or of a reducing agent such as hypophosphorous acid, it is converted into 4-nitro-β-naphthol, which is purified by extraction with hot water from which it crystallises in yellow needles, m.p. 120° C. 4-Nitro-β-naphthol couples with the diazo derivatives of the following compounds giving azo-derivatives which are useful as dyestuffs:—Sulphanilic acid, giving a dark red dye for wool or silk; paranitraniline-2-sulphonic acid, an orange dye for wool or silk; 4,4'-diminostilbene-2,2'-disulphonic acid and safranine, direct purple dyes for cotton or linen; paranitraniline and 2,4-dinitro-aniline, red ingrain dye on cotton; and primuline, a dark blue ingrain dye on cotton; and primuline, a reddish-brown developed dye on cotton.—G. F. M.

Sulphur dyestuff. M. Wyler, Assr. to Levinstein, Ltd. U.S.P. 1,358,490, 9.11.20. Appl., 27.3.19. SEE E.P. 124,589 of 1918; J., 1919, 318 A.

Medicinal preparations. G.P. 324,747. See XX.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Best fibres [nettles]; Retting of —. P. Kraiss. Z. angew. Chem., 1920, 33, 277—278. (Cf. J., 1919, 169 A, 530 A; 1920, 11 A, 443 A.)

BORAX cannot be used as a substitute for sodium bicarbonate because it largely hinders the retting of flax and nettles. At the end of the retting process, the liquors contain chiefly spores and but few pleotridae and rod bacilli. Nettle fibres cannot compete with the usual fibres of commerce, although they may be used in home spinning and weaving. The production of fibres suitable for spinning is carried out as follows:—Nettles are dried, the leaves removed, the stems retted in a 0.8% solution of sodium bicarbonate (if a 0.5% solution of sodium carbonate is used the retting takes longer) at 37° C., dried in the sun, and the bast fibres separated by heating. If the bast fibres be separated by heating with alkalis under pressure, a considerable loss of fibre occurs. Washing and bleaching of the fibres results in a loss of 30—35%. From 100 pts. of air-dried nettles is obtained 5.5 pts. of pure bleached nettle fibres.—A. J. H.

Wool and cotton fabrics; Analysis of mixed —. Duyk. Ann. Chim. Analyt., 1920, 2, 324—330.

THE following is recommended for adoption as a standard method for analysis of textile fabrics composed of a mixture of wool and cotton: From 10—15 g. of the material is cut up and dried at 100°—105° C., and then treated with suitable reagents to remove size, filling material, etc., and again dried and weighed. It is then heated with 2% sodium hydroxide solution in a covered vessel on the water bath to a maximum temperature of 90° C., and 20 mins. after that temperature is reached the cotton residue is washed with acidified water and then pure water, dried, and weighed. The amounts of cotton and wool are then calculated on the basis of the official limits for water, viz., 17% for carded wool, 18.5% for combed wool, 8.5% for cotton, and 10% for silk. For the separation of silk from wool, zinc chloride solution of sp. gr. 1.65 is used.—C. A. M.

Textiles of wool or mixtures of wool and silk; Method of reducing the permeability of —. G. Colombo. Giorn. Chim. Ind. Appl., 1920, 2, 234—237.

TEXTILES of wool or silk and wool are saturated with water, centrifuged to reduce the water content to, e.g., less than 50%, and exposed for 30 mins. in a closed chamber to the action of moist steam between 90° and 100° C. They are then dried within the same limits of temperature. The duration of the treatment required will vary with the size of the chamber, weight of the fabric, and moisture and permeability of the fabric. A woollen fabric thus treated required from 50 to 60 mins. for the filtration of 500 c.c. of water per sq. dem. under a constant pressure of 40 mm., whereas only 13 to 18 mins. was required prior to the treatment.—C. A. M.

Textiles; Laboratory apparatus for determining the permeability and heat-insulating value of —. G. Colombo. Giorn. Chim. Ind. Appl., 1920, 2, 167—168.

THE permeability of fabrics to water is determined by stretching the material across a metal wire framework which fits into a larger vessel so as to form a porous bottom, the overlapping edges being immersed in mercury seals. Water is maintained at a constant level above the stretched fabric, and the amount which filters through the material at a definite temperature is measured and expressed in vols. per sq. dem. of the fabric. In determining

the heat conductivity a cylinder 50 mm. in diam. is filled with mineral oil which can be heated electrically. The cylinder is closed by a cork through which passes a thermometer, by means of which it is suspended in a second metal cylinder with perforated walls, leaving a space of 15 mm. between the two cylinders. The fabric is stretched round the outer cylinder, and the apparatus placed under a bell jar. The time required for the oil to fall through a given range of temperature is then noted.—C. A. M.

Cellulose and textile fibres; Digestion of woody fibres with chlorine for the production of —.

P. Waentig and W. Gierisch. *Text.-Forschung*, 1920, 2, 69—79. *Chem. Zentr.*, 1920, 94, IV., 544.

For the production of cellulose from wood shavings of 0.2–0.3 mm. thickness the authors recommend a preliminary boiling for 1 hr. in 0.1 sodium hydroxide solution, then treatment with chlorine for several hours without heating, and after the removal of hydrochloric acid a final treatment in 1% sodium hydroxide solution at the ordinary temperature with stirring. Thick shavings and chips may be digested with chlorine under 6 atm. pressure. A preliminary heating with 1% alkali is necessary in the treatment of cereal straws. The chlorine process is not suitable for the production of textile fibres from jute and rushes, but it might be applicable to hemp, flax, ramie, and nettles.

—J. H. L.

Cellulose; Action of hydrogen peroxide on —. R. Haller. *Text.-Forschung*, 1920, 2, 79—81. *Chem. Zentr.*, 1920, 94, IV., 543—544.

Most vegetable textile fibres are disintegrated by the prolonged action of 30% hydrogen peroxide and eventually dissolve with formation of dextrose. A cellulose peroxide which reacts characteristically with potassium iodide is formed as intermediate product. The disintegration of the fibres is accelerated by certain cotton dyes and retarded by others. Cotton mordanted with chromium oxide is very rapidly broken down, whilst the simultaneous presence of aluminium has a retarding effect. It is possible that impregnation of laundry goods with alumina may protect them from injury from detergents containing hydrogen peroxide.—J. H. L.

Waste roofing felt. Hawley and Kress. See IX.

PATENTS.

[Pulp] digesters; Acid-charging system for —. F. J. Dooley. U.S.P. 1,338,496, 27.4.20. Appl., 23.5.19.

THE "acid" is fed into the digester at the bottom by means of a pump placed below the level of the liquid in the digester, but the pipe connecting the pump with the digester extends upwards and has a portion above the liquid level in the digester, and preferably above the top of the digester. The control valve is placed in this elevated portion of the pipe. After charging the digester, the valve can be disassembled and packed to render it fluid-tight for the cooking operation without risk of escape of "acid" from the digester.

Pyroxylin; Solvents of — and compositions containing the same. E. M. Flaherty, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,356,410, 19.10.20. Appl., 11.9.18.

The solvent contains *n*-butyl alcohol, a hydrocarbon, and an alcohol of the paraffin series.

—B. V. S.

Fibres; Method of making —. A. R. Fuller. U.S.P. 1,357,206, 26.10.20. Appl., 10.2.20.

FIBRES are obtained from a viscous material such as a solution of cellulose xanthate (viscose) by forcing

the material by means of a centrifugal pump through fine apertures into a coagulating liquid.

—B. V. S.

Sulphite-cellulose waste liquors; Utilisation of —. F. Siemens. G.P. 325,756, 25.12.19.

THE bisulphites and lignosulphonic acids are reduced by treating the liquor with carbon monoxide. Calcium bisulphite yields calcium hydrosulphide and from this hydrogen sulphide may be produced and converted into sulphur or sulphur dioxide. The precipitated wood constituents are directly fermentable.—J. H. L.

Paper; Method and machine for making —. Great Northern Paper Co., Asses. of C. E. Pope. E.P. 137,539, 8.1.20. Conv., 26.3.18.

IN starting the machine or in case of a break, the paper is carried from the drying rolls to and through the calendars by cutting a lead strip and directing it through the machine by a series of doctors and air-blasts; subsequently the strip is widened to the width of the web of paper.

—L. A. C.

Viscose; Process for the manufacture of —. L. Lilienfeld, Assr. to The Chemical Foundation, Inc. U.S.P. 1,355,985, 19.10.20. Appl., 19.1.12. Renewed 16.3.20.

SEE F.P. 439,040 of 1912; J., 1912, 581.

Cellulose; Manufacture of new products [carbanic esters] derived from —. P. E. C. Goissedet. U.S.P. 1,357,450, 2.11.20. Appl., 4.3.19.

SEE E.P. 130,277 of 1919; J., 1919, 714 A.

Container for alkaline substances. U.S.P. 1,355,976. See I.

Stain for wood. G.P. 325,731. See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENT.

Textile materials treated with Turkey-red oil or the like; Prevention of — acquiring a yellow tint. C. H. Boehringer Sohn. G.P. 325,470, 10.5.19.

THE textile materials are treated with Turkey-red oil or the like in the presence of salts of hydrolysed or non-hydrolysed bile acids. Thus, 2–3% of sodium cholate, or 4–5% of potassium glycocholate may be used.—J. S. G. T.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Nitric acid manufacture by Schönher's process; Experiments on —. F. Kodera, I. Kitawaki, and B. Yokoyama. Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Tokyo), 1920, 23, 1010–1015.

THE diameter of the inner tube of the furnace was 6 in. and the total length of the furnace about 20 ft. In the case when the are was most stable and every other condition was comparatively good the following data were obtained:—Voltage supplied from transformer, about 5000 volts; air quantity supplied per minute, 2000 l.; arc voltage, 2700 volts; intensity of current, 30 ampères; power required, 74 kw.; arc length, 13 ft.; concentration of nitric oxide produced, 1.8% by volume. The gas from the furnace was passed through a multitubular boiler, a water cooler, an oxidation chamber, and three absorption towers. Nitric acid of 42% concentra-

tion could be prepared by absorbing with water, but it would be more economical to stop the absorption when the concentration of the acid has reached about 35%. The theoretical yield of acid was 82 g. per 1 kw.-hr., while the yield actually obtained was about 65 g. Of the energy supplied to the furnace, about 3% was utilised to form nitric oxide, 60% was consumed by the cooling water of the furnace, and 12% by the boiler.—K. K.

Calcium cyanamide; Investigation of preparation and properties of — N. Kameyama. J. Coll. Eng., Tokyo Imp. Univ., 1920, 10, 173—207, 209—247, 249—263.

WHEN calcium cyanamide is prepared by igniting urea with lime, calcium cyanate is formed as an intermediate product at temperatures below 300° C., transformation into cyanamide taking place slowly at 340°—360° C., and rapidly at or above 450° C. The heating should be of short duration to prevent oxidation of the cyanamide; 5—7 mins. at 800° C. is sufficient. The percentage of cyanamide found in the mixed product of calcium cyanamide, calcium carbonate, and calcium oxide depends on the initial proportions of urea and lime. Mixtures of these substances in the proportions of 1:2, 1:1½, and 1:1, give products containing 16.5, 24, and 34% of calcium cyanamide, and 92—98% of the theoretical yield is attained. Ignition in a current of nitrogen gives a quantitative yield. If dicyanodiamide is used instead of urea, although a smaller proportion of the theoretical amount is obtained, the product contains more calcium cyanamide. Even at 300°—400° C. no cyanate is formed. Equal parts of dicyanodiamide and lime should be taken, and in order to decompose the calcium carbonate in the cyanamide mixture the ignition is best conducted at 900°—1000° C. for about 10 mins. The product will contain 50—53% of calcium cyanamide but this amount is increased up to 90% by reheating with excess of dicyanodiamide, and by further heating in a current of nitrogen 99% is reached. The reactions of calcium cyanamide, prepared from dicyanodiamide and lime, with oxygen, carbon dioxide, and carbon monoxide, were investigated on a sample of composition: CaCN_2 , 92.3%; CaCO_3 , 3.97%; CaO , 3.73%. At 420°—450° C. oxygen reacts rapidly with formation of calcium carbonate and nitrogen, but with no separation of carbon; at 840°—900° C. the reaction products consist of calcium oxide, carbon dioxide, and nitrogen. With carbon dioxide decomposition occurs between 700° and 1070° C. with no separation of carbon, the products being carbon monoxide, nitrogen, and calcium oxide or carbonate according to the temperature. Above 1110° C. the reaction is very rapid, and carbon is liberated, probably owing to the decomposition of carbon monoxide. In absence of a catalyst carbon monoxide is without action on calcium cyanamide up to 1000° C., but if carbon dioxide is produced from the monoxide the cyanamide may be indirectly oxidised by it. At temperatures above 1140° C., carbon monoxide decomposes calcium cyanamide, even without aid of catalysts, calcium oxide, carbon, and nitrogen being formed. The heat of combustion of pure calcium cyanamide was found to be 154,730 cals. per g.-mol. at constant pressure; and, calculated from this, the heat of formation is 91,480 cals. from crystalline carbon (diamond), or 94,820 cals. from amorphous carbon.—W. J. W.

Ammonium sulphate; The Feld process for production of — F. Raschig. Z. angew. Chem., 1920, 33, 260—262.

The author has investigated the reactions which occur in Feld's process for the production of ammonium sulphate from the ammonia and hydrogen sulphide present in coke-oven gas (J., 1912,

430). Formation of polythionates by the action of sulphur dioxide on thiosulphate only takes place with very concentrated solution; with dilute solutions sulphite and sulphate are chiefly produced. Addition of a trace of sodium or potassium arsenite ensures the conversion of thiosulphate into tri- and tetrathionate, pentathionate being a probable intermediate. The reactions are based on proportions of ammonia and hydrogen sulphide of 2:1. An excess of either gas retards the conversion, and necessitates in one case the introduction of more hydrogen sulphide, and in the other case its oxidation to sulphur. Careful control and testing of the gases are essential for successful running. (Cf. J.C.S., Jan., 1921.)—W. J. W.

Ammonium sulphate; Melting point and boiling point of — E. Jäcke. Z. angew. Chem., 1920, 33, 278—280.

THE equilibrium between sulphuric acid, ammonia, ammonium bisulphate, and ammonium sulphate has been investigated and the results are shown graphically. Under atmospheric pressure, ammonium sulphate decomposes completely at 357° C. Pure ammonium bisulphate has m.p. 251° C. and b.p. 490° C. (cf. J., 1895, 629; 1896, 3; 1920, 485 A). The melting point curve for mixtures of the acid and normal ammonium sulphates has an eutectic point at 195° C. (Cf. J.C.S., Dec.)—A. J. H.

Sodium hypobromite; Catalytic decomposition of an alkaline solution of — by copper sulphate. Antagonistic action of iodine. P. Fleury. Comptes rend., 1920, 171, 957—960.

THE presence of copper sulphate causes a steady decomposition of an alkaline solution of sodium hypobromite, thus interfering with the estimation of urea by this reagent. The rate of decomposition increases rapidly with the concentration of the copper salt. The presence of 1 pt. of copper in 100,000 causes the decomposition of 42% of the hypobromite in 24 hrs. at 36°—37° C. The addition of 1 pt. in 1000 of potassium iodide or potassium iodate to the solution completely inhibits this catalytic decomposition of the hypobromite solution.—W. G.

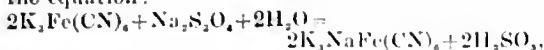
Sodium hydrosulphite; Valuation of — R. E. Crowther and A. D. Heywood. J. Soc. Dyers and Col., 1920, 36, 279—281.

TO 800 c.c. of air-free water in a litre flask 40 c.c. of 40% formaldehyde solution and 10 c.c. of 30% sodium hydroxide solution are added, and whilst the contents of the flask are vigorously swirled, 8—12 g. of the hydrosulphite powder, weighed by difference from a stoppered weighing bottle, is rapidly added through a dry funnel, and the volume of the solution finally adjusted to 1000 c.c. This formaldehyde hydrosulphite solution is then run from a burette into 25 c.c. of standard methylene blue solution (about 14 g. per l.), to which 10 c.c. of glacial acetic acid and 50 c.c. of water have been added, the liquid being gently boiled during the titration. The end point is quite sharp, and is indicated by a change from bluish-green to yellow. For very accurate work a stream of carbon dioxide is passed through the flask containing the methylene blue solution, but for technical purposes this is not necessary. Alternatively the formaldehyde-hydrosulphite solution may be added to an excess of methylene blue solution, and the excess titrated with titanous chloride solution.—G. F. M.

Hydrosulphite; Volumetric determination of — R. Formhals. Chem.-Zeit., 1920, 44, 869.

ABOUT 0.5 g. of hydrosulphite is dissolved in 50 c.c. of air-free water and the solution is titrated with N/10 potassium ferricyanide solution in presence of a few drops of ferrous ammonium sulphate solu-

tion as indicator, the end point being indicated by the formation of a permanent blue-green coloration of Turnbull's blue. The reaction is expressed by the equation:—



but in practice it is preferable to standardise the ferrieyanide solution by titration with pure hydrosulphite.—G. F. M.

Gels of earth-acids [titanic, niobic, and tantalic acids]; *Solubility of* — in mixtures of hydrogen peroxide and acid. O. Hahn and H. Gille. Z. anorg. Chem., 1920, 112, 283–292.

According to Weiss and Landecker (J., 1909, 1169), titanic, niobic, and tantalic acids can be separated from most other elements by dissolving the freshly precipitated acids, whether precipitated from acid or basic solutions, in acid hydrogen peroxide and re-precipitating by boiling with sulphurous acid. It is now shown that whilst titanic and niobic acids are practically completely soluble in acid hydrogen peroxide solution, tantallic acid, when precipitated hot, is practically insoluble, and when precipitated cold is very slightly soluble. In mixtures, tantallic acid lowers the solubility of niobic acid. Titanic acid may increase the solubility of tantallic acid or may have its own solubility reduced, according to the conditions of precipitation and the after-treatment of the precipitate. Weiss and Landecker's method therefore fails in presence of tantallic acid. Titanic acid solutions in hydrogen peroxide, examined with the ultramicroscope, appear to be truly molecular, whilst those of niobic and tantallic acids are colloidal.—E. H. R.

Iodic acid; Microchemical reactions of —. A. Bolland. Comptes rend., 1920, 171, 955–957.

A DESCRIPTION is given of the appearance under the microscope of the precipitates given by iodic acid with the following reagents:—Thallium nitrate, silver nitrate, barium chloride, strontium acetate, calcium acetate, rubidium chloride, caesium sulphate, rubidium cadmium chloride, potassium chloride, sodium acetate, ammonia, manganous chloride. The first two reagents give a precipitate with iodic acid diluted to 1 in 5000, the next with 1 in 2500, the next three with 1 in 300, whilst for the remainder the dilution should not be more than 1 in 80 and preferably about 1 in 20.—W. G.

Salts; Action of water on a mixture of —. Determination of the number of independent constituents. C. Raveau. Comptes rend., 1920, 171, 913–916.

A CRITICISM of Dubreuil's rule (Comptes rend., 1920, 171, 720) and a theoretical discussion of Rengade's work (J., 1917, 961) on the purification of salts by fractional crystallisation, in the light of this rule.

—W. G.

Sulphur monochloride; Determination of — in admixture with mustard gas. W. A. Felsing, S. B. Arenson, and F. J. Kopp. J. Ind. Eng. Chem., 1920, 12, 1054–1056

THE method is based upon results which show that sulphur monochloride reacts with sodium iodide as in the equation— $S_2Cl_2 + 2NaI = I_2 + 2S + 2NaCl$. About 25 c.c. of approximately N/10 sodium iodide solution and 10 c.c. of carbon tetrachloride are introduced into a 250 c.c. flask, the flask weighed, 2 c.c. of the mixture of mustard gas and sulphur chloride introduced, and the flask again weighed. A measured excess of sodium thiosulphate solution is then added, and the excess titrated with standard iodine solution. The results are accurate within 0.5% at higher concentrations (20–30%), and to about 0.1% with lower concentrations (2–5%) of sulphur monochloride.—C. A. M.

"Norit" and blood charcoal. Kruyt and Van Duin. See III.

Nitrites. Thévenon. See XIXa.

Sulphuric acid. Winkler. See XXIII.

PATENTS.

Sulphurous gases; Treatment of — in the chamber sulphuric acid process. Metallbank und Metallurgische Ges. A.-G. G.P. 300,061, 6.7.16.

THE sulphurous gases are employed for atomising the nitrous vitriol before it enters the Glover tower. The operation of the Glover is thus improved, and gases containing 2 to 4% SO_2 may be utilised, at a temperature of 100° to 200° C.

—W. J. W.

Sodium perborate; Manufacture of —. Frederikstad Elektrokem. Fabr. A./S. E.P. 139,753, 6.1.20. Conv., 4.3.19.

IN the electrolytic manufacture of sodium perborate from borax and soda, the sodium bicarbonate produced is neutralised by addition of sodium peroxide, yielding a further quantity of perborate.—C. I.

Sulphate of ammonia; Manufacture of —. R. Lessing. E.P. 152,766, 21.7.19.

THE impurities which colour commercial ammonium sulphate exist as a fine deposit on the surface of the crystals. A white salt is obtained by agitating the product from the saturator with an upward current of slightly acid saturated ammonium sulphate solution at the same temperature as the salt. The dirty solution is filtered and used again and the salt centrifuged and neutralised.

—C. I.

Artificial pearls and jewellery; Manufacture of —. K. Murakami. E.P. 152,914, 11.2.20.

THE crystalline lenses of fishes' eyes are boiled with water until solid and the white surface layer removed by treatment with dilute caustic soda solution. The solid transparent lenses, dyed if desired, are rendered opaque by deposition of such salts as lead carbonate or barium sulphate within them. They are then polished and coated with liquid paraffin or other preservative.—C. I.

Hydrogen and zinc oxide; Process of making —. R. H. McKee. U.S.P. 1,355,904, 19.10.20. Appl., 9.2.20.

PURE zinc oxide, and hydrogen free from carbonaceous impurities, are prepared by allowing substantially pure zinc vapour to react with steam in sufficient excess to secure substantially complete oxidation of the zinc.—H. S. H.

Alkali-metal phosphates; Process of making hydrogen-containing —, and a salt produced therein. S. S. Sadler. U.S.P. 1,357,120, 26.10.20. Appl., 30.1.19. Renewed 10.9.20.

CALCIUM monohydrogen phosphate is treated with the alkali salt of an acid the calcium salt of which is insoluble.—C. I.

Sulphur; Extraction of — from sulphide ores. H. C. Pedersen. E.P. 152,887, 10.12.19.

INOX pyrites or other sulphide ore is roasted in a shaft furnace with a quantity of coke insufficient for reduction of the ore to metal, the air supply at the bottom being so regulated that sulphur vapour distils off.—C. I.

Nitric acid; Manufacture of —. A. Henwood. E.P. 153,362, 29.7.19.

SEE U.S.P. 1,347,159 of 1920; J., 1920, 596 A.

Chrome alum; Process for manufacturing —. G. H. Hultman. E.P. 138,594, 3.11.19. Conv., 6.2.19.

SEE U.S.P. 1,343,725 of 1920; J., 1920, 545 A.

Ammonia; Methods and apparatus for the oxidation of —. C. Toniolo. E.P. 137,036, 1.12.19. Conv., 23.12.18.

SEE U.S.P. 1,318,936 of 1919; J., 1919, 945 A.

Basic sulphate of zirconium; Manufacture of —. H. Wade. From Pennsylvania Salt Manufacturing Co. E.P. 153,113, 8.8.19.

SEE U.S.P. 1,316,107 of 1919; J., 1919, 819 A.

Aluminium hydrate; Process of and apparatus for precipitating —. R. S. Sherwin. E.P. 153,352, 8.7.19.

SEE U.S.P. 1,314,709—10 and 1,315,045 of 1919; J., 1919, 818 A.

Alumina; Process of producing — from clay and similar materials. V. M. Goldschmidt and G. Ravner, Assrs. to Det Norske Aktieselskab for Elektrokem. Ind. of Norway. U.S.P. 1,357,089, 26.10.20. Appl., 23.12.18.

SEE E.P. 122,623 of 1918; J., 1919, 764 A.

[Alkali] silicates; Manufacture of readily soluble —. F. J. Phillips and E. J. Rose. U.S.P. 1,357,183, 26.10.20. Appl., 22.6.20.

SEE E.P. 151,339 of 1919; J., 1920, 748 A.

Nitrogen products; Apparatus for the production of —. C. T. Thorssell and H. L. R. Lundén, Assrs. to Aktiebolaget Kvaefveindustri. U.S.P. 1,357,196, 26.10.20. Appl., 13.8.19.

SEE E.P. 145,299 of 1919; J., 1920, 570 A.

Alkali carbonate and hydraulic cement; Process of producing — from alkaliiferous silicate minerals. E. W. Jungner. U.S.P. 1,357,873, 2.11.20. Appl., 9.9.16.

SEE E.P. 103,290 of 1916; J., 1918, 60 A.

Molybdenum compounds. E.P. 131,897. See X.

VIII.—GLASS; CERAMICS.

China clay; Shrinkage, porosity, etc., of — after being fired at various temperatures. E. M. Firth, F. W. Hodkin, and W. E. S. Turner. J. Soc. Glass Techn., 1920, 4, 264—267.

A SAMPLE of china clay of normal composition required 49.5% of water to bring it to a moulding consistency. On being fired its colour changed from creamy white to a slight pink between 600° and 1100° C., but at 1200° C. it was dead-white. The fracture of pieces fired at 1400° C. showed a slight glaze, and at 1500° C. this was more pronounced and the colour bluish-white. The shrinkage remained below 5% up to 1000° C. and then rose very rapidly to 22% at 1500° C. The porosity was 50% at 600° C., rose to a maximum (56%) at 900° C., and then fell rapidly to 2.4% at 1500° C.—A. B. S.

Transformations of quartz. Perrier and Wolfers. See X.

PATENTS.

Glass; Drawing —. R. L. Frink. E.P. 144,629, 2.6.20. Conv., 12.6.19.

A HEAD through which air can be supplied and which can be moved into any desired position, is mounted on a hollow standard passing through a cylindrical well of refractory material in the drawing pot. The circular jet of air impinges upon the inner walls of the hollow glass cylinder slightly

below the point of congealing of the glass. The volume, direction, and force of the air can be varied by altering the jet openings and inclining the head, and so the diameter of the cylinder and the thickness and uniformity of its walls can be controlled.—H. S. H.

Non-fragile glass; Manufacture of a — using natural silicates such as micaceous mineral, asbestos, and the like. P. B. Crossley. E.P. 152,780, 22.7.19.

A MIXTURE of the finely divided silicious material and powdered glass or a similar vitreous material is heated at a temperature high enough to melt the glass but insufficiently high to disintegrate the silicious material, until the whole is transformed into a more or less viscid mass. The product may be rolled or forged, or the temperature may then be raised until the mass liquefies and can be cast into moulds. By the addition of colouring matter, a coloured product may be obtained, or by varying the proportions the product may be transparent, semi-transparent, or opaque. The material can be turned and adheres tenaciously to metals and is thus suitable for the manufacture of electric and heat insulators, and for other purposes.—L. A. C.

Refractory article. S. C. Linbarger, Assr. to The Carborundum Co. U.S.P. 1,356,211, 19.10.20. Appl., 21.6.19.

A REFRACTORY article for use in preventing discoloration of ceramic ware in firing, comprises a body containing silicon carbide with a protective coating free from silicon carbide.—H. S. H.

Refractory material. J. L. Ohman, Assr. to N. K. Farish. U.S.P. 1,356,939, 26.10.20. Appl., 1.11.17.

A NON-RECRYSTALLISED refractory material consists of silicon carbide and crystalline graphite.
—H. S. H.

Tunnel-kilns; Method and means for firing —. J. B. Owens. U.S.P. 1,355,906, 19.10.20. Appl., 24.8.18.

A COMBUSTION chamber, communicating at its side with the interior of the tunnel, is provided with means for deflecting the heat and products of combustion into the tunnel. The combustion chamber is fired in the direction of its length.—H. S. H.

Tunnel-kilns; Method of firing — and convertible furnace therefor. J. B. Owens. U.S.P. 1,356,396, 19.10.20. Appl., 23.1.17.

HEAT is supplied to a combustion chamber communicating with the burning chamber from any one of a number of independent sources of fuel differing in kind and extraneous to the combustion chamber.—H. S. H.

Tunnel-kilns; Firing means for —. J. B. Owens. U.S.P. 1,356,397, 19.10.20. Appl., 14.8.18.

A TUNNEL kiln has a multiple firing zone with which combustion chambers communicate at different points of its length. Means for firing the combustion chambers in a direction other than towards their communication with the firing zone are provided. The hot products of combustion are delivered from the chambers on to the lower portions of the ware in the tunnel and are then caused to flow in the same direction through the firing zone. By the arrangement described the goods may be subjected to a relatively long "soaking" heat at the temperature necessary to burn them.—H. S. H.

Tunnel-kiln; Multiplex —. J. B. Owens. U.S.P. 1,356,398, 19.10.20. Appl., 18.2.19.

TRACKS along which cars carry the ware extend side by side along the length of the tunnel kiln.

Closed partitions rise between the car platforms and extend throughout the length of the kiln between the sets of tracks, and those parts of the tunnel which are below the cars can be sealed from those parts which are above the cars. The products of combustion pass lengthwise along the tunnel.—H. S. H.

Ceramic material; Grouped direct-fired furnaces for —. H. Herda. G.P. 324,977, 25.3.19.

COOL air from the cooling furnace of a pair of furnaces is mixed with waste gas from the fired furnace of the pair, in a mixing chamber situated in a common channel placed beneath the furnaces, and the mixture is passed to a preheating chamber. Carbon or soot in the gases is burnt, and any light ash carried by the mixture is deposited in the mixing chamber.—J. S. G. T.

Kiln; Annular —. H. Fehmer. G.P. 325,298, 17.7.18.

THE waste gases from the smoking zone and the firing zone are discharged to the chimney stack through separate flues. Warm gases derived from the cooling and preheating zones are delivered through separate flues to the smoking and drying zone; this air is then in part discharged and in part to the pre-smoking zone.—J. S. G. T.

Tunnel muffle furnace; Heating of —. C. Schürtler. G.P. 325,299, 29.11.17.

THE firing zone, smoking zone, and pre-smoking zone are heated by the furnace gases in the usual manner, but the zone in the immediate neighbourhood of the furnace is heated only by air from the tunnel which has been preheated in the cooling zone and then heated to a high temperature in the firing zone; this air is then in part discharged and in part delivered to the smoking zone.—J. S. G. T.

Glazed or enamelled sheets of iron, steel, etc.; Production of —. A. Meyer. G.P. 325,673, 15.3.13.

THE ground coat of enamel is overlaid with successive opaque layers of enamel composed of silica, aluminium silicate, lead oxide, and an alkali such as sodium borate. Powdered rutile in variable proportion is added to the successive layers in order to increase or diminish their respective plasticities.—J. S. G. T.

Copper deposit on glass etc. E.P. 152,835. See X.

IX.—BUILDING MATERIALS.

Portland cement and puzzuolana; Mixtures of —. F. Ferrari. Giorn. Chim. Ind. Appl., 1920, 2, 238—244.

THE results of comparative tests with pure Portland cement and sand (1:3), and mixtures of Portland cement and puzzuolanas of various origin with sand have shown that puzzuolana and similar materials act as a corrective to the excessive basicity of hydrated calcareous cements of the Portland type, to which cause must be attributed the permeability to dissolved salts of set cements of the latter type. On the other hand, mixtures of Portland cement and puzzuolana, which are more or less rich in calcium aluminates, do not offer sufficient resistance to the action of sea water, but are suitable for all purposes where the cement will not be submitted to the continual action of sulphates and chlorides. Cement made from Portland cement, puzzuolana, and sand in such proportions that the quantity of lime formed on hydration corresponds to the amount which will be fixed by the other ingredients (puzzuolana and sand) is practically impermeable to dissolved salts, and is stronger than Portland cement.—C. A. M.

Waste roofing felt; Recovery of —. L. F. Hawley and O. Kress. Paper, 1920, 27, 18.

WASTE roofing felt, which is at present burnt under boilers at the factories, was extracted with gasoline to ascertain whether a recovery of the material would be practicable on the commercial scale. The waste, cut into pieces about 1 in. square, was extracted three times in succession with gasoline of b.p. 90° to 110° C. in a closed apparatus fitted with a steam-coil and a reflux condenser. At the end of each extraction, the solution was run off from the bottom of the vessel. About 50–60% of pitch, referred to the original material, was removed in this way, and although a considerable amount of matter soluble in gasoline and chloroform remained in the extracted material, this did not interfere with the opening of the latter or with the running of the stock on the paper machine. In the case of shingle stock, the rock could be readily separated after extraction by mechanical means. For a commercial process, four extractions with 350 galls. of solvent per ton of material should yield approximately the same results as those obtained in the laboratory trials.—L. A. C.

PATENTS.

Vesicular product and method of making such product. U. W. Boynton and R. J. Wig. E.P. 153,030 16.4.19.

A SHALE or clay rich in iron and other fluxes (except lime and magnesia) and containing carbon, sulphur, or other substances which at proper temperatures will cause the production of gas, is ground, formed into any required shapes, and heated to incipient fusion. The gases generated cause the material to expand and become full of small cells. The product is strong and tough, has a low apparent specific gravity, is a non-conductor of heat and a good electrical insulator, and is substantially impervious to water under ordinary conditions, as the various cells are not in connexion.—H. S. H.

Moulded articles from acid-proof cement; Manufacture of —. W. E. Windsor-Richards. E.P. 153,017, 21.6.19.

GROUND glass, sand, or quartz is mixed with clay, asbestos fibre, and a solution of sodium silicate, and the wet mixture pressed into moulds. After removal from the moulds, the articles are heated for about 12 hrs., the temperature being gradually raised to 200° C., and are then impregnated with any suitable acid-proof bituminous medium.—H. S. H.

Shaft furnace [for burning powdered limestone]. F. Müller. G.P. 323,339, 20.1.16.

THE furnace has a vertical preheating chamber and a horizontal burning chamber, the horizontal or slightly inclined floor of the latter being formed by a mechanical conveyor which transports the powdered material through the chamber in thin layers.—J. S. G. T.

Weather-resisting building material; Production of —. M. Künkele. G.P. 325,777, 26.9.19.

A WEATHER-resisting building material with properties resembling those of cement is produced by firing together broken stones, pyrites, clinker, and trass or material with similar properties, together with shale, to which, if it contains but little chalk, some chalk is added.—J. S. G. T.

Orysalt [cement] compositions and processes of making same. C. Catlett. E.P. 126,646, 9.5.19. Conv., 9.5.18.

SEE U.S.P. 1,282,188 of 1918; J., 1919, 17 A.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Thermal analysis; Sensitive method of — and the transformations of quartz, iron, and nickel. A. Perrier and F. Wolfers. Arch. Sci. Phys. Nat., 1920, 125, 372—381.

A NEW differential method is described for studying heat transformations. Two identical specimens of the material to be examined were suspended in an electric furnace at different levels, so that there was always a small temperature difference, preferably about 10° , between them. In each specimen was inserted a thermo-couple, by which the temperatures were read during heating or cooling, and the temperature differences were plotted on a curve against time. At temperatures corresponding with changes of state in the materials, marked sinuositics appeared on the curve. By this means slight thermal effects were detected which are not apparent on an ordinary cooling curve. In the case of quartz the α - β change was shown by a very prominent sinuosity which appears to indicate a change of latent heat. With soft iron a very complex curve was obtained which, besides the usual thermal changes, showed irregularities at about 800° C. and just above 1100° C. The former appears to correspond with a change in magnetic susceptibility observed by Weiss and Foëx (J. de Phys., 1911. (5). I., 274, 744, 805), whilst the latter appears not to have been observed before. Nickel gave a simpler curve than iron. Two new thermal transformations were discovered, however, one at about 460° , which corresponds with the magnetic change observed by Weiss and Foëx (*loc. cit.*), the other near 890° C., corresponding with a discontinuity of Curie's constant observed by Bloch (Thesis, Zurich, 1912).

—E. H. R.

Corrosion of iron in sulphuric acid; Effect of chromium compounds on the —. G. W. Heise and A. Clemente. Philippine J. Sci., 1920, 16, 439—446.

THE influence of the addition of graduated amounts of potassium bichromate on the corrosion of iron in sulphuric acid of varying strengths has been investigated. With both $N/2$ and $N/1$ sulphuric acid corrosion increased in proportion to the amount of bichromate added up to a maximum at which passivity was reached, after which corrosion decreased. With $N/2$ acid the passive condition was obtained with bichromate concentrations above 19 g. per l.; with $N/1$ acid the results were irregular, but in individual cases passivity was observed at a concentration of about 50 g. per l. In the case of $2N$ sulphuric acid, addition of potassium bichromate even up to its saturation point failed to bring about a passive state; corrosion increased without subsequent diminution. The addition of a small amount of potassium bichromate or of potassium chromate to 10% sulphuric acid increased the corrosion of iron at first, but when reduction of the chromium salts had taken place, the rate of corrosion did not differ from that in sulphuric acid alone. Chromium compounds such as the sulphate, introduced as chromic hydroxide, and chloride, apparently exert no influence on the rate of corrosion.—W. J. W.

Steel; Determination of vanadium in —. E. Jaboulay. Rev. Mét., 1920, 17, 627—629.

ONE g. of the metal (or 0.5 g. if more than 2% V is present) is dissolved by heating with 50 c.c. of dilute sulphuric acid, the solution is oxidised with nitric acid, and any insoluble black residue is dissolved by adding small successive quantities of permanganate solution to the boiling mixture; the solution is then diluted to 200 c.c., boiled, filtered, and, if chromium is present, the cold filtrate is titrated

with ferrous sulphate and permanganate solutions. The solution is then diluted to at least 300 c.c., and $N/10$ ferrous sulphate solution is added until present in slight excess (about 0.5 c.c.), using ferricyanide as external indicator; the mixture is further diluted to 500 c.c., the excess of ferrous sulphate is oxidised by the addition of 1 c.c. of $N/10$ bichromate solution, and the vanadyl salt is titrated with permanganate solution. The number of c.c. of $N/10$ permanganate solution used is multiplied by 0.511 to obtain the percentage of vanadium.—W. P. S.

Chromium and tungsten steels containing double carbides. P. Oberhoffer and K. Daeges. Stahl u. Eisen, 1920, 40, 1515—1516.

THE influence of chromium and tungsten on the saturation limits of carbon in γ -iron was investigated by microscopical examination. The alloys were made from pure materials, and to ensure obtaining equilibrium conditions they were thoroughly annealed at 800° C. for 14 hrs., or in the case of alloys containing a small amount of eutectic at 1100° C. for a longer period. After annealing, the steels were forgeable, specially those in the martensitic region of Guillet's structural diagram (*cf.* J., 1910, 953). Steels with 0.3—0.5% C and 15—20% Cr showed after suitable treatment great resistance to oxidation and attack by acids. In the chromium steels the eutectic was scarcely distinguishable from ledeburite, but clearly distinguishable in the tungsten steels. The results obtained show that the saturation limit of carbon falls from 1.7% to about 0.9% in presence of 2% Cr or to 0.8% in presence of 2% W, and more slowly to about 0.1% at 13.8% Cr or 0.13% at 12% W. On appearance of the eutectic, extra care must be given to the choice of forging temperature. The degree of working necessary to obtain uniform distribution of the carbide and destruction of the cellular structure of the eutectic is proportional to the amount of eutectic present.—T. H. Bu.

Arsenic and phosphorus; Determination of — in metallurgical products. I. Compagno. Giorn. Chim. Ind. Appl., 1920, 2, 493—497.

THE distillation method of Lundin and Blair (Carnot, *Méthodes d'anal. des fontes, des fers, et des aciers*, 1895) is simplified, and copper is used as the reducing agent. Five g. of the finely divided metal is dissolved in dilute (1:1) nitric acid, with the subsequent addition of 10 c.c. of sulphuric acid. The solution is evaporated, and the residue calcined, ground, and again calcined until white fumes appear. The mass is dissolved in 200 c.c. of hydrochloric acid, and transferred to a distillation apparatus, and, after the introduction of 5 g. of pure copper turnings, the arsenious chloride is distilled into 300 c.c. of water chilled in ice-water. The arsenic in the distillate is precipitated cold as sulphide, the excess of hydrogen sulphide removed by means of carbon dioxide, and the arsenic sulphide collected in a Gooch crucible, washed with water, alcohol, and carbon bisulphide, dried, and weighed. For the determination of phosphorus when arsenic is present in any considerable quantity, an aliquot portion of the distillation residue is filtered from silica, the filtrate oxidised by means of nitric acid and potassium permanganate, and the phosphorus determined by one of the usual methods. If arsenic is not to be estimated, it may be removed by volatilisation by the action of hydrobromic acid on the hydrochloric acid solution of the alloy, or after reduction with pure copper.

—C. A. M.

Hardness. (1) Haigh. (2) Innes. (3) Primrose. See XXIII.

Lead in ores etc. Hastings. See XXIII.

PATENTS.

Iron composition. G. G. Marx. U.S.P. 1,339,469, 11.5.20. Appl., 16.6.19.

A FINE-GRAINED, soft, scaleless cast iron is made by melting a charge consisting of pig-iron (35%), scrap iron (60%), and a clay product, *e.g.*, firebrick (5%), to which may be added 5% of steel.

Nickel-steel; Manufacture of — P. F. Cowing, Assr. to H. F. Chappell. U.S.P. 1,356,312, 19.10.20. Appl., 16.6.20.

AN alloy of nickel and copper, obtained from nickel-copper matte, is dissolved electrolytically, the copper in the resulting solution is replaced by iron or by iron and nickel, and afterwards an alloy of nickel and iron is deposited from the solution electrolytically and incorporated with steel to produce nickel-steel.—J. H. L.

Molybdenum compounds; Extraction of — from yellow lead ores [wulfenite]. Deutsche Molybdaen-Werke G.m.b.H. E.P. 131,897, 27.8.19. Conv., 23.5.16. (Cf. E.P. 131,898; J., 1920, 724A.)

FINELY powdered ore is agitated with an excess of an aqueous solution of an alkali sulphide, polysulphide, or hydrosulphide, either hot or cold, and with or without pressure. The solution, which contains sulphomolybdate, is treated with a fresh charge of ore or with a lead salt or ferric hydroxide to remove the sulphur and leave a solution of alkali molybdate.—A. R. P.

Zinc sulphide ores; Roasting of — preparatory to leaching. Electrolytic Zinc Co. of Australasia Proprietary, Ltd. E.P. 131,825, 9.10.19. Conv., 7.11.18.

ZINC sulphide ores are roasted at a high temperature until the sulphur present as sulphide is reduced to 6–10%, the air supply is then increased, and the temperature allowed to fall to 650°–750° C., whereby a large proportion of the remaining sulphur is converted into sulphate. At the end of the first operation the charge may be mixed with a proportion of ore which has already been through the sulphating process. Alternatively the first operation may be prolonged until the ore is roasted "sweet," when it is mixed with sufficient unroasted ore to bring the amount of sulphur present as sulphide up to 6–10% and subjected to the sulphating roast; or, preferably, the unroasted ore is added in several lots during the second roasting operation so that the proportion of sulphide sulphur in the charge never exceeds 2%.—A. R. P.

Zinc solutions; Treatment of — F. Petersson, and The Metals Extraction Corp., Ltd. E.P. 152,752, 17.7.19.

DISSOLVED silica is precipitated from zinc solutions by treating the neutralised or substantially neutralised solution with a slight excess of an alkali or alkaline-earth carbonate at 90°–100° C.—J. W. D.

Furnace; Smelting — heated by oil or gas. K. Schmidt. E.P. 152,697, 25.3.18.

IN a furnace having an outlet shaft in the middle of the smelting chamber, the top or cover is made to slope downwards or the bottom to slope upwards so that the distance between the bottom and top of the chamber is smaller near the outlet shaft than at the outer walls. The inlet pipes or passages for the oil or gas are arranged in oblique or tangential relation to the outer wall so as to produce a whirling effect on the gases.—J. W. D.

Amalgam presses. S. H. Cocks. E.P. 152,815, 9.8.19.

A FILTER-PRESS to separate excess of mercury from amalgam consists of a chamber provided with a removable screwed airtight cap, and a perforated base

secured to the chamber by means of a removable clamp, and having a valve-controlled inlet pipe for introducing fluid under pressure to the interior of the chamber. The perforated base is covered by a filtering medium, such as canvas, through which the mercury is forced by the fluid under pressure introduced into the chamber.—J. W. D.

Copper; Deposition of — on non-conducting surfaces. J. D. Smith. E.P. 152,835, 5.9.19.

A STRONG solution of copper sulphate is added to a strong solution of hydrazine sulphate till a pale-blue powder is precipitated. This powder is then suspended in water and treated successively with a solution of ammonia, a solution of caustic soda, and a small quantity of hydrazine sulphate, yielding a clear liquid which deposits a coherent coating of copper when heated in contact with surfaces of non-conducting materials such as glass, porcelain, etc.—J. W. D.

Metallic reflectors; Manufacture of — S. O. Cowper-Coles. E.P. 152,860, 15.10.19.

IRON is deposited electrolytically upon a glass matrix or cathode which has been coated with silver, the deposited iron with the silver coating is removed from the glass cathode, the silver removed from the iron deposit, the deposit sherardised, and the sherardised face polished to form the reflecting surface.—J. W. D.

Aluminium; Process for tinning — A. L. Page, Assr. to S. Rubin. U.S.P. 1,339,710, 11.5.20. Appl., 30.9.19.

THE aluminium to be coated is rubbed until thoroughly clean whilst immersed in molten stearic acid, after which it is removed, heated in a non-carbonising flame, and then immersed in a bath of tin (80%), zinc (10%), aluminium (8%), and "phosphate-tin" (2%).

Magnesium powder; Apparatus for the manufacture of — D. S. Nicol, Assr. to American Magnesium Corp. U.S.P. 1,356,780, 26.10.20. Appl., 23.7.17.

THE apparatus consists of a crucible with an outlet at the bottom through which the metal is discharged into a disintegrating chamber where it meets a jet of fluid. The chamber is provided with a removable cup at the lower end in which the powdered metal collects.—A. R. P.

Mineral flotation; Process for — J. P. Ruth, jun. U.S.P. 1,356,832, 26.10.20. Appl., 21.2.20.

NAPHTHALENE is used as frothing agent.—A. R. P.

[Nickel-chromium steel] shells; Manufacture of — R. A. Hadfield. U.S.P. 1,357,705, 2.11.20. Appl., 10.8.17. Renewed 17.9.20.

SEE E.P. 128,961 of 1916; J., 1919, 640A.

Oil-flotation process and apparatus. D. F. Haley, Assr. to Metallurgical Co. of America. U.S.P. 1,357,556, 2.11.20. Appl., 4.6.15.

SEE E.P. 100,637 of 1916; J., 1916, 1161.

Metallic packings; Process for subdividing metal for use in plastic — W. Lewicki, Assr. to The Chemical Foundation, Inc. U.S.P. 1,355,984, 19.10.20. Appl., 10.9.12. Renewed 4.3.20.

SEE E.P. 17,618 of 1912; J., 1913, 146.

Alloy. K. Ihe. U.S.P. 1,356,367, 19.10.20. Appl., 16.8.18.

SEE E.P. 131,492 of 1918; J., 1919, 826A.

Furnace for ore-roasting. E. Bracq. U.S.P. 1,356,849, 26.10.20. Appl., 8.5.14.

SEE Addn. of 13.5.13 to F.P. 431,491; J., 1914, 88.

Metals; Process of recovering — from natural waters. O. Nagel, Assr. to The Chemical Foundation, Inc. U.S.P. 1,358,096, 9.11.20. Appl., 11.11.16.

SEE E.P. 103,310 of 1917; J., 1917, 653.

Water concentration of ores or the like; Apparatus for — or for similar purposes. W. M. Martin. E.P. 153,481, 16.12.19.

XI.—ELECTRO-CHEMISTRY.

Nitric acid manufacture. Kodera and others. See VII.

PATENTS.

Dry cells; Electric —. Burgess Battery Co., Assces. of A. W. Schorger. E.P. 135,502, 19.11.19. Conv., 19.11.18.

A DRY cell is constituted of an outer zinc electrode and an inner fragile moulded cathode encased in a bibulous envelope consisting essentially of plaster, to which preferably starch and borax are added to retard setting. The gelatinisable electrolyte employed is preferably composed of a solution containing about 25.5% of cereals such as starch, meal, or maize, and 36.7% of zinc and ammonium chloride (see E.P. 128,332; J., 1919, 686 A).—J. S. G. T.

Galvanic batteries. G., L., and G. J. A. Fuller. E.P. 152,818, 13.8.19.

A DRY galvanic battery of the type described in E.P. 102,294 (J., 1917, 38) is constituted of an inner carbon electrode and an outer zinc electrode, separated by a dry electrolyte composed of an excitant such as ammonium chloride, of a less hygroscopic nature than zinc chloride, together with gum tragacanth, tapioca, or similar absorbent material, the constituents being dried before mixing. If desired, a little zinc chloride may be added to the depolariser employed, the mixture being subsequently dried. The cell is rendered active when required by addition of water.—J. S. G. T.

Cell; Electric —. C. Féry. U.S.P. 1,356,977, 26.10.20. Appl., 19.2.18.

A FLAT horizontal zinc electrode rests on the bottom of a vessel and is covered by a solution saturated with zinc chloride. The exciting solution of ammonium chloride is of lower density than the zinc chloride solution and floats upon the latter. A vertical tubular carbon electrode rests upon the zinc electrode with a strip of porous insulating material in between, the major portion of the carbon electrode being outside of the dense liquid layer at the bottom of the vessel.—J. S. G. T.

Battery; Electric —. H. F. French, Assr. to National Carbon Co., Inc. U.S.P. 1,357,160, 26.10.20. Appl., 17.2.16.

THE tendency of the electrolyte to become progressively more basic during the life of the battery is reduced by the addition of an organic substance from which an acid is formed under the action of nascent hydrogen.—J. S. G. T.

Electrolytic apparatus. E. A. Ashcroft. E.P. 152,879, 9.7.19.

AN electrolytic apparatus, primarily designed for the electrolysis of magnesium chloride in accordance with E.P. 152,402 (J., 1920, 788 A) consists of a pair or several pairs of cells connected in series and provided with pockets communicating with the bottom of each cell. A weir extends across each cell at the height to which the alloy (used for collecting the separated magnesium) has to rise in the cell. Anodes of graphite or other material are suspended from the covers of the primary cells of

the pairs, while the iron or steel cathodes in the secondary cells are carried in sets attached to supplementary covers which are applied to openings in the main cover, the whole series of covers being included in the electric circuit. The supplementary covers are connected with the main cover by a flexible conductor.—J. S. G. T.

Electrolytic cell with multiple diaphragms. A. Barth. G.P. 325,313, 28.1.19.

LATTICE frames of which the horizontal septa are at various heights are introduced into the electrolyte between the diaphragms.—J. S. G. T.

Electrodes in electric furnaces; Suspension of —. Det Norske Aktieselskab for Elektrokem. Ind. Norsk Industri-Hypotekbank. E.P. 138,354, 28.1.20. Conv., 28.1.19.

AN electrode without a metallic core or casing, which is baked in the electric furnace in which it is used, is composed of a lower hard part and a comparatively soft upper part. It is suspended within the furnace by attaching an external or internal holder to the lower hard part of the electrode, so that the comparatively soft portion is in compression. In this manner, difficulties encountered in the use of cored electrodes such as those described in E.P. 116,853 (J., 1918, 429 A) are obviated. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Acts, 1907 and 1919, to E.P. 116,853; *loc. cit.*)

—J. S. G. T.

Graphitising carbon electrodes. E. Szarvasy. G.P. 319,087, 24.7.18.

THE electrodes are heated to a high temperature in an indifferent atmosphere containing methane or similar gas affording a supply of carbon, whereby a firm coating of graphite is produced.

—J. S. G. T.

Electrodes composed of lead peroxide or manganese dioxide, or a mixture thereof; Manufacture of —. H. Bardt. G.P. 325,154, 23.9.19.

THE respective oxides or a mixture thereof are deposited electrolytically from strong solutions of lead or/and manganese perchlorate, upon an anode of conducting material unattacked by perchloric acid. A thin, coherent, uniform deposit having been formed, the current strength is very much increased, whereby a honeycombed structure is produced in the deposit.—J. S. G. T.

[Electric battery] plate; Process of producing a double-copper-oxide —. Process for making alkaline gelatinous electrolytes. Double-copper-oxide battery element. Electrolyte for primary batteries. G. S. Engle, Assr. to American Elementary Electric Co. U.S.P. 1,356,197 and 1,356,434—6, 19.10.20. Appl., 8.10.12, 8.10.12, 6.4.15, and 28.5.15.

SEE E.P. 2395—6 of 1913; J., 1913, 758.

Condensing vapour. U.S.P. 1,356,196. See I.

Electrical precipitation. U.S.P. 1,356,462 and 1,357,201—2. See I.

Carbonisation of cellulose etc. G.P. 323,595. See IIb.

XII.—FATS; OILS; WAXES.

Fats and oils; Rapid determination of water in —. H. Oertel. Chem.-Zeit., 1920, 44, 854.

THE rise in temperature occurring when 10 c.c. of oil containing water is mixed with a definite quantity of a mixture of anhydrous magnesium sulphate, 2, and kieselgub, 1 part by weight, is a measure of the amount of water present. The

percentage of water is found by reference to tables compiled from results obtained with oils containing known quantities of water. Each 1% of water causes the temperature to rise approximately 1.3° C. Fats should be dissolved previously in an equal weight of an anhydrous solvent.—W. P. S.

Fish oils; Separation of the highly unsaturated fatty acids in —. M. Tsujimoto, Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Tokyo), 1920, 23, 1007—1010.

The method depends on the ready solubility of the lithium salts of the highly unsaturated acids in acetone containing a little water (95% acetone by vol.) and the insolubility or slight solubility of the saturated and less unsaturated acids in the same solvent. The lithium salts may be prepared by neutralising an acetone solution of the acids with lithium hydroxide, using phenolphthalein as indicator, or, for larger quantities, by treating an alcoholic solution of potassium salts of the acids with lithium acetate. The method may be used for approximate quantitative determinations. The amounts of highly unsaturated fatty acids in fish oils determined by the method are remarkably higher than those calculated from the yields of the fatty acid polybromides. On applying the method to vegetable and terrestrial animal oils, comparatively small yields of acetone-soluble salts were obtained. The formula, $C_{22}H_{32}O_2$, is proposed instead of $C_{18}H_{28}O_2$ for clupanodonic acid isolated from Japanese sardine oil. This oil probably contains also another highly unsaturated fatty acid of the composition, $C_{20}H_{30}O_2$.—K. K.

Acid earth [fullers' earth]; Property of — and its application in hydrogenation of oils. [Characters of dike whale oil.] S. Feno, Kōgyō-Kwagaku Zasshi (J. Chem. Ind., Tokyo), 1920, 23, 1028—1033.

METALLIC soaps and other impurities in oils, which act as catalyst poisons during hydrogenation, can be removed by treating the oil with 10% of acid earth (fullers' earth) at 120° C. for 20 mins. If hardened oils are treated with the acid earth, their content of nickel or other mineral matter is markedly reduced.

Dike whale (*Bolocnptera borealis*, Lesson) oil had the following characters:—sp. gr., 0.9224 at 15°/4° C.; acid value, 0.24; saponif. value, 185.2; iodine value, 144.2; acetyl value, 3.2; Hehner value, 96.0; n_D^{20} =1.4751; butyrometer reading at 20° C., 75°, and unsaponifiable matter, 0.7%. The hardened oil had m.p. 58° C.; acid value, 0.4, and iodine value, 3.0.—K. K.

Sugar-cane wax; Constituents of —. J. E. Q. Bosz, Arch. Suikerind, Nederl.-Indië, 1920, 28, 974—977.

A SAMPLE of cane wax extracted from the mud resulting from the defecation of juice in a Java factory was found to have m.p. 60°—62° C., acid value 47.3, and saponif. value 177. Cholesterol was absent. The fatty acids separated by saponifying, adding hydrochloric acid, and shaking out with benzene, had m.p. 51° C., and appeared to be an eutectic mixture of palmitic and stearic acids. In the liquid obtained on steam-distilling the product of saponification, myricyl alcohol, caproic acid, and formic acid were identified. Acetic, benzoic, and cinnamic acids could not be detected.—J. P. O.

Fluidity of oils. See IIA.

PATENTS.

Nickel carbonyl in hydrogenation processes; Method of applying —. R. Lessing, E. P. 152,740, 15.7.19.

NICKEL carbonyl is separated from admixed gases,

e.g., carbon monoxide, by passage over a cooled absorbent such as "activated" charcoal or an inert porous material, such as coke, impregnated with ground-nut (arachis) oil or the like. As soon as absorption is complete a supply of the hydrogenating gas (hydrogen) is passed through the apparatus, which is heated to about 100° C., whereby the absorbed nickel carbonyl is liberated and carried away by the hydrogenating gas.

—L. A. C.

Oil press; Automatic continuous hydraulic —. C. D. Helm and W. A. Diboll, E.P. 153,090, 29.7.19.

THE oil-bearing seed is brought under the press in a series of containers carried by a conveyor. Automatic arrangements are provided to stop the conveyor at the correct point, to lower the hydraulic cylinder (the ram being fixed) and effect the pressing, to raise the cylinder, and re-start the conveyor.

—B. M. V.

Beeswax; Apparatus for extracting and purifying —. L. H. Willis, E.P. 152,811, 12.9.19.

THE crude wax combs are placed in a perforated container, which is fixed in the bottom of an outer vessel, and sufficient water is added to cover the container. On heating the water the wax melts and rises through the perforations to the surface of the water.—L. A. C.

Fats; Recovery of — from waste liquids [by settling and skimming]. J. Blunn, E.P. 153,372, 31.7.19.

Revivifying filtering agents. U.S.P. 1,356,631. See IIA.

Fat from bones. G.P. 325,755. See XIXA.

Solvents. E.P. 152,550. See XX.

Emulsions. G.P. 324,012. See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Tungsten pigments. H. A. Gardner and A. Reilly, Paint Manufacturers' Assoc., U.S.A. Circ. 109, Oct., 1920, 9 pp.

A BY-PRODUCT from the manufacture of tungsten lamp filaments, containing 99.9% WO_3 , was a light canary-yellow powder of sp. gr. 5.75. It had a negligible tinting power in comparison with chrome yellow. When used as a pigment with linseed oil the oxide changed to a grayish-yellow colour when exposed to the direct rays of the sun for 15 mins. Another product from the same source contained 115% WO_3 together with large amounts of iron and manganese. It was of dark brown colour, very finely divided, and had sp. gr. 4.75. It possessed good staining and drying powers, and when heated with linseed oil it showed a good siccativ action, probably due to its high manganese content.—A. de W.

Mineral spirits as thinners; Solvent properties and colour effects of —. H. A. Gardner and P. C. Holdt, Paint Manufacturers' Assoc., U.S.A. Circ. 107, Oct., 1920, 9 pp.

SHORT-OIL varnishes free from thinner when mixed with different samples of mineral spirit, and allowed to stand for one month in tall glass-covered receptacles, showed varying degrees of darkening of colour. The darkening is attributed to combination between the lead drier of the varnish and sulphur compounds in the thinner. Elastic varnishes free from thinner mixed more readily with turpentine than with any of the mineral oil

thinners, separation of dark material or darkening of colour occurring with the latter after one month's standing. When highly heat-treated oil to which lime had been added during treatment was thinned with an equal weight of mineral thinner, and allowed to stand for one month, a flocculent layer insoluble in excess of thinner separated. With mixtures containing 10–20% of thinner, however, no separation occurred with the mineral spirit thinner, but turpentine caused a separation when added in quite small proportion. Whilst turpentine mixes quite readily with blown oils in all proportions, most samples of mineral spirits cause a cloudiness to develop on standing.—A. de W.

PATENTS.

Ultramarine; Process for the manufacture of —. J. B. Guimet and A. Guillochin. E.P. 152,916, 14.2.20.

In the manufacture of ultramarine an alkali bisulphite or sulphite, such as the sodium sulphite obtained as a by-product in the manufacture of phenol, is used instead of sodium sulphate and/or sodium carbonate. A mixture of 150 pts. of kaolin, 115 pts. of sodium sulphite, 21 pts. of resin, and 200 pts. of sulphur is heated for 6 to 12 hrs. at 850° C., and is cooled during 5 to 6 days or 20 days to produce respectively a green or blue product.

—L. A. C.

Aldehyde condensation product capable of technical utilisation; Manufacture of —. H. John. U.S.P. 1,355,831, 19.10.20. Appl., 25.10.19.

An aliphatic aldehyde is caused to react upon substances containing "carbonic acid amides" in a highly heated state without the use of a condensing medium.—A. de W.

Coating composition; Liquid —. A. N. McKay and W. L. Willis. U.S.P. 1,356,380, 19.10.20. Appl., 21.11.17.

Tar oil and "holder oil" are mixed in a cold still, solutions of granulated caustic soda and potassium permanganate together with milk of lime added, and the mixture heated approximately to 200° F. (93° C.). The residue is subsequently maintained at about 150° F. (65° C.) until all water is evaporated.—A. de W.

Itiopone; Manufacture of —. J. L. Mitchell. U.S.P. 1,356,387, 19.10.20. Appl., 20.8.18.

A product consisting of barium sulphate and zinc sulphide is washed, dried, and pulverised, and then calcined, with agitation, in a muffle furnace, quenched in cold water, and dried.—A. de W.

Stain for wood; Preparation of a substance suitable for producing a durable —. K. S. Fuchs. G.P. 325,731, 5.6.19. Addn. to 320,011 (J., 1920, 665 A).

The waste liquor from the alkaline digestion of vegetable matter, more especially straw, or a solution in alkali of the constituents of the liquor which are precipitated by acid, is treated with salts of heavy metals, such as ferric chloride or copper sulphate, whereby products are obtained which are converted into complex substances soluble in water by treatment with ammonia or amino-bases. The solutions of the products give clear brown tints when painted on wood, and the colour becomes quite insoluble after drying in sunlight or at 50° C.

—J. S. G. T.

Turkish bird-line; Process for producing a working form of —. C. A. Cleghorn, Assr. to Gayner Pneumatic Co. U.S.P. 1,356,267, 19.10.20. Appl., 26.12.18.

SEE E.P. 130,379 of 1918; J., 1919, 730 A.

Solvents. E.P. 152,550. See XX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Latex; Viscosity of — and its bearing on the rate of cure. H. P. Stevens. Bull. Rubber Growers' Assoc., 1920, 2, 214–215.

VARIATION in the fluidity of latex is not due to variation in the rubber content, but is caused by incipient coagulation. Viscous latex frequently yields a rapidly vulcanising rubber, possibly on account of bacterial action commencing at an earlier stage than usual.—D. F. T.

Alum as a [rubber] coagulant. H. P. Stevens. Bull. Rubber Growers' Assoc., 1920, 2, 142–144.

COAGULATION with the minimal amount of alum, to effect complete separation of the rubber from latex containing a very small proportion of sodium sulphite, gives a product of the same rate of vulcanisation as one obtained under similar conditions with acetic acid as coagulant. In the absence of sodium sulphite, the use of alum causes a retardation of the rate of vulcanisation relative to the effect with acetic acid, but coagulation with sulphuric acid produces a still greater retardation.

—D. F. T.

Latex coagulated with a saccharated solution of lime. H. P. Stevens. Bull. Rubber Growers' Assoc., 1920, 2, 144.

The yield of crêpe rubber obtained on coagulating latex with a solution of lime and sugar (saccharated solution of lime, B.P.) was 2% less than the normal; the rubber gave an acetone extract of 3.4% and was of good quality, with a somewhat higher breaking strength and greater rate of vulcanisation than the average.—D. F. T.

[Rubber:] Maturation [of —]. H. P. Stevens. Bull. Rubber Growers' Assoc., 1920, 2, 212–214.

"MATURED" rubber obtained by spontaneous coagulation of latex has approximately the same rate of vulcanisation as material produced under similar conditions, but with the addition of a little acetic acid; vulcanisation proceeds more rapidly, however, the thinner the sheet during the maturing process. These and earlier results indicate that both aerobic and anaerobic changes are involved in maturation, but that the alteration is mainly of the latter type.—D. F. T.

[Rubber:] Effect of acids in retarding the rate of cure [of —]. H. P. Stevens. Bull. Rubber Growers' Assoc., 1920, 2, 343–347.

If freshly coagulated rubber is soaked in a solution of an acid its rate of vulcanisation is decreased; sulphuric acid exerts a much more marked effect than acetic acid. The effect arises merely from the retention of part of the acid by the rubber because, by soaking in water for several months, it is possible to remove part of the acid and to increase the rate of vulcanisation of the rubber.—D. F. T.

Rubber; Variation in fine hard Parà and plantation —. H. P. Stevens. Bull. Rubber Growers' Assoc., 1920, 2, 347–350.

AN examination of the rate of vulcanisation of rubber from five leaves of wild Parà rubber revealed a variation comparable with that observed with properly prepared smoked sheet plantation rubber (cf. Eaton, Grantham, and Day, J., 1919, 111 A).

—D. F. T.

Rubber; Ageing of vulcanised plantation —. H. P. Stevens. Bull. Rubber Growers' Assoc., 1920, 2, 270–276.

EARLIER observations (J., 1918, 305 T, 340 T) have been extended by experiments with three mixtures of rubber 60, sulphur 3, and zinc oxide 37 parts, the rubber consisting of plantation pale crêpe,

plantation smoked sheet, and fine hard Pará rubber respectively. When kept in a vulcanised condition for 3½ years the coefficient of vulcanisation underwent a distinct increase, the extent of which (an average of 0.27 for an initial coefficient of 2–3 and 0.40 for 3–4) was rather greater than that observed with the earlier mixture of rubber 90% and sulphur 10% at a comparable initial degree of vulcanisation. Physical tests after an ageing period of 120 weeks showed that, if the initial coefficient did not exceed 3.5, the breaking strain first increased to a maximum and then began to decrease, the highest final values being obtained for the samples with the lowest coefficients. The initial breaking strength and elongation of each of the mixtures containing zinc oxide, unlike those of a simple rubber-sulphur mixture, were very similar whatever the degree of vulcanisation, the smoked sheet rubber giving the highest breaking strain and the lowest extensibility. The curves representing the alteration of tensile strength and elongation with ageing tend towards a horizontal course after the breaking strength has fallen below 600 g. per sq. mm. For relative stability in vulcanised mixtures of this type it is found that the coefficient of vulcanisation should not exceed a value of 2–2.5, this limit being lower than for a mixture of 90% rubber with 10% of sulphur.—D. F. T.

Rubber analysis. A. R. Pearson, Analyst, 1920, 45, 105–109.

Determination of total sulphur.—20 c.c. of nitric acid (sp. gr. 1.5) is placed in a flask and 0.5 g. of the rubber is added in small pieces at a time; the mixture is heated gradually and kept on a water-bath for 30 mins. Small successive quantities of permanganate are added until some manganese oxide remains unreduced after 1 hr.'s heating, 20 c.c. of concentrated hydrochloric acid is added, the mixture again heated, evaporated to dryness, the residue treated with hydrochloric acid, again evaporated, the residue treated with hot dilute hydrochloric acid, the solution filtered, and the sulphuric acid in the filtrate determined gravimetrically. *Determination of carbonates in rubber mixings.*—1 g. of the finely divided sample is heated with 25 c.c. of glacial acetic acid in a flask provided with a short reflux condenser, the latter being connected with a U-tube containing solid lead acetate, a U-tube containing in one limb sodium acetate and in the other calcium chloride, and two weighed tubes containing soda-lime and calcium chloride. The contents of the first two U-tubes must be saturated with carbon dioxide before use. A current of air is aspirated through the apparatus during the whole operation.

—W. P. S.

PATENTS.

Vulcanised caoutchouc and art of producing the same. W. Scott, Assr. to Goodyear Tire and Rubber Co. U.S.P. 1,356,195, 19.10.20. Appl. 10.11.19.

AN ortho-alkyl substitution derivative of a thiarylurea is employed as a vulcanisation catalyst.

—D. F. T.

Bark-shavings from rubber trees; Process for the utilisation of —. A. H. Claessen. U.S.P. 1,357,845, 2.11.20. Appl. 4.3.20.

SEE E.P. 112,946 of 1919; J., 1920, 495 A.

XV.—LEATHER; BONE; HORN; GLUE.

PATENTS.

Chamois leather substitute; Production of a —. O. Röhm. G.P. 325,881, 21.2.19.

A CHAMOIS leather substitute, which closely resembles the genuine material as regards softness

and tenacity, is produced by treating skins with a mixture of soap, fat, and an aldehyde, specially formaldehyde.—W. J. W.

Glue; Production of bone or hide — which remains liquid in the cold. Luftfahrzeugbau Schütte-Lanz. G.P. 325,216, 21.11.17.

Formic acid or a formate is added to the glue dissolved in warm water. Concentrated glue solutions can thus be maintained fluid even below 0° C.

—J. H. L.

XVI.—SOILS; FERTILISERS.

Superphosphates; Retrogradation of —. A. Aita. Giorn. Chim. Ind. Appl., 1920, 2, 227–233.

THE transformation of the monocalcium phosphate in superphosphates into dicalcium phosphate, and of the latter into tricalcium phosphate is commonly attributed to the presence of iron and aluminium in the phosphorites. Experiments described in detail have shown that the presence of free phosphoric acid tends to neutralise the action of the iron and aluminium compounds, and that eventually equilibrium is established. Hence, when phosphorites containing relatively large amounts of iron and aluminium are used in the manufacture, sufficient sulphuric acid should be used to leave a certain quantity of free phosphoric acid in the product.—C. A. M.

Nitrogen; Gas-volumetric determination of ammoniacal, nitric, and organic — in mixed fertilisers. P. Baddi. Giorn. Chim. Ind. Appl., 1920, 2, 376–378.

TEN g. of the fertiliser is extracted with hot water, the extract filtered, and the filtrate and washings made up to 200 c.c. Ammoniacal nitrogen is determined in 10 c.c. by the hypobromite method. Nitric nitrogen is determined by reducing 50 c.c. of the solution with Devarda's alloy in presence of sodium hydroxide, and the ammonia estimated as before. Organic nitrogen is determined by Kjeldahl's method in the solution, and in the residue from the extraction, the ammonia being estimated by the hypobromite method. The results are somewhat lower than those obtained by distilling the ammonia, but for most purposes are sufficiently accurate.—C. A. M.

Calcium cyanamide. Kameyama. See VII.

PATENTS.

Nitrogen compounds; Recovery of soluble — from solutions [sewage]. E. H. Richards and H. B. Hutchinson. E.P. 152,387, 14.6.19.

SEWAGE, or other dilute solution containing ammonium salts, preferably containing less than 0.1% of soluble nitrogen, is maintained under aerobic conditions in contact with activated carbonaceous material, e.g., activated straw, until the latter has become fully loaded with insoluble nitrogen produced from the soluble nitrogen in the liquid, or until the major part of the soluble nitrogen in the liquid, e.g., 90% or more, has been transformed into insoluble compounds by the bacteria. The carbonaceous material may be activated by prolonged contact, e.g., for 90 hrs. or longer, with a dilute solution of soluble nitrogenous compounds, and after use it is available as manure.—J. H. L.

Fertilisers; Production of mixed —. E. Reinau. G.P. 324,379, 13.8.18.

A SOLUTION of an alkali sulphate in heated aqueous nitric acid is caused to crystallise with agitation, at a temperature at which the mother liquor will be saturated or supersaturated not only with alkali nitrate, but also with polysulphate. The crystals

which separate are neutralised with ammonia. In this manner a mixture of potassium nitrate and ammonium sulphate with the same nitrogen content as 96–99% potassium nitrate may be obtained.

—J. S. G. T.

Fertiliser. J. R. Partington and L. H. Parker. U.S.P. 1,348,578, 3.8.20. Appl., 12.12.18.

SEE E.P. 151,024 of 1918; J., 1920, 759 A.

VII.—SUGARS; STARCHES; GUMS.

Oxalic acid; Presence of — in the sugar cane. J. E. Q. Bosz. Arch. Suikerind. Nederl.-Indië, 1920, 28, 969–974.

OPERATING upon about 15 kg. of the 100 P O J variety of sugar cane, and applying the procedure recommended by Berthelot and André (Comptes rend., 1885, 101, 354), the author has found 0.014% of oxalic acid ($C_2H_2O_4 \cdot 2H_2O$) to be present in water-soluble form.—J. P. O.

Beet juice; Extraction of — by the "rapid" method. S. Thielér. Centr. Zuckerind., 1920, 28, 468–470, 492–495, 522–524.

EXPERIMENTS have been made in several factories in Germany with the so-called "rapid" method, in which after preheating by Bosse's scalding process (U.S.P. 1,005,931; J., 1911, 1326) the slices are extracted by means of Paschen's inclined trough apparatus (U.S.P. 1,134,152; J., 1915, 567), the juice and slices being forced from one end to the other in counter-current through a series of compartments. In the Bük factory, in which a trough about 22 m. long, divided into 20 compartments, was used, the sugar content of the treated slices was found to be 0.4% (calculated on the weight of sliced roots); but the density of the juice finally drawn off, which amounted to 140% on the roots, was about 3° Brix less than in the case of the liquid extracted by the diffusion method. The purity of the juice was about the same as in the diffusion process; but the juice obtained by the new process was particularly low in coagulable substances.—J. P. O.

Sugar juice; Clarification [sulphitation and carbonation] of — regarded from the standpoint of colloid chemistry. W. H. T. Harloff. Arch. Suikerind. Nederl.-Indië, 1920, 28, 802–824. Int. Sugar J., 1920, 22, 705–706.

IN order to obtain the maximum adsorption of colloidal impurities from the raw juice by the precipitate produced during the sulphitation method of clarification, the quantity of the precipitate should be as great as economical considerations will permit; precipitation should be effected at as high a temperature as possible without incurring inversion or decomposition, namely about 85° C., in order to obtain a precipitate of good crystalline structure; and precipitation should be rapid. During settling the reaction should be slightly acid, this having been found to be the optimum reaction for the adsorption by the precipitate of gums and pectins, and colouring matters. In the carbonation process, by raising the initial temperature to 55° C., and thus obtaining a more "porous" precipitate of calcium carbonate, 40% less lime can be used. The following conditions for operating single carbonation are recommended: preliminary temperature of the raw juice, 55° C.; alkalinity after carbonating, 200 mg. CaO per l. (phenolphthalein indicator); final temperature of heating, 85° C.

—J. P. O.

Sulphitation method of cane juice clarification; Prevention of incrustation in the heaters used in the —. F. van Vollenhoven. Arch. Suikerind. Nederl.-Indië, 1920, 28, 918–922.

IN Java tubular heaters are used for raising the temperature of the raw juice to 65°–70° C. previous to sulphiting; while another set is employed for heating the juice to boiling point after it has been both sulphited and limed. In order to diminish the formation of scale in the second set of heaters, it is proposed that the two sets be connected together and used alternately for the untreated and the treated juice. This suggestion is based on the fact that sulphite scale is soluble to the extent of about 50% in a dilute solution of sugar.

—J. P. O.

Sulphitation method of cane juice clarification; Prevention of incrustation in the heaters used in the —. G. Benthem. Arch. Suikerind. Nederl.-Indië, 1920, 28, 1040–1042.

SATISFACTORY results may be obtained by the procedure recommended by Vollenhoven (*cf. supra*), but the author prefers to change over from one set of heaters to the other after an interval of, say, 6 hrs., instead of 3–5 days, as originally suggested. In this way the surface of the tubes is kept free from any considerable amount of scale, and a better transmission of heat is secured. A sample of the scale in question gave the following figures: Water (at 135° C.), 9.87; organic matter, 16.20; insoluble in hydrochloric acid, 5.75; P_2O_5 , 0.88; $Al_2O_3 + Fe_2O_3$, 0.54; MgO, 0.37; CaO, 34.51; SO_2 , 2.90; SO_3 , 28.84; and CO_2 and undetermined matter, 0.14%.

—J. P. O.

Sugars; Determination of alkalinity of raw —. G. Bruhns. Zentr. Zuckerind., 1920, 28, 935–936. Chem. Zentr., 1920, 91, IV., 535–536.

MANY raw (beet) sugars, if covered with water containing red phenolphthalein, give at first a yellow solution which becomes red after some time owing to the gradual solution of calcium carbonate contained in the sugar. This behaviour is sometimes shown by juices, syrups, and white sugar. The red colour may eventually vanish owing to absorption of carbon dioxide from the air. In testing the reaction of sugar which is to be stored, such delayed reddening should be neglected, i.e., the sugar should not be returned as alkaline, for solid calcium carbonate affords little protection against acidification and deterioration during storage. If, however, the sugar is to be inverted for the production of syrup or artificial honey, any calcium carbonate present should be reckoned as alkalinity in view of the very small quantity of acid used for inversion. In such cases the alkalinity may be determined by dissolving 20 g. of the sugar in 100 c.c. of water and 5 c.c. of N/10-hydrochloric acid, boiling for 5 mins., cooling, and titrating back with sodium hydroxide.—J. H. L.

Sugars; Volumetric determination of mixtures of —. T. von Fellenberg. Mitt. Lebensmittel. u. Hyg., 1920, 11, 129–153. Chem. Zentr., 1920, 91, IV., 536–537.

THE author uses a modified Fehling's solution containing sodium carbonate as well as sodium hydroxide; 20 c.c. of the solution is boiled with an equal volume of sugar solution, the precipitated cuprous oxide dissolved by addition of concentrated sodium chloride solution containing hydrochloric acid, the solution treated with a slight excess of sodium hydroxide, and the reduced copper determined iodometrically. Tables are given for dextrose, invert sugar, lactose, and maltose in terms of N/10-iodine solution. For the analysis of mixtures of sugars a scheme is given involving one or more of the following processes, viz., "weak inversion,"

in which 50 c.c. of sugar solution is heated with 1 c.c. of *N*/1-hydrochloric acid for ½ hr. in boiling water; "strong inversion," in which 50 c.c. of sugar solution is heated with 25 c.c. of 3*N*-hydrochloric acid for ½ hr. in boiling water; and destruction of reducing sugars by means of sodium hydroxide. For details the original paper should be consulted.—J. H. L.

Sugars; New methods for the volumetric determination of reducing —. A. Ionescu and V. Vărgolici. Bul. Soc. Chim. România, 1920, 2, 38—45. Chem. Zentr., 1920, 91, IV., 536.

REDUCING sugars may be conveniently determined with a solution containing 46 g. of potassium ferricyanide and 46 g. of potassium hydroxide per l. 10 c.c. of this solution, which is stable in the dark, is diluted with 20 c.c. of water, and heated to boiling, and the sugar solution is added from a burette until decolorisation occurs. With coloured liquids 10 drops of 1% picric acid solution are added as indicator, the formation of red picramic acid marking the end-point. 10 c.c. of the ferricyanide solution requires 10 c.c. of 0.5% dextrose solution. For the determination of lactose in milk, 10 c.c. of the latter is mixed with 10 c.c. of a solution containing 20 g. of glacial acetic acid and 10 g. of picric acid per l., and filtered; 10 c.c. of the filtrate is neutralised with sodium hydroxide, diluted to 40 c.c. and employed for the titration as above. 10 c.c. of ferricyanide solution corresponds with 0.0676 g. of lactose.—J. H. L.

Reducing sugars; Determination of —. C. A. Browne. J. Assoc. Off. Agric. Chem., 1919, 3, 261—263.

CRUCIOUS oxide obtained in the gravimetric determination of reducing sugars can be readily reduced to the metallic state by heating the crucible containing it to dull redness and plunging it into the vapour of methyl alcohol (*cf.* Wedderburn, J., 1915, 854). The alcohol used should be changed frequently, since oxidation products present in it may interfere with complete reduction; and there is danger of decomposition of alcohol with deposition of carbon if the crucible is heated to too high a temperature.—J. P. O.

Dextrose; Determination of small quantities of —. Perrier. J. Pharm. Chim., 1920, 22, 337—344.

THE iodometric method described by Bougault (J., 1917, 899) is recommended for the determination of very small quantities of dextrose. The dextrose solution is treated with an excess of *N*/100 iodine solution, 1 c.c. of 1.5% sodium carbonate solution is added for each 25 c.c. of the iodine solution used, and, after 2 hrs.' contact, the mixture is acidified with hydrochloric acid and the excess of iodine titrated with *N*/100 thiosulphate solution; 0.1 c.c. is deducted from the volume of iodine solution used for the actual oxidation and the remainder is calculated into dextrose. The method may be used for the determination of dextrose in body fluids after they have been suitably defecated.—W. P. S.

Copper; Determination of — (especially in sugar analysis) by means of potassium thiocyanate and potassium iodide. G. Brulins. Z. anal. Chem., 1920, 59, 337—359.

THE following procedure is given for determining the amount of unreduced copper when reducing sugars are heated with Fehling's solution. Twenty c.c. of the sugar solution (containing not more than 0.4% of reducing sugar) is mixed with 20 c.c. of Fehling's solution, heated to boiling, and the boiling continued for exactly 2 mins.; 50 c.c. of cold water is added, the mixture is cooled rapidly, 5 c.c. of potassium thiocyanate-iodide solution

(KCNS, 0.65, KI, 0.1 g. per 5 c.c.) is added, followed by 10 c.c. of 6.5*N* sulphuric acid, and the liberated iodine is titrated with thiosulphate solution (34.4 g. per l.). A control titration is made at the same time, using the same quantities of reagents but omitting the boiling operation; the difference in the amounts of thiosulphate solution used for the two titrations is a measure of the reducing sugar present. Tables are given showing the quantities of invert sugar corresponding with different quantities of thiosulphate solution.

—W. P. S.

Molasses; Pycnometer for the determination of the specific gravity of —. W. B. Newkirk, U.S. Bureau of Standards. Technol. Paper No. 161, 1920.

A *WUB* provided with a stop-cock is attached to the pycnometer, and after filling the pycnometer entirely and the bulb partly with the molasses, the apparatus is placed under reduced pressure in order to remove the dissolved gases and entangled air from the molasses; when bubbles no longer collect in the bulb, the pycnometer is disconnected, sealed off, and weighed. Results accurate to a few hundredths of a per cent. of dry substance can be obtained. The use of heat to aid the elimination of the gases causes sufficient decomposition to vitiate the readings.—J. P. O.

Lactose; Solubility of —. J. Gillis. Rec. Trav. Chim., 1920, 39, 677—678.

THE following results have been obtained for the solubility of lactose in water at temperatures above 50° C. The figures represent percentage by weight of lactose ($C_{12}H_{22}O_{11}$) in the solution:—57.1° C., 34.9; 63.9°, 39.1; 73.5°, 45.8; 79.1°, 49.6; 87.2°, 55.1; 88.2°, 56.0; 107.0°, 63.9; 121.5°, 69.4; 133.6°, 73.2; 138.8°, 75.2; 158.8°, 81.1; 178.8° C., 86.7. (*Cf.* Hudson, J., 1908, 1168; Saillard, J., 1919, 919 A)—W. G.

Potato starch manufacture; Chemistry of —. H. Tryller. Chem.-Zeit., 1920, 44, 833—834, 845—847.

ORDINARY potato starch may be considered to be the calcium salt of starch-phosphoric acid, the anhydrous substance having the formula $(C_6H_{10}O_5)_n(OH)PO_3Ca$, where *n* has a value of 260, corresponding with a P_2O_5 content of 0.165%; a part of the calcium is, however, always replaced by magnesium, potassium, iron, and manganese. In the potato itself, the starch is present as the potassium salt, but during the process of preparation the potassium is replaced by calcium derived from the water used for washing the starch. The calcium may also be replaced by heavy metals.—W. P. S.

"Norit" and blood charcoal. Krut and Van Duin. See IIa.

Sugar-cane wax. Bosz. See XII.

PATENTS.

Sugar manufacture; Horizontal evaporator, especially for —. V. Frynta. G.P. 323,341, 27.3.19. Conv., 11.1.18.

THE evaporator has a liquid chamber at each end, and these are connected by rows of inclined tubes supported at the ends by vertical tube-plates on the outer sides of which horizontal channels are fixed one above another. The liquid on the floor of the chamber covers the lower ends of the bottom row of tubes and extends along them for some distance. When the apparatus is working, this liquid is carried along the tubes by ebullition, and on emerging from their upper ends it falls into one of the horizontal channels. From this it is similarly carried up a higher row of tubes sloping in the

opposite direction. It thus passes from end to end of the tubular system, mounting from channel to channel.—J. H. L.

Oralic acid from sugar. U.S.P. 1,356,137. See XX.

XVIII.—FERMENTATION INDUSTRIES.

Malt; Mashing experiments with a defectively modified —. H. Lüers and M. Schneider. Z. ges. Brauw., 1920, 43, 313—315, 321—324, 329—331.

A VERY badly modified malt, containing 22% of hard and steely corns and 15% of ungerminated corns, was subjected to laboratory mashing tests by 15 different methods. The highest yield from grist of ordinary fineness was 75.62% of the dry substance and was obtained by a combination of cold pre-digestion, proteolytic digestion at 45° C., and mash-boiling.—J. H. L.

Yeast; Influence of temperature on various functions of —. H. Zikes. Zentr. Bakt., 1919, 11., 49, 353; 1920, 50, 385. Z. ges. Brauw., 1920, 43, 211—213, 316—319.

THE author deals with the influence of temperature on the reproduction, sporulation, fermentation, and liquefaction of yeast, on the shape of cells and the production of fat, glycogen, and pigments, on surface films and giant colonies, on the activity of yeast enzymes, and the transformation of bottom-into top-fermentation yeast. The exercise of some of these functions, e.g., reproduction, fermentation, and sporulation, is influenced, at least for a time, by the temperature to which the yeast has previously been accustomed. Production of acid, esters, and fat is more rapid at high temperatures (20°—30° C.) than at low ones (12°—15° C.), whilst the converse applies to the formation of colouring matters by pigment yeasts. Low temperatures favour the formation of elongated cells which remain attached, whilst high temperatures tend to produce round or ovoid cells which separate more readily.—J. H. L.

Acetaldehyde; Formation of — by certain pentose-fermenting bacteria. W. H. Peterson and E. B. Fred. J. Biol. Chem., 1920, 44, 29—46.

WHEN a fixative such as sodium or calcium bisulphite is present in the fermentation mixture, acetaldehyde is formed during fermentation by *Bac. acetothyllicum* (J., 1919, 734 A, 786 A), by *Lactobac. pentoaceticus* (J., 1919, 840 A; 1920, 381 A, 607 A), and by a pentose-fermenting organism isolated from sunflower silage and apparently belonging to the colon-aerogenes group. The formation of acetaldehyde is accompanied by a high proportion of volatile acids in the fermentation products, and the yield of alcohol decreases with increase in the production of aldehyde. (Cf. J.C.S., Dec.)

—J. C. D.

Emulsin enzymes; Resistance of — to the prolonged action of 70% methyl alcohol. M. Bridel. J. Pharm. Chim., 1920, 22, 323—327.

THE activity of the enzymes is decreased, but not destroyed, when the emulsin is kept for 5 years in 70% methyl alcohol; the enzymes which act on lactose and β -ethylgalactoside appear to be more resistant than is that which acts on β -glucosides.

—W. P. S.

Diastase in sweet potato. Gore. See XIXA.

Yeast as test organism for vitamin B. Souzt and McCollum. See XIXA.

Dehydration of amyl alcohol. Senderens. See XX.

PATENTS.

Glycerol; Manufacture of — from sugar. Verein. Chem. Werke A.-G. E.P. 138,330, 21.1.20. Conv., 19.5.16. Addn. to 138,099 (J., 1920, 608 A.)

A NEUTRAL salt of an alkaline-earth metal or of magnesium or aluminium is added to the fermentation liquid. This renders possible a large increase in the amount of alkaline substance or sulphite added, and consequently an increased yield of glycerol. If 1 kg. of sugar, 500 g. of anhydrous sodium sulphite, and 250 g. of magnesium sulphate are dissolved in 5 l. of water and fermented at 32° C. with 100 g. of yeast, the yield of alcohol obtained is 15% and that of glycerol 33% of the weight of sugar.—J. H. L.

Distillers' slops; Process of treating —. H. P. Basset. U.S.P. 1,357,133, 26.10.20. Appl., 3.11.19. Renewed 10.9.20.

DISTILLERS' slop is subjected to the action of the higher hydroxide of a metal which is capable of forming higher and lower hydroxides and capable also of forming an insoluble basic salt with volatile fatty acids; the hydroxide is maintained in the higher state of oxidation by suitable oxidising treatment, the liquid is acidified, and glycerol is separated from it.—J. H. L.

Medicinal preparations. G.P. 324,747. See XX.

XIXA.—FOODS.

Milk samples; Sour — and Gerber's fat method. F. E. Day. Analyst, 1920, 45, 411—412.

GERBER'S method for the determination of fat in milk yields high results when applied to sour milks, owing to the formation of amyl esters of the lower aliphatic acids. Lactic acid has no effect, acetic acid little, but butyric acid causes a considerable error. The addition of 1% of butyric acid to milk increases the observed fat content by 0.57%; in the case of a sample of milk, 5 weeks old and having a strong odour of butyric acid, the amount of fat found was 0.19% too high.—W. P. S.

Diastase; Occurrence of — in sweet potato in relation to the preparation of sweet potato syrup. H. C. Gore. J. Biol. Chem., 1920, 44, 19—20.

SWEET potatoes possess a high diastatic power, and it is possible to convert nearly all their starch into soluble carbohydrates by slowly cooking the potatoes in water.—J. C. D.

Yeast; Factors which interfere with use of — as a test organism for the antineuritic substance [vitamin B]. G. de P. Souza and E. V. McCollum. J. Biol. Chem., 1920, 44, 113—129.

THE use of yeast (Williams, J., 1919, 692 A; 1920, 608 A) is regarded as unreliable, being subject to many disturbing factors.—J. C. D.

Vitamin; Effect of cooking on water-soluble B— in carrots and navy beans. E. W. Miller. J. Biol. Chem., 1920, 44, 159—173.

ORDINARY cooking processes appear to cause no destruction of the vitamin in carrots, but some loss may occur in the case of navy beans. Much vitamin is found in the cooking water. (Cf. J.C.S., Dec.)—J. C. D.

Vitamin; Water-soluble B — in cabbage and onion. B. K. Whipple. J. Biol. Chem., 1920, 44, 175—187.

THE vitamin in these two vegetables is not destroyed by cooking (boiling), but much may be lost in the cooking water. (Cf. J.C.S., Dec.)—J. C. D.

Reducing sugars. Ionescu and Vărgolici. See XVII.

Solubility of lactose. Gillis. See XVII.

PATENTS.

Pasteurising apparatus. P. I. Buass. E.P. 140,131, 17.3.20. Conv., 17.3.19.

PASTEURISING apparatus of the regenerative type, for milk or other liquid, is provided with means for regulating the regenerative heating of the incoming milk, for preventing mixing of the fresh and pasteurised milk by leakage, and for reading the thermometer from the ground level.—W. F. F.

Buttermilk; Method of drying —. N. P. Collis, Assr. to Collis Products Co. U.S.P. 1,356,310, 19.10.20. Appl., 3.10.19.

BUTTERMILK in which the insoluble curd is maintained in a state of fine suspension by agitation is projected as a spray against a heated surface, on which the soluble and insoluble constituents are deposited, and the deposit is removed and completely dried and disintegrated by mechanical motion through air. (*U.S.P.* 1,317,777; J.L. 1919, 960 A.)—J. H. L.

Cream; Method of and solution for ascertainment of the quantity of fat in —. H. M. Höyberg. U.S.P. 1,357,168, 26.10.20. Appl., 26.1.20.

THE solution claimed contains 50–70 g. of sodium hydroxide and 70–130 g. of sodium potassium tartrate in 1 l. of water. To determine the fat in un-diluted cream, 5 vols. of the latter is mixed with 6.5 vols. of the above solution and 0.16 vol. of isobutyl alcohol in a butyrometer tube, and after the mixture has remained for 15–20 mins. in a water bath at 60°–70° C. the volume of fat is noted.

—J. H. L.

Bread; Type of —. C. O. Johns and A. J. Finks. U.S.P. 1,356,988, 26.10.20. Appl., 4.9.20.

CEREAL flour is mixed with a smaller proportion of soya bean flour and still smaller proportions of table salt, dicalcium phosphate, butter, water, and yeast, and the mixture is kneaded and baked.

—J. H. L.

Eggs; Production of dried yolk or white of —. K. Ebers. G.P. 325,114, 10.1.18.

YOLK or white of eggs, which has been preserved by means of antiseptics, is first freed from the latter by dialysis and then rapidly desiccated in a known manner by atomising in a current of dry or warm air. If antiseptics which are only slightly soluble in water or non-dialysable, e.g., benzoic or boric acid, are present, they may first be converted into dialysable substances, e.g., by addition of alkali.

—J. H. L.

Fodder rich in protein; Manufacture of —. Verein der Spiritus-Fabrikanten in Deutschland. G.P. (A) 325,443, 14.6.18, and (n) 325,444, 27.6.19.

UREA is added to (a) a fodder containing sugar, such as beets or molasses, or (n) carbohydrate fodders in general.—L. A. C.

Bones; Production of fat and protein substances from —. Militärkonserven-Fabr. Abt. Fette, Extrakte und Nebenprodukte Heinemann und Hanka. G.P. 325,755, 14.2.19.

AFTER a preliminary removal of fat by treatment under pressure in autoclaves the bone residues are boiled with a solution of sodium carbonate; the proteins dissolve, the fat floats on the surface, and the mineral matter is deposited.—J. H. L.

Butter making; Process of —. [*Determination of fat in milk and cream.*] H. M. Höyberg. E.P. 153,446, 10.10.19.

SEE U.S.P. 1,329,183 and 1,357,168 of 1920; J.L. 1920, 277 A, and preceding.

XIXB.—WATER PURIFICATION; SANITATION.

Nitrites [in water]; Detection of —. L. Thévenon. J. Pharm. Chim., 1920, 22, 336–347.

TEN c.c. of the nitrite solution is treated with 8 drops of 3% acetic acid and 3 c.c. of 0.5% p-aminophenol solution; a stable red coloration at once develops. The test may be used for the detection of nitrites in water, since the reaction is not given by nitrates, and the salts usually occurring in natural waters do not interfere.—W. P. S.

Air analysis apparatus. Frederick. See XXIII.

PATENTS.

Polysulphides [fungicides]; Method of stabilising — and the products of such method. D. Davidson, and Bradley and Vrooman Co. U.S.P. 1,338,678, 4.5.20. Appl., 6.10.19.

A POLYSULPHIDE in solution is incorporated with a protein substance, such as glue, and the solution is evaporated to yield a solid product consisting of particles of polysulphide encased in glue. The solid product contains 3–15% of glue.

Insecticide. Chem. Fabr. vorm. Weiler-ter Meer. G.P. 324,757, 11.7.19.

AN insecticide is composed of hydro-derivatives of naphthalene, such as α - or β -dihydro- or tetrahydronaphthalene, either alone or mixed with other substances.—J. S. G. T.

Germs, ferments, and toxins; Destruction of —. H. Bechhold. G.P. 325,505, 27.3.18.

Porous materials or powders, which present a large surface, are prepared for use as bactericides etc., by being coated with different metals to promote galvanic action, non-metallic substances being also added if desired. The germs are adsorbed at the surface of the coated material and are destroyed. Whilst bolus coated with silver has little activity when used alone, it greatly increases the activity of bolus coated with copper.—W. J. W.

Centrifugal separator and process [for purifying water]. R. A. Sturgeon. U.S.P. 1,356,665, 26.10.20. Appl., 11.4.19.

SEE E.P. 125,502 of 1918; J.L. 1919, 437 A.

Nitrogen compounds from sewage. E.P. 152,357. See XVI.

Medicinal preparations. G.P. 324,747. See XX.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Chloropicrin and water; Intersolubility of —. T. G. Thompson and J. H. Black. J. Ind. Eng. Chem., 1920, 12, 1066–1067.

CHLOROPICRIN was emulsified with water and the separating aqueous layers kept at 25° C. for 6 hrs. and the amount of chloropicrin determined as follows: The liquid (100 c.c.) was treated with alcoholic sodium sulphite solution, and then evaporated to 10 c.c. beneath a reflux air condenser to remove alcohol. The residue was diluted to 100 c.c., treated with excess of standard silver nitrate solution and 10 c.c. of nitric acid, boiled and cooled, and the excess of silver nitrate titrated

with ammonium thiocyanate solution. The amount of chlorine was then calculated into the equivalent chloropierin. The results obtained showed that chloropierin is only slightly soluble in water, the solubility decreasing with rise in temperature, whilst water is only slightly soluble in chloropierin, the solubility increasing with the temperature. (*cf.* J.C.S., Jan., 1921).—C. A. M.

Chloropierin; Control experiments in the manufacture of —. I. Effect of varying quantities of lime upon the yield of chloropierin. H. L. Trumbull, G. T. Sehl, W. I. Burt, and S. G. Seaton. *II. Active chlorine in the sludge from the manufacture of chloropierin.* H. L. Trumbull, S. G. Seaton, and H. Durham. *J. Ind. Eng. Chem.*, 1920, 12, 1068—1069.

UNDER conditions closely approximating those used in the manufacturing process, an excess of lime had no influence upon the yield of chloropierin obtained by the interaction of picric acid, lime, and bleaching powder. Iodometric estimation of the active chlorine in a sludge from a plant in which a fairly large excess of bleaching powder had been used to decompose the calcium picrate during the steam distillation showed that very little was retained. Similar results (0.22–0.32%) were obtained with the sludges from 10 other stills.—C. A. M.

$\beta\beta$ -Dichloroethyl sulphide; Solubility of — in petroleum hydrocarbons, and its purification by extraction with these solvents. T. G. Thompson and H. Odeen. *J. Ind. Eng. Chem.*, 1920, 12, 1057—1062.

DICHLOROETHYL sulphide (twice distilled at 13–14 mm.) is soluble in petroleum hydrocarbons at relatively low temperatures, the critical temperature of solubility increasing with the increase in the percentage of the more complex hydrocarbons. The following critical temperatures were observed: With ligroin (portion distilling below 100° C., sp. gr. 1.6677 at 25° C.), 19° C.; with gasoline (40% below 120° C., 90% below 180° C.), 20.4° C.; kerosene (30% below 200° C., 80% below 230° C.), 25.6° C., and railroad light oil (20% below 200° C., 80% below 260° C.), 37° C. Sulphur monochloride is soluble in all proportions, whilst only a small amount of sulphur dissolves in the petroleum hydrocarbons. Chlorinated mustard gas (prepared by the action of sulphur dichloride on dichloroethyl sulphide in carbon tetrachloride) dissolves in all proportions in railroad light oil above 85° C., but its critical solubility temperature is considerably lower than that of dichloroethyl sulphide. Dichloroethyl sulphide may be extracted with hydrocarbons from the crude commercial product at a temperature slightly above the critical solubility temperature, whilst most of the sulphur, together with the tar-like mass will remain undissolved. On then cooling the supernatant solution considerably below the critical temperature, two liquid layers will be formed, the lower of which is mainly a solution of the hydrocarbon in the mustard gas. If the temperature is lowered still further "solid mustard gas" will be precipitated. The upper layer of hydrocarbon solvent will contain all sulphur monochloride present, chlorinated products of dichloroethyl sulphide, and a certain amount of the sulphide. The lower layer will contain dichloroethyl sulphide and a certain amount of the hydrocarbon. By this means it is possible to extract dichloroethyl sulphide on a large scale from commercial mustard gas. Yields of 51–69% were obtained from a crude product containing 71%. —C. A. M.

Mustard gas; Decomposition of and pressure developed by — in steel shell at 60° C. W. A. Felsing, H. Odeen, and C. B. Petersen. *J. Ind. Eng. Chem.*, 1920, 12, 1063—1065.

MUSTARD gas, whether made at 60° C. or by Levin-

stein's process (*cf.* Green, J., 1919, 363 *n*) undergoes slight decomposition in a steel shell at 60° C., sulphur (5–8% of the liquid contents) being deposited. The pressure developed within the shell reaches a maximum at about 2 atm. after about 9 days. The acidity shows a considerable increase, but not sufficient to cause serious corrosion. Preliminary treatment of the mustard gas with gaseous ammonia retards the formation of pressure to a sufficient extent to justify its use in the case of mustard gas which is to be stored for some time in steel shells or containers.—C. A. M.

Mustard gas; Precipitation of sulphur from crude — by means of ammonia. W. A. Felsing and S. B. Arenson. *J. Ind. Eng. Chem.*, 1920, 12, 1065—1066.

THE "free" sulphur retained by mustard gas produced from sulphur monochloride and ethylene may be partly separated by moisture, freezing, and standing for a long time. By treating the product with moist ammonia 40–45% of the sulphur in this form is precipitated. Since its removal does not materially affect the m.p. of the mustard gas, it is probable that this sulphur is present in the colloidal state, whilst the remainder (55–60%) appears to be present in the form of compounds.—C. A. M.

[Organic] acids; Identification of — by phenacyl bromide. [Injurious action of phenacyl bromide on the skin.] H. E. Cox. *Analyst*, 1920, 45, 412.

IN using the method described by Rather and Reid (J., 1919, 199 *A*) care should be taken that the phenacyl bromide does not come into contact with the skin since it produces very painful blisters. —W. P. S.

Amyl alcohol; Catalytic dehydration of fermentation —. J. B. Senderens. *Comptes rend.*, 1920, 171, 916–919.

WHEN fermentation amyl alcohol is dehydrated by passing its vapour over aluminium silicate at 340–350° C., the product consists of a mixture of the three isomeric hydrocarbons, β -methyl- Δ^2 -butylene, β -methyl- Δ^3 -butylene, and γ -methyl- Δ^2 -butylene. If the product is collected in three equal fractions it is found that the relative proportions of these hydrocarbons in the fractions are 704:180:25, 595:238:65, and 302:392:182, respectively. The isomeric change of β -methyl- Δ^2 -butylene into β -methyl- Δ^3 -butylene diminishing as the catalyst decreases in activity.—W. G.

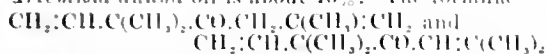
Cineol in eucalyptus oils; Cresineol method for the determination of —. T. T. Cocking. *Perf. Essent. Oil Rec.*, 1920, 11, 362–364.

IN reply to Bennett and Salamon (J., 1920, 704 *A*) the author claims that the phosphoric acid method gives results lower than the true content by amounts varying up to 13%, whereas the results by the cresineol process (J., 1920, 610 *A*) will be only 0–3% low if the non-cineol portion of the oil consists solely of terpene, or 0–3% high if of sesquiterpene. Even in presence of large quantities of other constituents such as esters, which in fact never occur to any appreciable extent in B.P. eucalyptus oils, the maximum error is only 6.3% for a mixture of 2 pts. of cineol with 1 pt. of ester.—G. F. M.

Essential oil of Artemisia annua, L. II. Constitution of artemisia-ketone. Y. Asahina and S. Takagi. *Yakugaku Zasshi* (J. Pharm. Soc., Japan), 1920, No. 464, 837–864.

ARTEMISIA-KETONE isolated from *Artemisia annua* oil (J., 1918 73 *A*) has been found to contain an isomer, and some constants given previously therefore need correction. When oxidised with potassium bichromate and sulphuric acid or concen-

trated nitric acid, tetrahydro-artemisia-ketone yielded acetone (?), acetic acid, and dimethylethyl-acetic acid, whilst artemisia-ketone yielded dimethyl-malonic acid when oxidised with potassium permanganate. By evaporating, *in vacuo*, the mother liquor, from which artemisia-ketone-semicarbazone was isolated, a new ketone was obtained as its semicarbazone compound, m.p. 70° – 72° C. The free ketone, isoartemisia-ketone, $C_{15}H_{24}O$, b.p. 182° – 183° C., sp. gr. 0.8711 at $17^{\circ}/15^{\circ}$ C., n_D^{20} 1.46880, resembles artemisia-ketone in its physical properties, but does not give a semicarbazone. It is converted into tetrahydro-artemisia-ketone when reduced with hydrogen in the presence of platinum-black, and is considered to be an $\alpha\beta$ -unsaturated compound. Artemisia-ketone is converted into the iso-compound by heating with 5% alcoholic sulphuric acid. The content of the iso-ketone in *Artemisia annua* oil is about 10%. The formulae



are given for artemisia-ketone and iso-artemisia-ketone respectively.—K. K.

Sulphur monochloride in mustard gas. Felsing and others. See VII.

PATENTS.

Alcohol or ether; Process and apparatus for the manufacture of—[from ethylene]. A. A. L. J. Damiens, M. C. J. E. de Loisy, and O. J. G. Piette. E.P. 152,495, 16.9.19.

ETHYLENE is absorbed, *e.g.*, from coal gas or coke-oven gas, by sulphuric acid of 66° B. (sp. gr., 1.84) containing one or more catalysts, and the sulphovinic acid formed is decomposed by subsequent distillation after appropriate dilution according to the relative proportions of alcohol and ether desired. Metallic sulphates or sulphuric, phosphoric, phosphorous, or arsenious anhydride may be employed as catalysts; good results are obtained with 1–2% of sulphuric and phosphoric anhydrides, 1–5% of ferrous sulphate, cuprous sulphate, etc. Absorption may be effected in the cold, or preferably at higher temperatures, *e.g.*, 100° – 120° C. Before absorption the ethylene, or gas mixture containing it, is dehydrated and if necessary freed from traces of higher hydrocarbons of the ethylene or acetylene series; and this may be effected by means of acid which has just been saturated with ethylene. The dilute acid from which the alcohol and ether have been distilled may be concentrated again for re-employment as absorbent, by means of gas which has been dehydrated by the process and by means of heat from waste furnace gases. The apparatus comprises a system of purification and absorption columns and distillation plant.—J. H. L.

Solvents [ethyl and methyl chlorides] for extraction purposes. A. Henning. E.P. 152,550, 21.12.19. Addn. to 131,573 (J., 1919, 751 A).

SOLVENTS for the selective extraction of fats, oils, resins, perfumes, etc., consisting of mixtures of ethyl and methyl chlorides, are produced from commercial mixtures of ethyl and methyl alcohols by the process of the chief patent. The mixture of chlorides first obtained may be fractionated to produce on the one hand mixtures relatively rich in methyl chloride, and on the other hand pure ethyl chloride. The use of the solvents for purposes of extraction is also claimed.—J. H. L.

Alkaloids; Process for the selective isolation of—Chem. Fabr. vorm. Sandoz. E.P. 153,219, 16.8.19. Addn. to 125,396 and 131,197 (J., 1920, 349 A, 675 A).

THE crude alkaloidal mixture, *e.g.*, opium, is mixed, if necessary, with previously extracted powdered

vegetable tissue or leaf dust which has been mixed with a concentrated solution of aluminium sulphate; the mixture is then exhausted with benzene, preferably containing up to 10% of alcohol; strongly basic alkaloids, such as morphine, are retained, whilst feebly basic ones, such as narcotine, pass into the extract. The alkaloids in the extract are mixed with more vegetable tissue which has been acidified with sulphuric acid, and the moist powder is exhausted with benzene for the removal of fatty matter. Separation of the alkaloids in each case from the powdered vegetable tissue then follows according to the method described earlier (*loc. cit.*).—D. F. T.

Sodium and potassium formates; Preparation of—Elektrochem. Werke G.m.b.H., and D. Strauss. G.P. 303,894, 1.10.16.

THE solution obtained by decomposing calcium formate with sodium or potassium sulphate, and containing calcium formate and sulphate and alkali sulphate, in addition to alkali formate, is treated with sodium or potassium oxalate and with barium formate. The precipitate formed consists mainly of calcium oxalate with a small quantity of barium sulphate, and may be used for the manufacture of oxalic acid. The solution containing nearly pure alkali formate is evaporated, giving a product suitable for the preparation of alkali oxalate by the known fusion method.—C. A. M.

Oxalic acid; Process of preparation of—[from sugar]. G. H. Gontard and A. Keller, Assrs. to Chemical Foundation, Inc. U.S.P. 1,356,137, 19.10.20. Appl., 30.12.14. Renewed 16.3.20.

SUCROSE (100 pts.) is treated with a mixture of pure sulphuric acid (320 pts.), pure nitric acid (300 pts.), and water (380 pts.) in the presence of a catalyst.—D. F. T.

Salves and emulsions; Bases for—capable of holding a large proportion of water. I. Lifschütz. G.P. 321,012, 17.2.17.

METACHOLESTEROL (G.P. 318,900; J., 1920, 576 A) is mixed or melted with fats or oils. Very pale salves and emulsions can thus be produced possessing great softness and uniformity and a high water-content. Metacholesterol melted with 2% of vaseline is capable of taking up 500% of water.—J. H. L.

Medicinal preparations; Production of—from basic organic dyestuffs and yeast protein. A.-G. vorm. Haaf und Co. G.P. 324,747, 3.4.11.

YEAST protein obtained by liquefying yeast with sodium chloride is coloured with basic organic dyestuffs possessing antiseptic properties, preferably Methyl Violet, Auramines or Safranines, and then heated to about 125° C. by means of superheated steam to fix the dyestuffs and render them inseparable from the protein by washing. The products retain the antiseptic properties of the dyestuffs, and may be used as foodstuffs, disinfectants or for medicinal purposes. That containing Methyl Violet (Pyocetanin) serves for the prevention and treatment of foot-and-mouth disease.—J. H. L.

Benzoic acid; Manufacture of—Commercial Research Co., Asses. of H. W. Walker and V. Coblentz. E.P. 141,333, 19.3.20. Conv., 7.4.19. SEE U.S.P. 1,332,028 of 1920; J., 1920, 291 A.

Mydriatic and process of making same. W. N. Nagai, Assr. to M. D. Bunnell. U.S.P. 1,356,877, 26.10.20. Appl., 1.4.16.

SEE E.P. 120,936 of 1917; J., 1919, 90 A.

Phenolphthalein: Process for preparing combinations of — with carbonates of alkali metal. A. Von Sztankay, Assr. to The Chemical Foundation, Inc. U.S.P. 1,357,482, 2,111,201. Appl., 19.6.14.

SEE G.P. 236,020 of 1913; J., 1915, 1167.

Hydrogenation processes. E.P. 152,740. See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Panchromatic sensitiveness without dyes; Production of —. J. G. Capstaff and E. R. Bullock. Communication No. 92 from Eastman Kodak Research Laboratory. Brit. J. Phot., 1920, 67, 719—720.

THE fog obtained on the development of some specially treated plates was found to be due to colour-sensitising of the film followed by fogging by the dark-room lamp. The colour-sensitising was produced by a preliminary bathing in a solution of sodium bisulphite and subsequent thorough washing; the degree of sensitising increases with the length of washing up to certain limits dependent on temperature and other conditions. Washing with distilled water is not effective nor is washing with tap water without the preliminary treatment. Addition of alkali to the washing water, or a separate bath of alkali, increases the sensitising effect, and washing in neutral potassium bicarbonate solution is more effective still. Very small quantities of chlorides and bromides in the washing water, but not in the bisulphite bath, reduce the effect. Sensitising is also produced by a preliminary bath of sulphurous acid, but not by one of normal sodium sulphite. The sensitising effect obtained extends to $\gamma=800\mu$.—B. V. S.

Photographic deposits; Measurement of graininess in —. L. A. Jones and N. Deisch. Communication from Eastman Kodak Research Laboratory. Brit. J. Phot., 1920, 67, 689—693, 706—709.

By graininess is meant a visual appearance in a photographic negative or positive of a lack of homogeneity in the silver deposit. It may be due to the size and distribution of the individual particles when viewed under high magnification, to the grouping of the particles in clumps, under small magnification as in projection or enlargement, or to the further agglomeration of the clumps, visible to the unaided eye. The second form is the one chiefly dealt with. An apparatus is described by means of which it is possible to obtain a number to express graininess which is reasonably free from subjective influence. An image of the object under examination is projected by means of a microscope, giving known and adjustable magnification, on to a screen of magnesium carbonate alongside another patch of the screen illuminated by a light of controlled intensity. The two patches are viewed through a mirror, the distance of which can be adjusted until the grainy appearance of the image first disappears. The standard for comparison is a cross-ruled screen, 500 lines to the inch. If D_1 , M_1 are the distance and magnification respectively of the cross-line screen, D_2 , M_2 those of the object, N the ruling of the screen, and E the ruling equivalent to the object, then the graininess being inversely proportional to E is $10^6 M_2 D_1 / N M_1 D_2$, the constant 10^6 being arbitrarily chosen. Graininess generally varies with density; observations are therefore usually made on photometric strips and logs, graininess plotted against density. Graininess also varies with γ , the degree of development, but this has not been fully examined. The shape of the graininess-density curve is constant for a given

batch of emulsion and approximately so for different batches of a given emulsion, but varies greatly with different emulsions. Graininess varies considerably with different developers, although not appreciably with changes of bromide or of temperature for the same developer. Of two pyro developers examined the less alkaline gave the less graininess, while of three quinol-carbonate developers the least alkaline gave the most graininess, but a caustic quinol developer gave still more graininess. As the size of the individual grain is independent of the developer used, graininess cannot be a function of grain-size.—B. V. S.

PATENTS.

X-ray plate holders [and intensifying screens]. Siemens und Halske A.-G. E.P. 143,261, 3.7.17.

AN enhanced intensifying effect, dependent on the emission of secondary radiation by bodies exposed to primary X-rays, is obtained by backing up the sensitive film by a composite screen containing elements of varying atomic weights, the lighter elements being nearer the sensitive film. For example, a screen may consist of 2 or 3 layers of tin foil, a layer of mercurous iodide, and a layer of lead, the tin foil being in contact with the sensitive surface of the plate. The graininess of the ordinary intensifying screen composed of a mineral salt is avoided.—B. V. S.

Silver compounds and products; Process of reducing the [light-] sensitiveness of —. N. Sulzberger. U.S.P. 1,356,236, 19.10.20. Appl., 26.4.15. Renewed 7.9.20.

THE light-sensitiveness of photographic silver compounds is reduced by treatment with a solution of a soluble ferrocyanide.—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Trinitrotoluene; Utilisation of — as a blasting explosive. C. E. Munroe and S. P. Howell. U.S. Dept. of Agric., Circular 94, May, 1920, 24 pages.

TESTED with the pendulum friction device and the impact machine, T.N.T. showed less sensitiveness to percussion than straight or gelatin dynamites or picric acid, and it failed to detonate when fired at from a 35-cal. rifle. In the explosion by influence test, at 20° — 23° C., with a No. 6 detonator negative results were obtained at 6 in., and a positive effect was sometimes obtained at 5 in. On the other hand, with a No. 8 detonator, explosion always occurred at 5 in. and sometimes at 6 in. 20% ammonia dynamite was detonated with a No. 6 detonator at 8—9 in. The ballistic pendulum unit defective charges of grades I., II., and III. were 201, 199, and 209 g., that of 40% dynamite being 227 g.; and the propulsive capacities were respectively 113, 114, and 109% of that of 40% dynamite. The velocity of detonation of the three grades, of which the apparent specific gravities were 0.91, 0.88, and 0.86, was 4747, 4852, and 4482 m. per sec., equivalent to 99, 102, and 94% of that of 40% dynamite of apparent sp. gr. 1.24. In the Trauzl block test granulated T.N.T. produced an expansion equal to 113.1% of that given by 40% dynamite. Comparative tests of the three grades of T.N.T. and of 40% straight and ammonia dynamites showed that T.N.T. grade III. is best so far as water-resisting properties are concerned. T.N.T. was successfully detonated with a No. 8 detonator after addition of 10% of water, and with a No. 6 detonator when mixed with 5% of water. As desensitising agents, to prevent detonation of T.N.T. with a No. 8 detonator, 14% of water, or 16% of kieselguhr, or 96% of sodium chloride may be added; and to prevent continuous combustion, 2% of water or 52% of kieselguhr is sufficient. The results of prac-

tical field tests with T.N.T. by the U.S. Bureau of Mines and by the Agricultural College of Wisconsin University indicate that the explosive can be safely used for splitting boulders, removing stumps, and splitting logs, the best effects being obtained with a No. 8 detonator and justifying its increased cost as compared with a No. 6. The amount of T.N.T. required is about 75% of the necessary charge with 20% ammonia dynamite; overloading of improperly cartridge material may prove dangerous. Moisture does not materially affect the work, and grade III. will detonate completely even under water, so that the removal of stumps may be carried out in the wet season, when the soft ground facilitates the boring of holes and the working cost is therefore considerably reduced. For most purposes grade III. is found to be most suitable. The health of the workers is not noticeably affected in open-air work.—W. J. W.

Trinitrotoluene; Products of detonation of —. C. E. Munroe and S. P. Howell. Proc. Amer. Phil. Soc., 1920, 59, 191—223. (Reprint.)

THE trinitrotoluene used had solidif. p. 80.2° C., and nitrogen content 18.32% as determined by Dumas' method, or 18.14% as found by a modification of the Kjeldahl method. A Bichel pressure gauge was used, the charges being wrapped in tin-foil and exploded with a No. 8 detonator, and the detonation products were analysed in Burrell's modification of the Orsat apparatus (J. Ind. Eng. Chem., 1916, 8, 228—231). From ten experiments with trinitrotoluene charges of 25, 50, 75, 100, 150, 200, 250, 300, 350, and 450 g., with loading densities increasing from 0.0016 to 0.003, the maximum and minimum amounts of gaseous products were as follows:—CO₂, 6.4, 1.1%; CO, 60.7, 53.2%; H₂, 27.6, 19.9%; CH₄, 2.3, 0.1%; N₂, 15.6, 13.3%. An odour of ammonia was detected in every case; its estimation in a single experiment gave 0.031. Two experiments with trinitrotoluene of density 0.86 and 1.50 respectively showed that the density causes no alteration in the detonation products. Solid combustion products were found in the gauge after the experiments, the amounts from three 200 g. charges being 5.5, 8.0, and 9.25%, and from a 400 g. charge 3.75 g. The amount of solid matter appears to decrease with the weight of the charge and to increase with its density. The composition after eliminating contaminating substances was:—H, 1.03%; C, 96.07%; N, 2.90%. A determination of the gases absorbed by the solid products indicated 6.11 c.c. per g.; the composition of the absorbed gas was CO₂, 71.3%; CH₄, 1.0; N₂, 27.7%. The amount absorbed is too small to affect the analysis of the gaseous detonation products.—W. J. W.

Mercuric azide; Preparation and properties of —. A. Stettbacher. Z. ges. Schiess- u. Sprengstoffw., 1920, 15, 211—212.

THE extraordinary sensitiveness of mercuric azide prohibits its manufacture and employment commercially, but it may be prepared with comparative safety on a laboratory scale if stringent precautions are adopted. A solution of 6.5 g. of sodium azide is decomposed with concentrated sulphuric acid, and the gaseous hydrogen nitride is passed into 10 g. of mercuric oxide in 200 c.c. of boiling water. The hot mercuric azide solution produced is passed through a filter provided with a hot-water jacket, and the filtrate is gently stirred with a wooden splinter during cooling to prevent formation of crystals longer than 1 mm., which are extremely sensitive even under water, and to secure the azide as a crystalline mass in which state it is not appreciably more sensitive than silver fulminate. Great care is demanded in the handling of the filter and funnel after filtration on account of possible deposition of needle crystals. Mercuric azide may

also be produced as a powdery mass of less sensitiveness than lead azide by mixing solutions of sodium azide and mercuric nitrate. The sensitive form may be produced from this variety by solution and crystallisation. Although mercuric azide has the same molecular weight as mercuric fulminate, and develops an equal volume of gas on detonation, its brisance is about 20—30 times as great.—W. J. W.

Smokeless powders; Determination of mineral residue in —. C. Lutri. Giorn. Chim. Ind. Appl., 1920, 2, 214—216.

FIVE g. of the finely divided powder is treated with about 8 g. of pyridine in a beaker which is immersed at intervals in a water bath. After 48 hrs. the material will have been converted into a mobile liquid; the contents of the beaker are treated with 20 c.c. of alcohol and filtered through a Gooch crucible, and the residue washed with alcohol, ether, hot benzene, dilute hydrochloric acid, and water, and then ignited to incipient incandescence and weighed. The method is also suitable for the determination of graphite in smokeless powders.

—C. A. M.

Hexanitrodiphenyl; Sensitiveness of — to mechanical shock as compared with that of hexanitro-compounds of similar constitution. C. F. van Duin. Rec. Trav. Chim., 1920, 39, 685—688. (Cf. J., 1919, 697 A; 1920, 676 A.)

HEXANITRODIPHENYL is more sensitive to mechanical shock than trinitrobenzene, but is much less sensitive than trinitrophenylmethylamine and hexanitrodiphenyl oxide and sulphide.—W. G.

PATENTS.

Propellant powder, and process of making same. C. I. B. Henning, Assr. to E. I. du Pont de Nemours and Co. U.S.P. 1,357,865, 2.11.20. Appl., 3.3.20.

AN oxygen derivative of a metal capable of reducing fouling is added in a finely-divided condition to a propellant powder.—W. J. W.

Detonating agent for shells, mines, and detonators. Westfälisch-Anhaltische Sprengstoff A.-G. G.P. 291,830, 20.4.15.

DINITROALKYLOXAMIDES, especially dinitrodimethyl-oxamide, may be used as detonating agents. Dinitrodimethyl-oxamide is a white substance, which does not attack the skin, and is not toxic. It gives a lead block expansion equal to that of picric acid, but is less sensitive to shock than the latter, and also than aliphatic nitric esters, such as gun cotton.—W. J. W.

Smokeless powder; Manufacture of —. Chem. Fabr. vorm. Weiler-ter Meer. G.P. 300,635, 14.9.16.

TO increase the stability, and reduce the ignition temperature of nitrocellulose, or nitrocellulose and nitroglycerin powders, alkylarylexanamides or diarylexanamides, such as methylphenylexanamide or diphenylexanamide, are added.—W. J. W.

Smokeless powders; Treatment of —. Claessen. G.P. 300,726, 25.4.17.

SMOKELESS powder is treated superficially with a fusible, aromatic nitro-compound in order to promote progressive combustion and to control the gas pressure.—W. J. W.

Nitration of webs of cellulose; Process for the —. T. Niederländer. G. P. 300,749, 14.5.16.

THE cellulose (e.g., paper) is passed through the nitration bath by means of mechanically operated suction rollers, whereby the nitration acid is drawn through the cellulose. To avoid decomposition of the nitrocellulose after nitration, a water spray is

employed during the subsequent operations (pressing etc.) preceding pulping.—W. J. W.

Compressed explosive charges; Production of —. von Schrötter. G.P. 304,299, 9.11.17.

THE sensitiveness of explosive charges may be reduced by melting the explosive, and then breaking it up into large fragments before pressing.

—W. J. W.

Explosive for military purposes; Manufacture of an —. Deutsche Sprengstoff A.-G. G.P. 325,612, 23.4.18.

NITROCELLULOSE is mixed under water with insoluble nitrohydrocarbons, such as trinitrotoluene or trinitronaphthalene, and the wet mixture is then compressed. The finished charges can be detonated either in a wet or dry state.—W. J. W.

Incendiary compositions; Manufacture of —. Oberschlesische Sprengstoff A.-G. G.P. (A) 302,420, 10.8.16, and (B) 302,600, 12.9.16.

(A) An incendiary composition consists of nitrocellulose dissolved in a combustible solvent which may have an oxygen-carrier in suspension. (B) To ensure rapid and safe ignition with an enhanced effect, a mixture of perchlorate, nitrohydrocarbon, and wood meal may be incorporated with the material.—W. J. W.

Fireworks composition; Manufacture of —. E. Bock. G.P. 304,356, 29.10.16.

IN fireworks composition, such as Bengal lights, charred woodmeal or sawdust may be substituted for sulphur, resin, shellac, etc.—W. J. W.

XXIII.—ANALYSIS.

Litmus paper; Treatment of red —. T. J. Ward. Analyst, 1920, 45, 412—413.

RED litmus paper, as bought, is not readily wetted by aqueous solutions owing to the sizing of the original paper with rosin. The latter may be removed by immersing the books of papers for some hours in alcohol containing 1% of concentrated hydrochloric acid and then extracting them with alcohol in a Soxhlet apparatus until no more red colouring matter is removed. The books are then dried at 100° C. Some of the red colouring matter of the litmus is extracted in addition to the rosin.

—W. P. S.

Air analysis apparatus; Improvement in the Haldane general —. R. C. Frederick. Analyst, 1920, 45, 409—410.

THE combustion chamber of the apparatus is made $\frac{3}{4}$ in. shorter than usual, the stem $1\frac{1}{8}$ in. longer, and the mark on the stem is placed $\frac{1}{2}$ in. above the shoulder. These modifications prevent, to some extent, the obscuration of the graduation by the products of combustion, and frequent cleaning is not required. Further, the removal of drops of mercury collecting in the bore of the inlet tap is rendered easier.—W. P. S.

Prism-hardness [of metals etc.]. B. P. Haigh. J. Inst. Mech. Eng., 1920, 891—913.

TEST pieces, preferably square prisms, of the same materials are used to indent one another, when placed cross-wise, and consistent hardness values are obtained by dividing the applied loads by the square of any characteristic linear dimension of the depression produced; measurement of the width gives the greater accuracy, but the depth is more easily measured. In the tests carried out the prism

hardness, P.H., was calculated from the formula $P.H. = P/x^2$, where P is the load and x the width of the indentation from corner to corner along the cutting edge. Among the metals and alloys tested were copper, annealed and hard drawn; naval brass; delta metals; pearlitic steels with 0.18 and 0.43% C, forged, water quenched, water quenched and tempered, and oil quenched; spring steel; austenitic manganese steel; and martensitic steel. Consistent results were obtained, the test being sensitive to slight differences of hardness even in the hardest metals. The scale of hardness given by this test is equally applicable to the hardest and softest metals.—J. W. D.

Hardness; Measurement of high degrees of —. J. Innes. J. Inst. Mech. Eng., 1920, 915—932.

THE hardness (H) of a material depends on the elastic properties, and the following formula is given as a means of determining and measuring it:—

$$H = \frac{\pi^2}{.96} \cdot \frac{(3+4N)^2(3+N)}{N^3} \cdot \frac{Q^3}{C^3}$$

where N is the ratio of modulus of change of size to modulus of change of shape, C the modulus of change of shape, and Q the limiting shear stress. Those three factors can readily be determined separately, and if test pieces of moderate size (rods of 0.1 in. diam. and 3 in. long) are available, two of those constants, C and N, may be measured with almost any desired degree of accuracy. Q is more difficult to measure, especially with brittle materials.—J. W. D.

Hardness testing. H. S. and J. S. G. Primrose. J. Inst. Mech. Eng., 1920, 933—954.

Two new types of testing machines are described, the elastic column dynamometer and a hardness machine with a pendulum dynamometer. In the first type use is made of the principle of the elastic deformation of three sufficiently strong steel columns to measure the load applied to the test piece. This deformation is magnified and measured by means of a mercury calibrating box. The load is applied by a 5-ton screw press. The general arrangement of the method is described, and also a simple form of depth indicator, by means of which the depth of the ball or cone imprint made in the test piece while still in the machine can be measured accurately to 0.01 mm. In the second type of testing machine a depth indicator is used for accurately reading the depth of the imprint; the load is applied by means of an oil-operated hydraulic press, the load indications of which can be varied in four degrees of sensitiveness up to 5 tons, and the load applied is measured by means of a pendulum dynamometer. Tables are given showing comparisons between hardness values obtained with different steels tested by the different methods.—J. W. D.

Immersion refractometer; Use of the — for determining the concentration of aqueous and alcoholic solutions. B. Wagner. Z. angew. Chem., 1920, 33, 262—264.

THE immersion refractometer is recommended as a means for the simple and accurate estimation of the concentrations of solutions, for the preparation and checking of standard solutions, and for the estimation of purity of reagents. Readings of 0.1, or even 0.05, of a scale division can be made with accuracy. Before use the instrument should be checked with distilled water at 17.5° C., the beakers containing the solutions under examination being placed simultaneously in the bath. If the scale reads 15 after an interval of 2 to 3 mins., it may be assumed that the solutions have attained the cor-

rect temperature, but this is preferably checked by a thermometer. Differences of temperature of the prism and of the solutions will be indicated by a blurred or double line. In practice, single readings of the instrument are insufficient; they should be supplemented by readings of 15 to 20 dilutions of the solution. In the compilation of refractometer tables, average values must not be given, as equal increases in the concentration do not correspond to equal increments on the scale. Thus sodium chloride concentrations of 5, 10, 15, and 20° give readings of 37.2, 58.5, 79.2 and 99.5, which, taking water as 15, correspond to 22.2, 21.3, 20.7, and 20.3 scale divisions. When results of analysis of reagents are quoted, it is desirable to quote the refractometer value and the specific gravity, in addition to the percentage purity.—W. J. W.

Acid and basic functions; Indication of the — and their estimation. I. M. Kolthoff. *Rec. Trav. Chim.*, 1920, 39, 672—676.

The acidic or basic nature of a substance may be ascertained by determining the H-ion or OH-ion concentration colorimetrically by the method of indicators after the addition of a known amount (insufficient for neutralisation) of strong acid or base, as the case may be. For determining the quantity of acidic or basic group present, the conductivity titration method (J., 1920, 529 A, 558 A, 735 A) is recommended.—W. G.

Sulphuric acid; Gravimetric determination of — in presence of calcium, phosphoric acid, and chromium. L. W. Winkler. *Z. angew. Chem.*, 1920, 33, 287—288.

To prevent precipitation of a small amount of calcium as sulphate the substance is dissolved in 25 c.c. of 10% hydrochloric acid containing 0.5 g. of ammonium chloride, and 5 c.c. of 10% barium chloride added, drop by drop, to the boiling solution. The liquid is then diluted with 100 c.c. of water, again boiled, and filtered next day. The error due to precipitation of phosphoric acid as barium phosphate is also largely eliminated by the same method. In the presence of 0.05—0.5 g. of phosphoric acid the weight of the dried precipitate (0.3—0.05 g.) should be reduced by 1.1 mg., or that of the ignited precipitate increased by the same amount. Sulphuric acid is only incompletely precipitated from a solution of chromium sulphate, but the remainder may be separated by evaporating the filtrate and washings to dryness, treating the residue with hot water and a few drops of hydrochloric acid, and adding the amount of the precipitate to that of the first precipitate.—C. A. M.

Titanium and iron; Determination of — in rocks. F. Ferrari. *Giorn. Chim. Ind. Appl.*, 1920, 2, 497—498.

For the simultaneous determination of titanium and iron in rocks, an aliquot part of the filtrate from the silica, containing the iron in the ferric condition, is reduced by means of zinc, while a current of carbon dioxide is passed through the flask, and the titanium is titrated with Methylene Blue. Another portion of the filtrate is then titrated with titanous chloride, using potassium thiocyanate as indicator, to obtain the amount of ferric iron.

—C. A. M.

Lead; Determination of small quantities of — [in ores etc.]. J. H. Hastings. *Eng. and Min. J.*, 1920, 110, 867—868.

Is the titration of small quantities of lead sulphate in ammonium acetate solution, with ammonium molybdate solution, using tannin solution as indicator (Alexander; J., 1893, 376), the end-point is indefinite owing to the slowness of the reaction between the molybdate and the lead. This may be

overcome by adding a definite amount of standard lead solution to the test solution before the lead is titrated, allowance being made subsequently for the amount of the added lead.—W. P. S.

PATENT.

Fat in cream. U.S.P. 1,357,168. See XIXA.

See also pages (v) 812, *Hydrocarbons* (Darmois); *Fluidity of oils*, 813, *Lubricants* (Abrams and others), 815, *Naphthalenesulphonic acids* (Ambler and Wherry), 816, *Old fustic and quercitron extracts* (Justin-Mueller); *Wool and cotton fabrics* (Duyk); *Permeability and heat-conductivity of textiles* (Colombo), 818, *Sodium hyphobromite* (Fleury); *Sodium hydrosulphite* (Crowther and Heywood); *Hydrosulphite* (Formbals), 819, *Iodic acid* (Bolland); *Titanic, niobic, and tantalic acids* (Hahn and Gille); *Sulphur monochloride in mustard gas* (Felsing and others), 822, *Vanadium in steel* (Laboulay); *Arsenic and phosphorus* (Compagno), 824, *Water in fats and oils* (Oertel), 825, *Unsaturated fatty acids* (Tsujimoto), 827, *Rubber analysis* (Pearson); *Nitrogen in fertilisers* (Baldi), 828, *Alkalinity of raw sugar* (Bruhns); *Sugars* (Von Fellenberg), 829, *Reducing sugars* (Ionescu and Vargolici, also Browne); *Dextrose* (Perrier); *Determination of copper* (Bruhns); *Pycnometer* (Newkirk), 830, *Fat in sour milk* (Day), 831, *Nitrites* (Thévenon), 832, *Organic acids* (Cox); *Cineol* (Cocking), 831, *Smokeless powders* (Lutri).

Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

I.—GENERAL; PLANT; MACHINERY.

APPLICATIONS.

Aylen and Parsons. Filtering and drying apparatus, 34,997. Dec. 11.

Bayley. Centrifugal separating-machines, 35,001. Dec. 11.

Bohrmann. Process for evaporating liquids, 34,919. Dec. 10.

Bottomley, Weeks, and Merz and McLellan. Large-scale power production, 34,260. Dec. 3.

Bottomley and others, 34,261. See II.

Brown. Mixing-machines, 33,810. Nov. 10.

Edser, Reynard, and Sulman. Processes involving reactions between gases and liquids, 33,850. Nov. 30.

Fox. Means for concentrating liquids, 34,273. Dec. 4.

Gill (Sharples Specialty Co.). Centrifugal separation of substances, 33,638. Nov. 29.

Grimsley. Mixing-machines, 33,928. Dec. 1.

Hall and Mills. Grinding and crushing mills, 34,774. Dec. 9.

Henning, 34,814. See XX.

Hudson, Lyles, and Hudson, Lyles, and Co. Drying, 34,614. Dec. 8.

Jung. Filter-presses, 34,517. Dec. 7.

Morgan, and Thermal Industrial and Chemical Research Co. Producing chemical reactions by action of heat, 34,731. Dec. 8.

Ohno. Centrifugal separators, 33,755. Nov. 30.

Raper and Sutcliffe. Recovery of gases or vapours, 34,835. Dec. 9.

Thomas and Williams, 33,904. See II.

COMPLETE SPECIFICATIONS ACCEPTED.

- 10,101 (1918). Surface Combustion Co. Apparatus for mixing gases. (117,612.) Dec. 8.
 11,183 (1919). Du Pont de Nemours and Co. Recovery of solvents. (130,962.) Dec. 15.
 13,706 (1919). McIntyre. Centrifugal machines for separating liquids, semi-liquids, etc. (154,641.) Dec. 15.
 17,221 (1919). Barbet et Fils et Cie. Regulating operations in rectifiers for rectifying mixtures of liquified gases. (129,648.) Dec. 15.
 21,674 (1919). Marks (Akt. Industare). Concentration, evaporation, etc. (154,355.) Dec. 8.
 25,169 (1919). Stead. Evaporating processes. (154,762.) Dec. 15.
 28,209 (1919). Broadley. Grinding-machines. (154,434.) Dec. 8.
 1678 (1920). Bosch. Filtering or dewatering presses. (154,817.) Dec. 15.
 1975 (1920). Frey. Heat-interchange devices such as evaporators etc. (142,077.) Dec. 8.
 2827 (1920). Ellis (Foundation Oven Corp.). Distillation ovens. (154,819.) Dec. 15.
 10,227 (1920). McIntyre, and Miln and Son. Drying-cylinders. (154,508.) Dec. 8.
 10,582 (1920). Schmidt. Recovery of volatile solvents. (141,739.) Dec. 15.
 12,827 (1920). Bakema. *See* IX.

II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

APPLICATIONS.

- Armstrong. Process of making liquid fuel. 34,843. Dec. 10. (Austria, 16.2.14.)
 Bates. Production of fuel. 34,824. Dec. 9. (U.S., 10.12.19.)
 Boberg, and Techno-Chemical Laboratories, Ltd. Preparation of lignite. 34,207. Dec. 3.
 Bottomley, Weeks, and Merz and McLellan. Production of steam for carbonising plant for large-scale power production. 34,261. Dec. 3.
 Bottomley, Weeks, and Merz and McLellan. Low-temperature distillation of fuel. 34,262. Dec. 3.
 British Thomson-Houston Co. (General Electric Co.). Manufacture of carbon. 34,126. Dec. 2.
 Broadbridge, Jones, and Stenning. Production of coal briquettes. 34,339. Dec. 4.
 Cleary. Manufacture of combustible gas from liquid fuel. 23,645. Nov. 29.
 Duckham, Morgan, and Thermal Industrial and Chemical Research Co. Heating liquids by introduction into molten metal. 34,732. Dec. 8.
 Edser and Vautin. Drying coal etc. concentrates. 34,002. Dec. 1.
 Fleischer. Manufacture of coal distillation products. 34,590. Dec. 7. (Ger., 8.12.16.)
 Fonblanque and Moellar. Manufacture of gas from carbonaceous materials. 33,708. Nov. 30.
 Fonblanque and Moellar. Retorts for low-temperature carbonisation. 33,709. Nov. 30.
 Foster. 34,545. *See* VII.
 Fyeman. Separating asphaltum, bitumen, tar, etc., from mineral matter. 33,983. Dec. 1.
 Helps. Gas manufacture. 34,874-5. Dec. 10.
 Hughes. Gas-generators or producers. 35,012. Dec. 11.
 Knibbs. Distillation of solid hydrocarbon-containing materials. 34,555. Dec. 7.
 Koppers. Gas producers. 33,674. Nov. 29.
 Koppers. Regenerative retort settings. 33,675. Nov. 29.
 Koppers. Coke manufacture. 34,346. Dec. 4.
 Koppers. Ovens for producing gas and coke. 34,429. Dec. 6.

- Lamplough, and Townsmead Construction Co. Manufacture of coal etc. briquettes. 34,396. Dec. 6.
 McTier and Meddleycutt. Neutralising coal dust in mines. 34,235. Dec. 3.
 Persch. Treating petroleum etc. 34,419. Dec. 6.
 Rigby. Treatment of peat. 33,864 and 34,147. Dec. 1 and 3.
 Rigby. Manufacture of briquettes. 33,865. Dec. 1.
 Rigby. Drying peat etc. 34,473. Dec. 7.
 Seccombe. Artificial fuel. 34,151. Dec. 3.
 Thomas and Williams. Appliance for converting pressure produced by reaction of water and calcium carbide into work etc. 33,904. Dec. 1.

COMPLETE SPECIFICATIONS ACCEPTED.

- 15,324 (1919). Underfeed Stoker Co., and McEwen. Carbonisation or destructive distillation. (154,253.) Dec. 8.
 17,777 (1919). Ryan. Recovery of bitumens from shale etc. (129,992.) Dec. 15.
 18,837 (1919). Bussey. Apparatus for treating carbonaceous materials. (154,658.) Dec. 15.
 20,277 (1919). Macdonald, and Densified Peat Fuel and Products, Ltd. Composition fuel. (154,289.) Dec. 8.
 20,735 (1919). Leadbetter. Manufacture of artificial fuel. (154,319.) Dec. 8.
 21,573 (1919). Jackson (Metals Recovery Co.). Concentration of bituminous coal by flotation. (154,702.) Dec. 15.
 23,167 (1919). Holt and Burgoyne. Purification of gas. (154,742.) Dec. 15.
 28,294 (1919). Bloomfield and Morgan. Suction gas producer. (154,436.) Dec. 8.
 31,073 (1919). Underfeed Stoker Co., and McEwen. Carbonising finely-subdivided fuel. (154,458.) Dec. 8.
 32,019 (1919). Knibbs. Treatment of hydrocarbon oil. (154,464.) Dec. 8.
 2827 (1920). Ellis. *See* I.
 8796 (1920). U.S. Industrial Alcohol Co. Liquid fuels. (140,796.) Dec. 15.
 28,995 (1920). Marks (U.S. Industrial Alcohol Co.). Non-freezing fuel. (154,867.) Dec. 15.

III.—TAR AND TAR PRODUCTS.

APPLICATIONS.

- Atack. Oxidation of hydrocarbons. 34,274. Dec. 4.
 Fyeman. 33,983. *See* II.
 Kagan. Separating and purifying anthracene and carbazole. 34,733. Dec. 8.
 Scheibler. Manufacture of sulphur preparations of the thiophene series. 35,014. Dec. 11. (Ger., 28.4.14.)

IV.—COLOURING MATTERS AND DYES.

APPLICATIONS.

- British Dyestuffs Corp., Clemo, and Perkin. Manufacture of basic blue colouring matters. 33,837. Nov. 30.
 British Dyestuffs Corp., Clemo, and Perkin. Manufacture of colouring matters. 33,970. Dec. 1.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

APPLICATIONS.

- Bostock. Rendering paper translucent and durable. 34,845. Dec. 10.
 Dreyfus. Manufacture of solutions, compositions, etc., having a basis of cellulose acetate etc. 33,948. Dec. 1.
 Gassmann. Manufacture of durable masses from viscose. 34,840. Dec. 9. (Ger., 12.12.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 20,675 (1919). Burby. Process of re-working old paper. (151,670.) Dec. 15.
 20,975 (1919). Dreyfus. Manufacture of compositions etc. having a basis of cellulose acetate. (151,311.) Dec. 8.
 21,527 (1919). Drews and Noorden. Treatment of manufactured fabrics to reclaim the fibres. (151,315.) Dec. 8.
 22,146 (1919). Jennison and Chanaz. Paper manufacture. (151,366.) Dec. 8.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

- Hodson. Process of bleaching textile fabrics and materials. 31,965. Dec. 11.
 Leek, and Leek and Sons. Dyeing, bleaching, tin-weighting, scouring, etc. machines. 31,612. Dec. 8.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

- Bergdahl. Production of ammonia. 31,715. Dec. 8.
 Casalo and Leprestre. Apparatus for catalytic synthesis of ammonia. 31,124. Dec. 2.
 Casale and Leprestre. Production of nitrogen or mixtures of nitrogen and hydrogen. 31,582. Dec. 7.
 Dunningham and Hargreaves. Manufacture of sodium thiosulphate. 33,958. Dec. 1.
 Fabr. de Prod. Chim. de Thann et de Mulhouse. Manufacture of anhydrous zinc sulphide. 35,008. Dec. 11. (Fr., 16,12,19.)
 Foster. Preparing lime from limestone, and manufacture of power gas and ammonia. 34,545. Dec. 7.
 Harding and Jones. Production of sodium pentaborate, borax, or boric acid from boron ores. 34,193. Dec. 3.

COMPLETE SPECIFICATIONS ACCEPTED.

- 12,695 (1919). Cataldi. Electrolytic decomposition of chlorides. (127,255.) Dec. 15.
 20,574 (1919). British Cellulose and Chemical Manuf. Co., Soller, and Hotz. Manufacture of acetic acid. (151,304.) Dec. 8.
 20,931 (1919). Wilton. Manufacture of sulphate of ammonia. (151,328.) Dec. 8.
 21,140 (1919). Dreyfus. Manufacture of acetic acid. (154,680.) Dec. 15.
 21,690 (1919). Island. Fixation of atmospheric nitrogen. (151,356.) Dec. 8.
 22,166 (1919). Matheson. Apparatus for manufacture of acetic acid. (151,368.) Dec. 8.
 32,853 (1919). Heberlein. Electrolysis of nickel salt solution. (151,471.) Dec. 8.
 309 (1920). Datta. Production of potassium and sodium bichromates and sulphates. (151,810.) Dec. 15.
 433 (1920). Napp (Tschudi). Manufacture of hydrobromic acid. (151,472.) Dec. 8.
 4695 (1920). Diehl. *See X.*
 6366 (1920). Lamm. Manufacture of potassium and sodium carbonates. (154,498.) Dec. 8.

IX.—BUILDING MATERIALS.

APPLICATIONS.

- Alletson. Kilns for burning blue bricks. 31,576. Dec. 10.
 Earle. Hydraulic cement. 31,077. Dec. 2.
 Hensman. Treatment of timber with fluid. 33,704. Nov. 29.

- Winkler. Making mortar, cement, etc. 33,633. Nov. 29.

COMPLETE SPECIFICATIONS ACCEPTED.

- 5385 (1918). Friedrich. Producing a cold glaze on building materials, particularly cement. (151,236.) Dec. 8.
 9256 (1920). Simon. Reducing or softening the colours of exotic woods. (141,052.) Dec. 15.
 12,827 (1920). Bakema. Shaft furnaces especially for making cement. (154,510.) Dec. 8.

X.—METALS; METALLURGY. INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

- Andrews. Process for rust-proofing metal. 31,102. Dec. 2.
 Ballantine, Stig, and Stig. Refining ferro-alloys. 31,005. Dec. 1.
 Ballantine, Stig, and Stig. Production of ferro-alloys. 31,006. Dec. 1.
 Chappell and Cowing. Manufacture of nickel steel. 31,140. Dec. 2. (U.S., 16,6,20.)
 Costar, and Enamelled Wires, Ltd. Enamelling or coating wire. 31,162. Dec. 3.
 Evans and Hamilton. Manufacture of steel and alloy steels. 31,294. Dec. 1.
 Ges. f. Wolfram-Industrie. Alloy for wire-drawing dies etc. 31,575. Dec. 7. (Ger., 23,1,18.)
 Groff. Electroplating metallic bodies. 33,966. Dec. 1.
 Miller. Method of enamelling metals. 33,748. Nov. 30.
 Minerals Separation, Ltd., and Simpson. Concentration of ores containing sulphur. 31,141. Dec. 2.
 Moa Iron and Development Corp. Treating ores etc. 33,818. Nov. 30. (U.S., 8,12,19.)
 Raffo. Manufacture of iron and steel. 31,125. Dec. 2.
 Wickett. Electrical treatment of ferrous metals during casting. 31,265. Dec. 3.
 Wood. Flux and solder. 34,608. Dec. 8.

COMPLETE SPECIFICATIONS ACCEPTED.

- 12,969 (1918). Garred. Combustion processes and apparatus for smelting ores, melting metals, etc. (151,210.) Dec. 8.
 4838 (1919). Adam, Stevenson, and Mabbitt. Detining of tinned iron scrap. (154,242.) Dec. 8.
 18,337 (1919). Wiberg. Reduction of ores etc. (130,334.) Dec. 8.
 18,868 (1919). McConnell. Manufacture of electric steels. (154,659.) Dec. 15.
 21,691 (1919). Wickett. Electrical treatment of ferrous metals during casting. (154,711.) Dec. 15.
 21,819 (1919). Mackay. Treatment of sulphide ores containing lead and zinc. (151,718.) Dec. 15.
 22,376 and 29,817 (1919). Coles. Production of spring or tempered copper. (154,373.) Dec. 8.
 32,831 (1919). British Thomson-Houston Co. (General Electric Co.). Method of coating metals. (154,808.) Dec. 15.
 4695 (1920). Diehl. Arrangement for blowing air through blast-furnace slag, particularly for recovering the sulphur as sulphur dioxide. (139,173.) Dec. 8.
 22,568 (1920). Russell, and Manchester Furnaces, Ltd. Furnaces for heat-treating. (154,521.) Dec. 8.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

- Groff. 33,966. *See X.*
 Metropolitan-Vickers Electrical Co. Electrolytes for electrolytic cells. 31,117. Dec. 2. (U.S., 9,12,19.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 4948 (1919). Adam, Stevenson, Mabbitt, and Fieldhouse. Electrolysis. (154,635.) Dec. 15.
 12,695 (1919). Cataldi. *See* VII.
 18,868 (1919). McConnell. *See* X.
 21,334 (1919). Royston (Federal Storage Battery Co.). Electric storage batteries. (154,343.) Dec. 8.
 22,787 (1919). Oldham, Oldham, and Oldham. Galvanic batteries. (154,737.) Dec. 15.
 29,114 (1919). Erichsen. Electric furnaces. (151,444.) Dec. 8.
 32,853 (1919). Heberlein. *See* VII.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

- Dalley. Machine for expressing oil. 34,170. Dec. 3.
 Distillates, Ltd., and Jarman. Treatment of solid residues containing grease. 33,609. Nov. 29.
 Ham and Jarrett. Soap powder and process for preparing same. 34,805. Dec. 9.
 Schueler. Extraction of oil from seeds, nuts, etc., by means of solvent. 34,853. Dec. 10.
 Tseng. Manufacture of soap. 34,831. Dec. 9.

COMPLETE SPECIFICATIONS ACCEPTED.

- 1126 (1920). Nordiske Fabr. De-No-Fa. Catalysts for use in hydrogenating unsaturated organic compounds. (140,371.) Dec. 15.
 10,934 (1920). Townsend. Detergent. (154,843.) Dec. 15.
 14,623 (1920). Vakil. Apparatus for refining oils and fats. (154,514.) Dec. 8.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

- Fabr. de Prod. Chim. de Thann et de Mulhouse. 35,008. *See* VII.
 Kulas and Pauling. Manufacture of resinous condensation products from phenol. 33,875. Dec. 1. (Ger., 23.2.20.)
 Nosawa. Paint for ships' bottoms. 34,722. Dec. 8.
 Shimozaki. Copying-inks. 34,439. Dec. 6.
 Whalley. Recovery of varnish etc. from waste micanite etc. 33,696. Nov. 29.

COMPLETE SPECIFICATIONS ACCEPTED.

- 12,002 (1918). Soc. Anon. l'Helice Integrale. Processes of lacquering. (118,616.) Dec. 8.
 18,827 and 31,391 (1919). Vickers, Ltd., Ioco Rubber and Waterproofing Co., and Nuttall. Condensation products of phenolic bodies with aldehydic compounds. (154,656.) Dec. 15.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

- Bilbrough. Rubber compounds and process of manufacture. 33,793. Nov. 30.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

- Carmichael and Ockleston. Treatment of hides and skins. 34,481. Dec. 7.
 Maries. 34,561. *See* XIX.
 Turner. Preparation for treating leather. 34,228. Dec. 3.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

- Blichfeldt and Grove. Saccharification of starchy materials. 34,139. Dec. 2.
 Delafond. Manufacture of sugar direct from juice. 34,457. Dec. 6.
 Dutt and Dutt. Decolorising and purifying saccharine solutions. 34,413. Dec. 6.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

- Fitzgerald. Production of commercial products from blood. 33,637. Nov. 29.
 Hartley and Hartley. Purification of sewage. 34,485. Dec. 7.
 Luft. Food products. 34,026. Dec. 2.
 Maries. Manufacture of solid plastic material from casein. 34,561. Dec. 7.
 Martin and others. Manufacture of condensed milk. 34,709 and 34,710. Dec. 8.
 Martin and others. Manufacture of milk powder. 34,711. Dec. 8.
 Standen. Insecticide. 34,788-9. Dec. 9.

COMPLETE SPECIFICATIONS ACCEPTED.

- 10,457 (1917). Monhaupt. Manufacture of a colloidal solution neutral to the taste from casein and vegetable albumen including gluten. (154,627.) Dec. 15.
 13,087 (1919). Greville. Treatment of flour. (154,639.) Dec. 15.
 13,492-3 (1919). Pique, and Imperial Trust. Cooling and freezing fish etc. (154,250.) Dec. 8.
 19,825 (1919). Lendrich. Manufacture of coffee substitutes. (131,304.) Dec. 8.
 20,667 (1919). Hardy, Pique, and Imperial Trust. Cooling and freezing fish etc. (154,669.) Dec. 15.
 22,349 (1919). Adams. Separating semi-solids from sewage etc. (154,370.) Dec. 8.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

- Henning. Non-inflammable volatile liquid. 34,814. Dec. 9.
 Meister, Lucius, u. Brüning. Manufacture of derivatives 3,3'-diamino-4,4'-dioxarsenobenzene. 33,848. Nov. 30. (Ger., 12.12.19.)
 Meister, Lucius, u. Brüning. Manufacture of esters of dioxidiethyl sulphide. 34,231. Dec. 3. (Ger., 20.1.19.)

COMPLETE SPECIFICATIONS ACCEPTED.

- 1126 (1920). Nordiske Fabr. De-No-Fa. *See* XII.
 17,860 (1920). Tweedale and Tweedale. Manufacture of perfumery and flavouring essences, extracts, etc. (154,517.) Dec. 8.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATION.

- Dye Impression Photos., Ltd., and Edridge. Photographic films. 34,810. Dec. 9.

XXII.—EXPLOSIVES; MATCHES.

APPLICATION.

- Rathsburg. Manufacture of explosives and primers. 34,241. Dec. 3. (Ger., 3.2.20.)

BY-LAWS OF THE SOCIETY OF CHEMICAL INDUSTRY.

INCORPORATED BY ROYAL CHARTER 17th day of June, 1907.

BY-LAWS

To be submitted for adoption at the Annual General Meeting to be held on 13th July, 1920.

PART I.—PRELIMINARY.

Short Title and Commencement.

(1.) These By-laws made pursuant to the Charter may be cited as "Society of Chemical Industry By-laws, 1920," and shall come into operation so soon as they may be allowed by the Lords of His Majesty's Most Honourable Privy Council.

By-laws divided into parts.

(2.) These By-laws are divided into parts as follows:—

- Part I.—Preliminary.
- Part II.—Members.
- Part III.—The Council.
- Part IV.—Local Sections.
- Part V.—Subject Groups.
- Part VI.—The Journal.
- Part VII.—Financial.
- Part VIII.—Meetings.
- Part IX.—Miscellaneous.

Interpretation.

(3.) In and for the purposes of these By-laws terms and words and expressions to which meanings are assigned by the Charter or the Act made applicable thereto have the same respective meanings, unless there be something in the subject or context repugnant to such construction.

"The Charter" means the Charter granted the 17th day of June, 1907, as amended or extended by supplemental or additional Charters granted to and accepted by the Society after the date aforesaid.

"By-laws" means any By-law of the Society made and allowed pursuant to the Charter.

"Committee" means any Committee duly appointed by the Council, and where qualified by any prefix means the Committee appointed as aforesaid for the purposes denoted or indicated by such prefix.

"Treasurer" means the person appointed pursuant to the By-laws as Honorary Treasurer of the Society.

"Foreign Secretary" means the person appointed pursuant to the By-laws as Honorary Foreign Secretary of the Society.

"The General Secretary" means the Secretary of the Society appointed by the Council, or the person acting in that capacity by the direction of the Council.

"The Editor" means the Editor of the Journal appointed by the Council, or the person or persons acting in that capacity by the direction of the Council.

"Sectional Officers" means the Chairman and Hon. Secretary of the several Local Sections.

"Group Officers" means the Chairman and Hon. Secretary of the several subject Groups.

"Member" means any Member of the Society.

"The Schedule" means the Schedule of Forms annexed to the By-laws.

"The Register" means the Register of the Society of 1881, continued as the Register of the Society.

Forms.

(4.) The forms set forth in the Schedule, and such other forms as the Council may from time to time authorise, shall be used in the several cases to which they are applicable, with such amendments, alterations, or additions thereto, if any, which the Council may from time to time determine, and all notes and directions printed thereon shall be read and construed as

part of such forms, and be observed accordingly, but it shall be sufficient if the forms are substantially followed, and no act or proceeding shall be invalid merely by reason of any formal defect.

PART II.—MEMBERS.

Proposal of Candidate.

(5.) Every person desirous of being admitted as a Member of the Society shall be duly proposed as a candidate for admission upon Form (A) set out in the Schedule, or such other form as the Council may from time to time prescribe. The proposal form shall be signed by two Members, to one of whom at the least the candidate shall be known personally, and every candidate shall make and subscribe the declaration therein contained—that, if elected, he agrees to be bound by the Charter and By-laws and by any Regulations made thereunder, and to further the objects of the Society.

Election of Members.

(6.) All proposal forms of Candidates, duly signed and subscribed as hereinbefore provided, received by the General Secretary prior to an Ordinary Meeting of Council, shall be considered thereat, and the Council may in their absolute discretion, by resolution passed by a majority of those present, elect or postpone the election of any candidate as a Member of the Society, or may refuse to elect any candidate whom they consider to be not a fit or proper person to be elected as aforesaid, and upon such election may also decide whether any Member so elected shall be required to pay for the year then current the whole or any part of the annual or other subscription or other fee hereafter provided, and thereupon the General Secretary shall give notice of such election and decision to the Member elected as aforesaid, and upon such notice and the payment of the said subscription or other fee, the person so elected shall, pursuant to the Charter and By-laws, become and continue to be subject to all the duties and obligations, and entitled to all the rights and privileges of a Member.

Register of Members.

(7.) The Register shall be continued as the Register of the Society by the entry therein by the General Secretary, or other person authorised by the Council, of the names of all persons elected Members as hereinbefore provided, together with the date of their several elections, and sufficient particulars of their several professions or occupations and addresses, and such particulars shall be amended or corrected as occasion may require.

Entrance Fee.

(8.) Except as otherwise decided by the Council, every Member shall, upon notice of his election, in addition to a subscription for the current year in which he is elected, or any payment in lieu thereof, pay to the Treasurer, or other person appointed by the Council to receive Members' fees or subscriptions, an entrance fee of One Guinea, or such other amount as may for the time being be prescribed by Regulations.

Annual Subscription.

(9.) Every Member shall on the first day of January in each year pay to the Treasurer, or other person as aforesaid, the sum of Two pounds ten shillings, or such other sum as the Council may prescribe by Regulations, and every Member shall upon his election pay the like amount as his subscription for the year current in which he is elected, or such part thereof as may be

decided by the Council, but in lieu of any such payment a Member may compound for that and all future payments, as hereinafter provided.

A Member joining under the age of 25 shall be entitled to pay a subscription of £2 per annum until attaining the age of 25, or for 3 years from date of election whichever is the longer period, but subject to the provisions that his application for Membership must be accompanied by (1) satisfactory evidence of age, and (2) a declaration that he is not joining the Society as a nominee of any firm or corporation but solely in his own interests as an individual member.

Life Compositions.

(10.) A Member, upon his election, or any other Member whose subscription is not in arrear, may at any time compound for all annual subscriptions by payment to the Treasurer, or other person as aforesaid, the sum following (that is to say):—

(a) A Member whose age does not exceed 30 years, the sum of £40.

(b) A Member whose age exceeds 30 years, the sum of £35, less such sum as is ascertained to be equal to one-fourth part of the total of the sums which have been paid by such Member as annual subscriptions after reaching the age of 30 years. But before accepting any compounding payment the Council shall require from any Member desirous of compounding such evidence of his age as they may think fit.

Subscriptions in Arrear.

(11.) Any Member whose annual subscription, payable on the first day of January, is unpaid on the first day of March next following, shall, while remaining subject to the duties and obligations, not be entitled to any of the rights of a Member until such subscription shall be paid and at any time after the first day of March, the General Secretary shall give notice to all members whose subscriptions are so in arrear, and if any Member on the next ensuing thirty-first day of December shall continue in arrear, such Member shall cease to be a Member, and his name shall then be removed from the Register by the General Secretary, but without prejudice to the right of the Council to recover any such arrears or to enforce any duty or obligation to which, as a Member, he may have become liable at the time of such removal, in any Court of competent jurisdiction. Notwithstanding anything in this By-law provided, the Council in the exercise of their sole discretion may, having regard to the circumstances in which the subscription of any Member shall be in arrear as aforesaid, postpone or remit the payment thereof, and continue such Member in full enjoyment of all rights of Membership as if such subscription had not at any time been or continued in arrear.

Certain Disqualifications of Members in Arrear.

(12.) A Member who has not paid before the first day of March any fee or subscription payable and due by him shall until payment be made be disqualified for nomination as President, Vice-President, and Ordinary Member of Council, and to continue in any such office, and to become or remain an *ex-officio* Member of Council, and to sign the form of nomination of an ordinary Member of Council.

Resignation of Members.

(13.) Any Member may at any time determine his Membership by notice in writing to that effect, sent to the General Secretary, but such notice in order to operate as a determination shall be accompanied by payment of any subscription or fee which at the date of the notice shall have become payable by the Member, and which remains unpaid.

Suspension or Determination of Membership by Council.

(14.) Any Member in respect of whom a complaint of misconduct is made to the Council, which complaint, the Member first having had an opportunity of being heard, is sufficiently established, in the opinion of the Council, may, by resolution of the Council as hereinafter provided, be suspended from rights of Membership for such period as the Council may decide, or the Council may in like manner decide that his Membership shall be determined, and there-

upon such Member shall cease to be a Member, and his name shall be removed from the Register; and in the event of it being brought to the notice of the Council that any Member has been adjudged by a Court of competent jurisdiction in the country in which he is alleged to have committed an offence to be guilty of an offence which in Great Britain or Ireland would be an indictable offence punishable by imprisonment, then, the Council may order his name to be removed from the Register, and thereupon he shall cease to be a Member. A resolution of the Council as aforesaid shall not be passed unless twelve or more Members of the Council are present at the meeting at which it is proposed and not less than two-thirds of those present vote in favour of such resolution.

Introduction of Visitors.

(15.) Every Member shall be at liberty to introduce, at any meeting of a Local Section or Subject Group at which the Member introducing is entitled to be present, such visitors as the rules of the Local Section or Subject Group may prescribe.

Restriction on use of Distinctive Title or Initials.

(16.) Any Member who shall persistently use after, or in conjunction with, his name, letters or initials which purport to indicate or convey that Membership of the Society confers upon him any degree, qualification, or authority to practise in any profession or calling, or a right to use any distinctive title, after notice from the General Secretary authorised by resolution of the Council to discontinue such use, may be deemed by the Council to be guilty of misconduct, but this By-law shall not prevent any Member from *bona fide* subscribing or describing himself as a Member of the Society or any office bearer thereof.

Re-admission of a former Member.

(17.) Any former Member whose Membership has ceased or been determined under any provisions of the By-laws, may apply for re-admission to Membership, and such application shall be considered and decided upon by the Council at the next ensuing Ordinary Meeting thereof in all respects as if it were a proposal for Membership of the Society, and if such application be granted, then after notice thereof by the General Secretary to the applicant, and upon making such payments and complying with such conditions, if any, which the Council may decide, the applicant shall be re-admitted to Membership, and shall again become subject and entitled to all the duties, obligations, and rights of a Member.

PART III.—THE COUNCIL.

Constitution.

(18.) The Council shall consist of:—

(a) Twenty-five elected Members of Council, comprising:—

(i.) A President;

(ii.) Twelve Vice-Presidents;

(iii.) Twelve ordinary Members of Council;

(b) *Ex-officio* Members of Council, comprising:—

(iv.) The Sectional and Group Officers;

(v.) The Treasurer, the Foreign Secretary;

all of whom shall be Members of the Society.

The elected Members of Council, except Members appointed to fill casual vacancies, shall be elected by ballot at the time, in the manner, and for the respective periods prescribed by the By-laws, and all Members of Council in office at the commencement of the By-laws shall in their several capacities continue in office, but subject to all incidences, whether as to powers, duties, retirement, re-election, nomination, election, or otherwise, as if they had been elected and were holding office pursuant to and under the several provisions of the By-laws applicable thereto.

The President.

(19.) The President shall be nominated from amongst the Members by the Council, and such member so nominated shall be submitted by the Council for election at the Annual General Meeting in manner hereinafter provided. He shall retire from office at the next ensuing Annual General Meeting, and shall be eligible for re-election, but a Member who has been elected or appointed President two years or one

year and a part of another year in succession, shall not be eligible for election as President before the Annual General Meeting next but one ensuing after his last retirement. A retiring President not nominated for re-election shall be nominated by the Council as one of the four Vice-Presidents to be submitted for election at the next ensuing Annual General Meeting.

In the event of the illness or absence from Great Britain of the President, the Council shall have power to elect a Chairman of Council who shall act as President.

Vice-Presidents.

(20.) The Vice-Presidents shall be nominated from amongst the Members by the Council, and such Members so nominated shall be submitted by the Council for election at the next ensuing Annual General Meeting in manner provided by the By-laws.

Four Vice-Presidents shall retire from office at each Annual General Meeting, none of whom shall be eligible for election as a Vice-President, or an ordinary Member of Council before the Annual General Meeting next ensuing after the said retirement, but a Member appointed Vice-President to fill a casual vacancy shall not, upon the expiration of the term for which he is so appointed, be ineligible as aforesaid.

All Vice-Presidents shall retire in rotation in accordance with the seniority of their respective terms of office, and if in ascertaining which four of them shall retire as aforesaid, it shall appear that there is equality of term of office in the case of more than four of them of the highest degree of seniority, or in the case of two or more of them of a lower degree of seniority required to make up four retiring Vice-Presidents, the Council shall decide which of them having such respective equality of term shall retire.

A Vice-President nominated as President in accordance with the last preceding By-law shall, upon election as President, vacate the office of Vice-President, and the vacancy thereby created shall be deemed to be a casual vacancy.

Ordinary Members of Council.

(21.) Ordinary members of Council shall be nominated from amongst the Members by the Members and such Members so nominated shall be submitted by the Council for election at the next ensuing Annual General Meeting in manner prescribed by the By-laws.

Four ordinary Members of Council shall retire from office at each Annual General Meeting, none of whom shall be eligible for election as an ordinary Member of Council before the Annual General Meeting next ensuing after the said retirement, but an ordinary Member of Council appointed to fill a casual vacancy shall not, upon the expiration of the term of office for which he is appointed, be ineligible as aforesaid.

All ordinary Members of Council shall retire in rotation, in accordance with the seniority of their respective terms of office, and if in ascertaining which four of them shall retire as aforesaid it shall appear that there is equality of term of office in the case of more than four of them of the highest degree of seniority, or in the case of two or more of them of a lower degree of seniority required to make up the four retiring ordinary Members of Council, the Council shall decide which of them having such respective equality of term shall retire.

An ordinary Member of Council nominated either as President or as Vice-President in accordance with the last two preceding By-laws respectively shall, upon election as President or Vice-President, as the case may be, vacate the office of ordinary Member of Council, and the vacancy, if any, thereby created shall be deemed to be a casual vacancy.

Qualification of an ordinary Member of Council.

(22.) An ordinary Member of Council shall be nominated by five or more Members upon Form (B) in the Schedule, a copy of which form shall be furnished by the General Secretary upon the written or verbal request of any Member, but a Member shall not be eligible to sign more than one such nomination form, and the Member nominated shall sign the declaration set forth on the form. A nomination shall be declared invalid by the Council if:—

- (a) The Member thereon nominated is disqualified for election, or ineligible to be elected, as provided by the By-laws.
- (b) The nomination is not made on the authorised printed form, or substantially not in the manner directed thereon.
- (c) The nomination form is signed by less than five Members not disqualified, or not ineligible to nominate as provided by the By-laws.
- (d) The nomination form is not received before or upon the day appointed therefor.
- (e) The Member nominated has not signed the declaration printed upon the form.

A Member whose nomination aforesaid is declared to be invalid shall receive notice thereof from the General Secretary and shall not be submitted for election.

Council to Notify all Vacancies and Nominations by them.

(23.) In every year the Council shall cause a list to be prepared setting forth the names of all Members of the Council, showing which of them are due to retire at the next ensuing Annual General Meeting, and which (if any) of them are eligible for re-election, and such list shall be approved by the Council at any meeting thereof, not less than two months prior to the Annual General Meeting aforesaid, and the Council shall at the same meeting nominate the Members to be submitted to such meeting for election as President and Vice-Presidents respectively, and shall also appoint Members as Treasurer and Foreign Secretary respectively to take the places of the Treasurer and Foreign Secretary who will retire at the said General Meeting. The said list, together with the names of the Members nominated and appointed as aforesaid, shall be printed as a notice to Members, which notice shall contain a statement inviting Members to nominate the ordinary Members of Council to be elected at the said General Meeting, and appointing a day before or upon which such nominations shall be received by the General Secretary according to Form (B) in the Schedule, and the said notice, as approved, shall be sent to all the Members with, or in, the next issue of the Journal.

Nomination of ordinary Members of Council.

(24.) All nomination forms of ordinary Members of Council received by the General Secretary shall be considered by the Council at the meeting thereof next ensuing after the date appointed for their delivery, and the Members whose nominations are not then declared to be invalid, together with the Members nominated by the Council as President and Vice-Presidents respectively, shall be submitted for election at the next ensuing Annual General Meeting. But if the number of Members validly nominated shall be less than the number of vacancies, the Council shall thereupon nominate such Members as are requisite to make up the number required, and in that event, and also whenever the nominations considered and not declared to be invalid by the Council are equal to the number of vacancies, although the names of Members nominated in either case shall be submitted to the General Meeting, no ballot shall be taken thereon.

Election of Council.

(25.) The election of all elected Members of Council in whatever capacity, shall, except as provided by the By-laws, be by ballot taken upon the first day of each Annual General Meeting, and shall be conducted in manner and otherwise subject to such Regulations relating thereto as the Council may make, but a Member may record his votes either by delivering his ballot list personally or by post at the said General Meeting in accordance with the Regulations.

The Council in every year shall cause to be prepared, and shall send to all the Members not less than twenty-one days before the Annual General Meeting, a ballot list of the Council, showing the names of all Members thereof in their several capacities, and which of them are to retire, and setting forth separately thereon the names of Members submitted for election, in each capacity, and also the names of the Treasurer and Foreign Secretary appointed by the Council to succeed the retiring Treasurer and Foreign Secretary respectively, and the name of the retiring Auditor.

The ballot list shall be according to such form as the Council may from time to time authorise.

Meetings of Council.

(26.) One Ordinary Meeting of the Council at the least shall be held every month to transact such business relating to the affairs of the Society as may be necessary, except that if the Council so decide an Ordinary Meeting of the Council need not be held during the months of August and September, but a Special Meeting of the Council may be convened by the President at any time and shall be convened by the General Secretary whenever he shall receive a notice signed by not less than seven Members of Council calling upon him to convene such meeting, and stating the matter to be considered thereat, and notice thereof shall be given to all Members of the Council as and in the manner provided by the Standing Orders of the Council, and no other business shall be considered at such Special Meeting of Council. Except where otherwise provided by the Charter or the By-laws, any seven Members of the Council shall constitute a quorum.

Appointment of Committees.

(27.) Subject to the provisions of the Charter and the By-laws, the Council may appoint Committees from amongst themselves, and may delegate any of their powers to such Committees, and in furtherance of either the objects of the Society, may also appoint Committees composed partly of Members of the Council, and partly of non-members of the Council, and may fix the quorum and make rules for regulating the proceedings of any of the Committees aforesaid. The President and the Honorary Treasurer shall be Members of all Committees, and the General Secretary, unless the Council shall otherwise decide, shall act as Secretary to each Committee appointed as aforesaid.

Resignation of Member of Council.

(28.) Any Member of the Council may resign his office by notice in writing to the General Secretary of such his desire, and upon the acceptance thereof by the Council he shall cease to hold his said office, but not until then.

Vacation of Office of Member of Council

(29.) The office of any Member of the Council, whether elected or *ex-officio*, shall be deemed to be vacated for any of the following reasons:—

- (a) If any such Member ceases to be a Member of the Society, or his Membership thereof becomes suspended for any period, or determined as provided by the By-laws.
- (b) If any such Member becomes bankrupt, or suspends payment, or compounds with, or makes an assignment of his property for the benefit of his creditors.
- (c) If any such Member becomes a lunatic or is declared of unsound mind.

Casual Vacancies.

(30.) Any vacancy in the elected Members of Council, from whatever cause occurring, between any two Annual General Meetings, shall be filled by the Council. The Member appointed to fill a casual vacancy shall retire at the time when the person in whose stead he is appointed would in due course have retired if the office held by him had not become vacant, but the Member appointed shall be eligible for re-election unless it be otherwise provided in the By-laws.

Honorary Treasurer.

(31.) The Council shall appoint a Member as Honorary Treasurer of the Society, who shall, subject to the direction and control of the Council, receive and give receipts, and account for all subscriptions, fees, and other payments payable or due to or by the Society, and shall cause to be made, and shall, as occasion may require, submit to the Council a statement of the income and of current payments or expenditure which are, or ought to be, made by the Council, and shall prepare or cause to be prepared, in sufficient time to be submitted to the Annual General Meeting, a balance sheet showing the income and expenditure, and all liabilities and assets and investments of the Society for the year ending the thirty-first day of December prior to the said General Meeting, and as

far as possible a general statement of the financial affairs of the Society for the year then current.

Honorary Foreign Secretary.

(32.) The Council shall appoint a Member as Honorary Foreign Secretary of the Society, who shall, subject to the direction and control of the Council, have the management of such correspondence of the Society with Departments of State or Corporate Bodies, Societies, or persons of or in foreign countries as occasion may require, and he shall report to the Council on all matters connected therewith or relating thereto.

Appointment of Treasurer and Foreign Secretary.

(33.) The Treasurer and Foreign Secretary shall be appointed annually at the meeting of the Council at which the President and Vice Presidents are nominated, and except when appointed to fill a casual vacancy, shall remain in office from the next ensuing Annual General Meeting after their respective appointments, until the Annual General Meeting next following, and during such period shall be *ex-officio* Members of the Council. Any casual vacancy in either of the said offices shall be filled by the Council as soon as may be after the occurrence of the vacancy, but the Member appointed to fill such casual vacancy shall retire at the next ensuing Annual General Meeting after his appointment, and either of the said offices shall be deemed to be vacated for any of the reasons provided by the By-laws for the vacation of office of Member of Council.

The General Secretary.

(34.) The Council shall appoint a General Secretary of the Society of Chemical Industry at such remuneration and subject to such terms and conditions as they may from time to time decide, and may in their discretion determine the appointment of any such General Secretary, and appoint another General Secretary.

The Editor.

(35.) The Council shall appoint an Editor or Editors of the Journal, at such remuneration as they may from time to time decide, and may in their discretion determine the appointment of any such Editor and appoint another Editor. The Editors shall be responsible to the Council and subject thereto, and to the directions of any Committees delegated by the Council to manage or conduct the preparation or publication of the Journal, shall have the conduct of the literary management and the preparation of all matter, the editing, and the printing and publication of the Journal, and the organisation, control and direction of such literary or other staff as may be engaged on the Journal for the time being.

Council may make Regulations.

(36.) The Council may, except as otherwise provided by the Charter, make Regulations for carrying into effect the Charter and the By-laws and for the general management of the affairs of the Society, but a regulation shall not become operative until confirmed by a resolution passed by a majority of the Members present at a General Meeting at which such regulation has been submitted for consideration, and notice of such intended submission shall be given in the notice for such General Meeting, and any regulations so confirmed may be altered, amended, or repealed after notice given as aforesaid at an ensuing General Meeting summoned for that purpose in manner provided by the By-laws.

Standing Orders of Council.

(37.) Subject to any of the provisions of the Charter or the By-laws, the Council may for the due and proper regulation of its own proceedings make Standing Orders, and subject to any provisions therein contained, and relating thereto, may at any time add to, alter, amend, or rescind such Standing Orders.

PART IV.—LOCAL SECTIONS.

Formation of a Local Section.

(38.) A Local Section may be formed by the Council, whenever thirty or more Members residing in, or otherwise identified in interest with a place, locality

or district, whether situate within or without Great Britain or Ireland, make application in writing to the Council, signed by such Members, to cause a Local Register to be made for that purpose, and represent to the Council that the establishment of such Local Section will further more efficiently the objects of the Society. If the Council, upon consideration of such application, consider that it would be expedient to form such Local Section they may require the signatories to form a provisional committee, and to submit draft rules under which the business of the Local Section is intended to be managed and conducted, and may provisionally approve such draft rules with any modifications thereof which to the Council may seem requisite or desirable, and upon such provisional approval, to elect a committee and officers as hereinafter provided. Upon notice to the Council that such committee and officers have been elected at a meeting of any of the signatories and any other Members numbering in the whole not less than thirty, and that the draft rules approved by the Council as aforesaid have been adopted at such meeting, the Council shall allow the adopted rules as the rules of the said Local Section, and from the date of such allowance the said Local Section shall be deemed to be constituted in accordance with the Charter and By-laws.

Rules of a Local Section.

(39.) The rules of every Local Section shall contain the following provisions:—

- (i.) That the business of the Local Section shall be conducted by a Committee chosen from Members on the Register of the Local Section, consisting of a Chairman, Honorary Secretary, and ordinary Members of Committee as provided by Rules.
- (ii.) That any Member of the Society, whether resident at or otherwise interested in the place or locality where the Local Section is constituted, shall, upon application to the Honorary Secretary, be entitled to have his name entered upon the Register, and to become and remain a Member thereof upon payment of the annual sectional subscription, if any, for that and each succeeding year during which he remains a Member of the Local Section, without any entrance fee and without being elected.

Alterations of Rules.

(40.) Amendments in, alteration of, or additions to the rules of a Local Section shall be submitted to the Council, and shall not become operative or of any effect unless and until they have been allowed by the Council, and notice of such allowance has been given by the General Secretary.

Expenses of Local Section.

(41.) The expenses of a Local Section shall be defrayed out of the annual sectional subscriptions (if any) of the Members of such Section, but the Council in its discretion may defray the whole or may make a grant in aid thereof out of the General Funds of the Society, and a Local Section shall not pay, or incur, any expenses except reasonable sums for the clerical assistance, printing, stationery, advertising, reporting, and postage required for conducting the business, and the hire of rooms for meetings thereof without the previous sanction in writing of the Council, and an account of the income received and the expenditure paid or incurred by a Local Section during the year ending on the thirty-first day of December, shall on or before the fifteenth day of February in the following year be sent to the Council.

Communications to Local Sections.

(42.) The Committee of a Local Section, in furtherance of the objects of the Society, may invite or receive communications from any person, whether a Member of the Society or not, upon any subject coming within the scope of the said objects, and such communications may be made by the reading of an essay or paper, or the delivery of a lecture at a meeting of the Members of the Local Section, and every such communication—if intended for publication in the Journal—shall, before or after such meeting as the circumstances may require, be sent to the Council for their con-

sideration, and the Council may in their discretion, subject to any condition or to any by-law or rule relating thereto, cause the same, with or without any discussion thereof, or any condensed abstract thereof, to be published in the Journal.

Sectional Officers ex-officio Members of Council.

(43.) The Chairman and Honorary Secretary of every Local Section, duly constituted, shall become, and remain, during their respective terms of office *ex-officio* Members of the Council.

Restriction on Communications by Local Sections.

(44.) Neither the Committee of a Local Section nor any meeting thereof shall cause to be published, or authorise or countenance the publication of any notice upon any matter or subject whatsoever either in the name of the Society or in the name of the said Committee or Section, to the general public, except it relate wholly to the ordinary business of such Local Section, and except as provided in By-law 55, and all other notices and communications shall be conducted through or shall be subject to the express sanction of the Council.

Cesser of a Local Section.

(45.) The Council may, at a special meeting called for that purpose, declare by resolution that a Local Section shall be suspended in operation, or shall be dissolved:—

- (a) If it shall appear that the further continuance of the Section is not in accord with or conducive to the interests of the Society, or the furtherance of its objects.
- (b) Whenever three-fourths of the Members of the Local Section vote in favour of a resolution, proposed at a meeting called for the express purpose, that it is desirable such Section shall be suspended or dissolved.

PART V.—SUBJECT GROUPS.

Formation of a Subject Group.

(46.) A Subject Group may be formed by the Council, whenever thirty or more Members identified in interest with a particular subject within the objects of the Society make application in writing to the Council, signed by such Members, to cause a Register to be made for that purpose, and represent to the Council that the establishment of such Subject Group will further more efficiently the objects of the Society. If the Council, upon consideration of such application, consider that it would be expedient to form such Subject Group they may require the signatories to form a provisional committee, and to submit draft rules under which the business of the Subject Group is intended to be managed and conducted, and may provisionally approve such draft rules with any modifications thereof which to the Council may seem requisite or desirable, and upon such provisional approval, to elect a committee and officers as hereinafter provided. Upon notice to the Council that such committee and officers have been elected at a meeting of any of the signatories and any other Members numbering in the whole not less than thirty, and that the draft rules approved by the Council as aforesaid have been adopted at such meeting, the Council shall allow the adopted rules as the rules of the said Subject Group, and from the date of such allowance the said Subject Group shall be deemed to be constituted in accordance with the Charter and By-laws.

Rules of a Subject Group.

(47.) The Rules of every Subject Group shall contain the following provisions:—

- (i) That the business of the Subject Group shall be conducted by a Committee chosen from Members on the Register of the Subject Group consisting of a Chairman, Honorary Secretary, and ordinary members of Committee as provided by Rules.
- (ii.) That any Member of the Society shall, upon application to the Honorary Secretary, be entitled to have his name entered upon the Register, and to become and remain a Member of the Group upon payment of the annual Group subscription, if any, for that and each succeeding year during which he remains a

Member of the Subject Group without any entrance fee and without being elected.

- (iii.) Ordinary meetings of the Subject Group may include Conferences on special topics and may be held at the discretion of the Committee in centres where the subjects to be discussed have particular local industrial importance. The Secretary of the Local Section in the area of which the meetings are to be held is to be consulted as to the proposed dates of such meetings.

Alterations of Rules.

(48.) Amendments in, alterations of, or additions to the rules of a Subject Group shall be submitted to the Council, and shall not become operative or of any effect unless and until they have been allowed by the Council, and notice of such allowance has been given by the General Secretary.

Expenses of a Subject Group.

(49.) The expenses of a Subject Group shall be defrayed out of the annual Group subscriptions (if any) of the Members of such Group, but the Council in its discretion may defray the whole or may make a grant in aid thereof out of the General Funds of the Society, and a Subject Group shall not pay, or incur, any expenses except reasonable sums for the clerical assistance, printing, stationery, advertising, reporting, and postage required for conducting the business, and the hire of rooms for meetings thereof without the previous sanction in writing of the Council, and an account of the income received and the expenditure paid or incurred by a Subject Group during the year ending on the thirty-first day of December shall, on or before the fifteenth day of February in the following year, be sent to the Council.

Communications to Subject Groups.

(50.) The Committee of a Subject Group, in furtherance of the objects of the Society, may invite or receive communications from any person, whether a member of the Society or not, upon any subject coming within the scope of the said objects, and such communications may be made by the reading of an essay or paper, or the delivery of a lecture at a meeting of the Members of the Subject Group, and every such communication, if intended for publication in the Journal, shall, before or after such meeting, as the circumstances may require, be sent to the Council for their consideration, and the Council may in their discretion, subject to any condition or to any by-law or rule relating thereto, cause the same, with or without any discussion thereon, or any condensed abstract thereof, to be published in the Journal.

Group Officers ex-officio Members of Council.

(51.) The Chairman and Honorary Secretary of every Subject Group shall become, and remain, during their respective terms of office *ex officio* Members of the Council.

Restriction on Communications by Subject Groups.

(52.) Neither the Committee of a Subject Group nor any meeting thereof shall cause to be published, or authorise or countenance the publication of any notice upon any matter or subject whatsoever, except as provided in By-law 50, either in the name of the Society or in the name of the said Committee or Group, to the general public (except it relate wholly to the ordinary business of such Subject Group), and, except as aforesaid, all other notices and communications shall be conducted through or shall be subject to the express sanction of the Council.

Cesser of a Subject Group.

(53.) The Council may, at a special meeting called for that purpose, declare by resolution that a Subject Group shall be suspended in operation, or shall be dissolved :—

- If it shall appear that the further continuance of the Group is not in accord with or conducive to the interests of the Society, or the furtherance of its objects.
- Whenever three-fourths of the Members of the Subject Group vote in favour of a resolution, proposed at a meeting called for the express purpose, that it is desirable such Group shall be suspended or dissolved.

PART VI.—THE JOURNAL.

Conduct of the Journal.

(54.) The Journal shall be under the sole conduct and management in all respects of the Council, but they may at any time, subject to any rules and regulations made by them in reference thereto, appoint from amongst the Members any Committees which seem to them desirable to assist in such conduct or management, and subject as aforesaid, shall be at liberty to delegate any of their duties or functions as regards the Journal to any such Committee. The Council may also, in addition to the Editor or Editors, appoint upon and subject to such terms, and at such reasonable remuneration as they may decide, any managers, contributors, sub-editors, abstractors or other persons to assist in the compilation, preparation, and publication of the Journal, any of whom may or may not be Members.

Prior Right of Publication.

(55) It shall be a condition of acceptance of every communication made to any meeting of the Society or of a Local Section or Subject Group or submitted to the Council for publication in the Journal, whether made by a Member or not, that, unless there shall have been a previous agreement with the Council to the contrary, the Society shall thereby become entitled to a prior right of publication thereof, in whatever form the Council decide to make such publication in the Journal, for three months after its acceptance, and if the person by whom the communication is intended to be made is not a Member, he shall be informed of this condition by notice in writing from the General Secretary, or the Honorary Secretary of the Local Section or Subject Group. If, however, a communication to a Local Section or Subject Group is deemed by the Council not to be of general interest to the Members of the Society, the Committee of the Local Section or Subject Group shall have the right to publish and circulate such communication, subject to such rules as the Council may from time to time determine.

Publication of Journal.

(56.) The Council shall cause the Journal to be published and sent to the Members at least once in every month, upon such dates as they may think fit, and they may sell, or present gratuitously, or exchange copies, for any part thereof to or with any State or public department, or any institution, library, society, corporate body, company or person whatsoever.

Contents of Journal.

(57.) Nothing shall be printed or published in the Journal the printing and publication whereof has not been authorised by the Council, or any Committee appointed by them, and acting in such respect for and on behalf of the Council, but the Council or such Committee may authorise the printing and publication of any matter or thing coming within or relating to the objects of the Society, in such manner as they may determine, and may also accept and publish for payment any advertisements of which they may not disapprove, and all payments made therefor, or for any copies of the Journal sold as aforesaid, shall be credited to the cost of preparing, editing, printing, publishing, and distributing the Journal.

PART VII.—FINANCIAL.

Financial Management by Council.

(58.) The administration of the Funds, and of all financial matters of or relating to the Society, shall be conducted and managed by the Council, who shall act as a general Committee of Finance, but they may delegate any of their duties or functions in respect thereof to Committees composed of Members of and appointed by the Council, and such Committees shall act only subject to the control of the Council, and of any rules made by them in that behalf.

Annual Balance Sheet.

(59.) The Council shall receive and consider and may adopt the Annual Balance Sheet and General Financial Statement submitted by the Treasurer, and the report of the Auditors thereon, and shall, whether adopted or not, cause the Balance Sheet, Statement, and

Report aforesaid to be printed and sent to all the Members as a notice under the By-laws, and such notice shall be sent not later than the thirtieth day of June in every year.

Receipts and Payments.

(60.) All moneys due or payable to the Society shall be paid to and received by the Treasurer, or other person in that behalf appointed by the Council, and a receipt for every such payment shall be given in the name of the Society by the said Treasurer or other person. All moneys received as aforesaid shall be paid forthwith into the account of the Society kept at such bankers as may for the time being be appointed by the Council to be the bankers of the Society, and every cheque drawn on the said account shall be signed by two members of the Council duly authorised by the Council and countersigned by the General Secretary.

Borrowing Powers.

(61.) The Council may at any time, in the exercise of their discretion, borrow money temporarily, and may pay interest thereon, and may charge the property, assets, and investments of the Society, so far as they are enabled lawfully so to do, for the repayment and payment of such borrowed money and interest respectively.

Investments.

(62.) All life compositions, and all donations, bequests, and endowments, unless given subject to any other condition, or otherwise provided by any Deed or Trust relating thereto, shall, and all funds of the Society not needed immediately for the ordinary purposes thereof may be invested in the name of the Society or in the names of three Members of the Society who shall not be Members of the Council in any securities in which Trustees are from time to time authorised to invest by virtue of the Trustee Act, 1893, or any subsisting statutory modification thereof, with power, upon the authority and by the direction of the Council, from time to time to sell or vary such securities, and the Council may also from time to time place money on deposit with the bankers of the Society.

Auditor.

(63.) The Members present at the Annual General Meeting shall elect as Auditor of the Society, at such remuneration as the meeting shall determine, an Accountant who shall be a member of the Institute of Chartered Accountants in England and Wales, or a firm of Accountants the partners whereof are members of the said Institute, and the person or firm so elected shall, on election, become and remain the Auditor of the Society until the next Annual General Meeting, and then shall cease to be Auditor unless re-elected at such General Meeting.

Duties of Auditor.

(64.) The Auditor shall have access at all reasonable times to all the accounts and books of account in anywise relating to the income and expenditure and otherwise the financial affairs of the Society, and shall report upon the Balance Sheet prepared by the Treasurer to be submitted to the Annual General Meeting, as provided by the By-laws, and shall certify as to the correctness or otherwise of the liabilities, assets, and investments of the Society as existing upon the thirty-first day of December prior to the General Meeting at which the said Balance Sheet is to be presented, and his report and certificate as aforesaid, whatever its nature, shall be printed and sent to all the Members accompanying such Balance Sheet.

Casual Appointment and Removal of Auditor.

(65.) If at any time between two Annual General Meetings the office of Auditor becomes vacant, the Council may appoint an Auditor possessing the qualifications aforesaid, who shall remain in office until the next ensuing Annual General Meeting, and the Members may at any time after an Annual General Meeting, by a resolution passed by a majority of two-thirds of the votes of Members present and voting at an Extraordinary General Meeting at which not less than thirty Members are present and vote, remove any Auditor from office, and may, by a resolution

passed by a majority of the votes of Members present and voting at such meeting in number not less than aforesaid, elect at such remuneration as the meeting may then determine, any person or firm qualified to act as aforesaid.

PART VIII.—MEETINGS.

Annual General Meeting.

(66.) The Annual General Meeting for the transaction of the ordinary general business of the Society shall be held at such place and time in every year as the Council may determine.

The ordinary annual business of the Society shall comprise:—

The election of:—

Members of the Council; the Auditor;

And any other officers provided by the Charter or By-laws, to be elected at such meeting.

The reception and adoption of the Annual Report of the Council, and the Balance Sheet, Statement of Investments, and Financial Statement of the Treasurer submitted by the Council, together with the Auditor's report thereon.

And may include:—

An Address by the President.

Any communications accepted by the Council, and the discussion of the same if deemed desirable.

Conferences or discussions between the Members, or between Members and persons who are not Members, and visits to and inspection of works, manufactories, and other places of interest to the Members in furtherance of the objects of the Society.

Such other business as may be desirable and of which notice, according to the By-laws, has been duly given.

Extraordinary General Meeting.

(67.) The Council may, whenever they so decide, call an Extraordinary General Meeting of the Society, and such meeting shall be called by them on receiving a requisition in writing so to do signed by not less than thirty Members, and stating the object of the proposed meeting, but business other than that for which the meeting was called shall not be discussed or transacted at an Extraordinary General Meeting.

Notices of General Meetings.

(68.) Notice of the day, hour, and place of the commencement of any Annual General Meeting, shall be sent to all the Members two months at the least before such commencement. In the case of an Extraordinary General Meeting not less than fourteen days' notice thereof shall be sent either with or in the next succeeding issue of the Journal after the decision of the Council to call such meeting, or after the Council meeting next following the date upon which the requisition relating thereto was received by the General Secretary. The business to be considered at any such Extraordinary General Meeting shall be set forth on the said notice.

Quorum at General Meetings.

(69.) The business of an Annual General Meeting shall not commence until the Members present at the meeting, personally or by proxy, number thirty or upwards.

The business for which an Extraordinary General Meeting is called shall not commence until the Members present at the meeting, personally or by proxy, number thirty or upwards, and if within one hour after the time appointed for the commencement of such meeting the said number of Members are not present together, the meeting shall be thereupon and thereby dissolved. A Member may appoint another Member as his proxy at any meeting, by giving to the General Secretary notice in writing to that effect not less than 24 hours before the time fixed for such meeting.

Conduct of Business at Meetings.

(70.) Subject to the provisions of the By-laws relating thereto, the order of and the manner of conducting the business, and the way in which Members may record their votes thereat, or any other matter or thing of or relating to a General Meeting of the

Society not provided for in the Charter or By-laws, shall be regulated by rules made from time to time by the Council, but such rules or any addition thereto, alteration, amendment, suspension, or rescission thereof, shall, before becoming operative, be submitted to and approved at an Annual General Meeting.

Notice of Motion at Annual General Meeting to be given.

(71.) A Member wishing to bring before the Annual General Meeting any motion not relating to the ordinary annual business of the Society as aforesaid, shall give notice to the Council not less than one month before such Annual General Meeting, and no motion shall come before the meeting unless notice thereof has been so given, and on receipt of such notice of motion the Council shall give notice thereof in the next issue of the Journal.

Chairman of Meetings.

(72.) At all meetings of the Society the President for the time being, shall be Chairman, or in his absence the Chairman shall be elected from among the Members present.

Minutes to be Evidence.

(73.) Minutes shall be made in proper books, to be provided for the purpose, of all resolutions and proceedings of meetings of the Society or of the Council, or of each Local Section or Subject Group, and of any committee appointed by the Council or a Local Section or Subject Group, and every Minute signed by the Chairman of the meeting to which it relates, or by the Chairman of a subsequent meeting, shall be sufficient evidence of the facts stated therein.

PART IX.—MISCELLANEOUS.

Notices to Members.

(74.) Any notice required by the Charter or the By-laws to be given to Members whether relating in anywise to the affairs of the Society or of a Local Section may be a written or printed notice, and may be sent to Members either separately or in, or with, or if printed may be printed under the heading of "Notices," as part of the next issue of the Journal, as the Council may direct, and any notice shall be deemed to have been duly sent to a Member when such notice, or the Journal in which it is sent, or printed, and addressed to the last address of the Member appearing in the Register, has been put into the post. All notices shall be signed by the General Secretary.

Provisions as to Register.

(75.) The Register shall be kept by the General Secretary, subject to the direction of the Council, and the last address of any Member entered therein shall for the purpose of all notices and otherwise be deemed to be the address of that Member, but a Member may at any time, by notice in writing to the General Secretary, require his address last entered in the Register to be altered.

Award of a Medal by Society.

(76.) The practice of awarding a medal periodically to a person selected by the Council may be continued by the Council, and the Council in its discretion may for such purpose apply the funds of the Society, subject to the following conditions (that is to say):—

- (a) The medal shall be called "the Medal of the Society of Chemical Industry," and shall not be awarded more frequently than once in every two years.
- (b) The recipient of the medal shall be a person who, in the opinion of the Council, has rendered conspicuous service to Applied Chemistry by research, discovery, invention, or improvements in processes, or to the Society in furtherance of its objects, and may or may not be a Member, and may be of any nationality.
- (c) More than one medal shall not be awarded in any year.
- (d) The presentation of the medal shall be made to the recipient either in person or proxy at the Annual General Meeting for that year in which the Council decides to award the medal, and the

name of the recipient and the said service in respect of which the medal is awarded shall be notified to the Members in the notice of such General Meeting.

And the proposal to award a medal shall be considered at any meeting of the Council held after previous notice of such proposal.

Library.

(77.) Every paper, map, plan, drawing, or model presented to the Society shall become the property of the Society, except the presenter thereof shall stipulate to the contrary at the time of presentation, and all such, together with all publications presented to or purchased by the Society, and a set of bound copies of all issues of the Journal and indices thereof published by the Society of 1881, or the Society, shall be kept upon the premises of the Society for the use of the Members, in charge of a Librarian or other person appointed by the Council, and shall not be removed from such premises or kept elsewhere except upon the authority of the Council.

Pensions.

(78.) The Council may, with the consent of a General Meeting, make such provision for pensioning retired officers and servants receiving remuneration in respect of their several offices and services as to the Council may seem just.

Custody of Certain Books.

(79.) The Register and all Minute and Account books, or other books dealing with or recording the conduct of the business of the Society, except the business transacted by a Local Section, shall be kept upon the premises of the Society in the custody of the General Secretary or other person appointed by the Council for that purpose, and shall not be removed from the said premises or kept elsewhere except by the authority of the Council.

Custody of Charter Deeds and Securities.

(80.) The Charter and all deeds, securities, and other documents of title shall be deposited for safe keeping with the bankers of the Society, who shall, whenever required, produce them for purposes of inspection and verification to the Auditor.

Custody and use of Common Seal.

(81.) The Common Seal of the Society shall be kept in such custody as the Council may for the time being determine, and such Seal shall not be affixed to any document or instrument except by order of the Council, and in the presence of two Members thereof, and every such document or instrument when sealed shall be signed by the two Members of the Council in whose presence the Seal was affixed, and countersigned by the General Secretary.

Indemnity of Officers.

(82.) All Members of the Council, the Auditor, General Secretary, Editors, and other officers shall be indemnified by the Society in respect of all losses and proper expenses incurred by them in or about the discharge of their respective duties, except such as may arise from their own respective wilful default, and none of them shall be liable for any other Member of the Council, Auditor, General Secretary, Editor, or other officer, or for joining in any receipt or document, or for any act of conformity, and for any loss or expense caused to the Society except in the case of the like default.

Interpretation of By-laws.

(83.) Any difficulty arising out of the proceedings of Local Sections or Subject Groups, or as to the interpretation of the By-laws shall be referred to the Council for decision.

Alteration of By-laws.

(84.) A new By-law, or the alteration, suspension, or repeal of any existing By-law, may be proposed at any time by the Council, and shall be proposed by them after receipt of a requisition so to do signed by at least fifty Members. The proposal made shall in all cases be set forth in the notice of the meeting at which it is to be considered and decided.



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